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Final Technical Report of
TÜBİTAK MRC, FI
on the
UNISWORK VIII
International Study Tour and Visit Programme on
Food Laboratories Management and Practice

Project No.: US/INT/08/006, XP/INT/08/007

Organized jointly by: UNIDO and the Government of Turkey through the Ministry of Industry and Trade, Turkish International Cooperation and Development Administration (TICA) and the TUBITAK Marmara Research Center Food Institute

Date of Project: 16 Nov.-20 Nov. 2009

Place of Project: Gebze/Kocaeli, Turkey

Report prepared by: Dr. Sena Saklar Ayyıldız

Date of the Report: 07.12.2009

Approved by: Assoc. Prof. Dr. Güner Özay

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1. SYNOPSIS OF ABSTRACT

UNIDO jointly with Turkey launched the eight technical support programme (UNISWORKVIII) in the field of agro-related industries. The idea was through the exchange of various experiences in different regions to enhance cooperation among various countries in the field of food laboratory management and practice for the development and upgrading of food laboratories local capabilities. The project facilitated sharing the national experiences, and provided 11 participants from food-processing enterprises, national food laboratories and the related institutions coming from Azerbaijan, Bangladesh, Moldova, Ethiopia, Kenya, Rwanda, Tanzania, Palestine, Tajikistan and Turkey (2) with theoretical knowledge and practical experience in food laboratory issues. The programme included the lectures, laboratory practices, technical visits, cultural visits, round table discussions, country presentations, opening and closing ceremonies. The programme was conducted on 16–20 Nov. 2009, at TUBITAK Marmara Research Centre, Food Institute, Gebze, Kocaeli, Turkey. At the end of the UNISWORKVIII programme, participants enhanced, upgraded and exchanged their skills, knowledge and practical experience on the project topics to improve the local laboratory and analyses capabilities in their countries.

2. INTRODUCTION

With the support of the Ministry Industry and Trade (MIT) Turkey and TICA (Turkish International Cooperation and Development Administration), a tailor-made technical programme was prepared by UNIDO jointly with the TUBITAK MRC, FI. UNISWORK VIII, the eight workshop and study tour programme was conducted in Gebze/ Kocaeli, Turkey, on 16-20 Nov., 2009. The project included the organization of an International Regional Study Tour and Visit programme for selected developing countries (i.e. Africa, Asia and Middle East) in Turkey looking to contribute to the exchange of experiences, increasing regional cooperation and eventually the economic development among the participating countries/institutions or enterprises. The project facilitated the sharing of national experiences, and provided 11 participants from food processing enterprises, national food laboratories and related institutions coming from different countries such as Azerbaijan, Bangladesh, Moldova, Ethiopia, Kenya, Rwanda, Tanzania, Palestine, Tajikistan and Turkey (2) with theoretical knowledge and practical experience on food laboratory management and food analyses. This programme created a platform for interactive discussions including theoretical and practical issues regarding food laboratory management and practice as well as technical, cultural visits and demonstrations.

A financial support to implement this programme was provided by the Turkish Government from its special purpose contributions to UNIDO's IDF and it was co-financed from UNIDO's resources. International travel expenses were supported by TICA. The Scientific and Technical Research Council of Turkey (TUBITAK), the Marmara Research Centre (MRC), Food Institute (FI) was the national counterpart, which was recognized as leading organization within the country and with respect to the management of food laboratory and other food related issues.

At the end of the programme, participants from selected geographical regions increased their theoretical knowledge and practical experience in the field of: food laboratory management and practice including to quality control systems, good laboratory practices, food testing laboratory accreditation (general principles, ISO17025:2005 requirements), quality control in food

testing laboratories, reference materials and proficiency testing, principles of method validation and measurement uncertainty in chemical and microbiological analysis. Practical experiences were gained with different analyses in microbiology, nutrition, food chemistry, mineral and heavy metal, HPLC and GC laboratories.

This programme also led to the start up a fruitful cooperation among the developing countries (i.e. Africa, Asia and Middle East) and Turkey to reinforce their exchange network for industrial cooperation and business. An example of this cooperation is that a high level delegation from Bangladesh will visit TUBITAK MRC laboratories in Dec., 2009 for business.

3. DEVELOPMENT OF THE INTERNATIONAL STUDY TOUR AND VISIT PROGRAMME

3.1. Programme Arrangements

3.1.1. Training Administration

Administrative arrangements were performed to implement the programme. The arrangements are in below:

- Arrangement of lecturers: Lecturers from institutions, Ministry of Agriculture and FI were invited to the programme. The agreements were made with the lecturers to arrange their programmes and to prepare their presentations and related notes. Logistics requirements such as accommodation, travel, etc. were given for the invited lecturers. Invited lecturers were paid according to the national rules.
- Arrangement of facilities: Training room facilities and training equipment were prepared before the programme started.
- Arrangement of training material: Training materials were prepared, and copied for each participant.
- Arrangement of technical visits: Arrangements were done to visit national food laboratories of Turkish Ministry of Agriculture.
- Meeting at the airport: All participants were picked up at the airport upon their arrival and transferred to hotel. They also were picked up from their hotel to the airport on their departure. Accommodation and

internal travel arrangements for the representatives from MIT, TICA and lecturers were also provided.

- Arrangement of internal travel: Arrangements were done for the internal travel between hotel and TUBITAK, MRC for every day, internal travel for technical visits from Gebze to laboratories (Bursa) and cultural visits (Istanbul). Local travel arrangements were done for the invitees participated in welcome and closing ceremonies.
- Arrangement of accommodation: Individual hotel rooms in Istanbul were booked for the participants. Logistic letter was prepared giving some introductory information about the programme and organization.
- Arrangement of pocket money: Every participants were paid 20$ pocket money and plus 7 $ for dinner per day. 15 $ per day was also paid for early arrival and late leaving participants.
- Arrangement of lunch, coffee breaks
- Arrangement of cultural visits: English spoken tourist guide was invited to the cultural visits, and the history of cultural places was explained. Necessary arrangements were done for booking of tourist guide and entering of cultural places.
- Arrangements of welcome reception and closing ceremony: Welcome reception was held in a restaurant within the TUBITAK MRC campus at the starting day of the programme. Closing ceremony was held in a restaurant in Istanbul, all participants, lecturers and representatives from MIT and UNIDO were invited.

3.1.2. Preparation of Training Materials

Presentation notes were prepared as power point presentations, which were distributed to the participants at the first day of the programme. Lecture notes were also prepared with detailed information as in a textbook and CD, distributed to the participants. Country presentations were also copied in the form of booklet. In addition, some training material such as bags, notebooks, pencils, etc. were supplied to the participants.

3.1.3. Lecturers

Lecturers and their lecture names are listed below:

- Pinar Yildizlar (Turkak, Turkish Accreditation Agency), Introduction to laboratory quality systems and laboratory management, Good Laboratory Practices
- Dr. Hayrettin Ozer (TUBITAK MRC, FI), Food Testing Laboratory accreditation (general principles, ISO17025:2005 requirements), Principles of method validation and measurement uncertainty in chemical analysis
- Nihat Ozcan (TUBITAK MRC, FI), Quality control in food testing laboratories, reference materials and proficiency testing
- Asli Kisikkaya (TUBITAK MRC, FI), Principles of method validation and measurement uncertainty in microbiological analysis
- Birdem Cetinkaya (TUBITAK MRC, FI), Food labelling (regulations, importance and applications)

3.1.4. Laboratory visit

In the content of programme, Turkish Ministry of Agriculture National Food Control and Research Institute laboratories were visited in Bursa. Participants had a chance to visit many different laboratories, departments and sample reception such as: quality management department, biotechnology laboratory, pesticide laboratory, agricultural food products laboratory, animal food products laboratory, microbiology laboratory and feed and feed products laboratory. They obtained very useful information about laboratory management and Turkish laboratory control system.

3.1.5. Other Activities

Mr. Izzet Ahmet Bozbey from TICA participated in the opening session and made an opening speech. Assoc. Prof. Dr. Güner Özay, Director of FI, presented TUBITAK MRC and FI facilities to participants. At the first day of programme, participants had a welcome dinner in a special restaurant in MRC campus. TICA representative, project coordinator, organisation team and participants joined the welcome dinner, and everybody shared their

experiences. Cultural issues specific to regions and countries were discussed. Participants talked about traditional issues in their countries.

Also, in the content of cultural programme, participants visited to Blue Mosque, Hagia Sophia Mosque and its museum, Suleymaniye Mosque, The Basilica Cistern, Grand Covered Bazaar, Sultan Ahmet region in Istanbul.

A closing ceremony was held on the last day of the programme at a restaurant in Istanbul. Vice President of TUBITAK MRC, UNIDO representative, representative of Turkish Ministry of Industry and Trade, Director of FI, participants, lecturers, organisation team and laboratories staff were attained the closing ceremony. Representatives of UNIDO, Turkish Ministry of Industry and Trade, TUBITAK and participants made closing speeches, and then certificates were given.

3.2. Session

The programme started by the opening speeches of the representative from TICA and the Director of FI. Lecture notes and presentations were published in a textbook, and given to the participants to use them after the programme. Lecture notes were also copied to CD's and distributed to the participants. Some training materials such as notebooks, pencils, bags, etc. were given to participants to use them during lectures.

The programme was organized for 12 participants coming from Azerbaijan, Bangladesh, Moldova, Ethiopia, Kenya, Rwanda, Tanzania, Palestine, Tajikistan, Uganda and Turkey (2). Ugandan participant did not participate in the programme and she informed us her passport was in courier and they did not deliver her passport at the exact time before her departure.

Training programme was held according to the schedule of the lectures written in the programme agenda. Training programme consisted of 15.4% theoretical sessions, 49.5% practical (lab. and in-plant) sessions, 35.1% others (round table discussions, exchange of experiences, etc.).

On the first day of the programme, participants presented their country papers. They discussed the problems and needs for the food industry and food laboratory systems in their countries. Presentations and round table discussions were helpful to exchange the experiences and to develop cooperation among the participants and countries.

Theoretical sessions consisted of the lectures: introduction to laboratory quality systems and laboratory management, good laboratory practices, food testing laboratory accreditation (general principles, ISO17025:2005 requirements), quality control in food testing laboratories, reference materials and proficiency testing, principles of method validation and measurement uncertainty in chemical and microbiological analysis, food labelling (regulations, importance and applications).

Practical sessions consisted of different laboratory applications. They were done in microbiological laboratory (Total viable count, *E.coli*, *S.aereus* analyses), nutrition laboratory (protein, Vit C and Vit A analyses), food chemistry laboratory (total sugar, invert sugar, salt, HMF, total acidity analyses), mineral and heavy metal laboratory (Ca, Zn, Pb, Cd analyses), HPLC laboratory (sugar components, aflatoxins and deoxynivalenol analyses), oil/fats laboratory (free fatty acid, peroxide, fatty acid composition analyses by using GC).

In the technical visit part, Participants visited the Food laboratories of Turkish Ministry of Agriculture and Rural Affairs in Bursa. Participants were informed about the laboratory management, sample reception and different food analyses techniques.

3.3. Conclusion and Recommendations

Participants evaluated the programme with positive remarks. The level of training and training materials was evaluated as excellent/good. TUBITAK MRC's reactions on participants' comments are as follows:

1. Some participants expected more laboratory applications.

UNISWORKVIII contained more laboratory application sessions, and less theoretical session. It is not possible to cancel all theoretical part, this time theoretical sessions were very limited. Programme time is limited to 5 days, and programme contained many activities such as cultural visits, technical visits, country presentations, etc. It is always tried to place more practical time in the programme as much as not to cancel other cultural or technical activities.

2. Many participants wanted longer duration of programme time
It depends on the available funds.

3. Many participants are not satisfactory about the visa procedures
Unfortunately, it is out of the TUBITAK MRC scope.

4. Some participants understand the programme as a training programme because "training" is mentioned in many questions of the Evaluation report. Participants sometimes misunderstand the concept of the programme and the questions on the Evaluation report confused some of them. Because, it is actually study tour and workshop programme, 5 days is too short for training of these subjects. Many participants consider as it is a training programme and expect to have more time to learn these subjects.

5. Many participants request more focused programmes.
It would be better if we could organise more specific and focused programmes such as applied microbiology laboratory or pesticide analysis. It would be more time to learn specific topics in a 1 week of the programme.

3.4. Conceptual Follow-up Activities

Participants evaluated the training programme. Most of the participants recommended repeating the programme for future participants. The programme can be repeated for different groups of participants in future. Additionally, questionnaires were prepared by TUBITAK MRC FI, and distributed to the participants to evaluate the lecturers after each lecture. It is an internal-self evaluation system of TUBITAK MRC FI to improve the future programmes.

4. Terminal Section

Participants evaluated the programme generally with positive remarks. Their recommendations and requests were evaluated and the results were given in 3.3. part of the report. Their recommendations will be taken into consideration in future programmes.

APPENDIX

PROGRAMME SUMMARY

UNIDO, in cooperation with the Ministry of Industry of Turkey, Turkish International Cooperation and Development Administration through TUBITAK MRC FI, was successfully organized technical support programme in the field food laboratory management and practice.

Objective of the programme was to enhance the cooperation among various countries and to facilitate the exchange of experiences and to upgrade the technical knowledge and managerial capabilities of specialists from selected developing countries such as, Azerbaijan, Bangladesh, Moldova, Ethiopia, Kenya, Rwanda, Tanzania, Palestine, Tajikistan, Uganda and Turkey (2) with modern techniques and management in the field of food laboratory management and practice. It was expected that this training programme led to the start up of a fruitful cooperation among the developing countries and Turkey to reinforce their exchange network for industrial cooperation and business.

PROGRAMME AGENDA

UNIDO International Training Programme on
Food Laboratory Management and Practice

TUBITAK, Marmara Research Centre
Food Institute
Gebze- Kocaeli, TURKEY
16-20 November 2009

Programme for the UNISWORK VIII

14-15th Nov. 2009 (Saturday, Sunday)

Arrivals

16th Nov. 2009, Day 1 (Monday)

10:00-10:30	Opening session
10:30-11:15	Introduction to laboratory quality systems and laboratory management (P.Yildizlar)
11:15-11:30	Coffee break
11:30-12:30	Good Laboratory Practices (P.Yildizlar)
12:30-13:30	Lunch break
13:30-16:30	Country presentations:
13:30-13:45	Ms. Nurana Huseynova, Azebaijan
13:45-14:00	Mr. Mostafa A. Swapan, Bangladesh
14:00-14:15	Mr. Samson G. Gabre, Ethiopia
14:15-14:30	Mr. Clarkson Nyambok, Kenya
14:30-14:45	Ms. Oxana Usatii, Moldova
14:45-15:00	Coffee break
15:00-15:15	Mr. Mohammad Mousa, Palestine
15:15-15:30	Mr. Philip Nzaire, Rwanda
15:30-15:45	Mr. Iraj Ahmadov, Tajikistan
15:45-16:00	Ms. Agnex Mneney, Tanzania
16:00-16:15	Mr. Kudret Avci and Yildiray Istanbullu, Turkey
16:15-16:30	Ms. Annette Nabbengo, Uganda
16:30-17:00	Food Institute laboratories visit
17:30-19:30	Welcome dinner

17th Nov. 2009, Day 2 (Tuesday)

09:00-09:45	Food Testing Laboratory accreditation (general principles, ISO17025:2005 requirements) (H.Özer)
09:45-10:00	Coffee break
10:00-10:45	*Quality control in food testing laboratories, reference materials and* proficiency testing (N.Özcan)

10:45-11:30	Principles of method validation and measurement uncertainty in chemical analysis (H.Özer)
11:30-12:15	Principles of method validation and measurement uncertainty in microbiological analysis (A. Kısıkkaya)
12:15-13:00	Lunch break
13:00-17:00	Cultural visit

18th Nov. 2009, Day 3 (Wednesday)

09:00-18:00	Technical visit to Food laboratories of Ministry of Agriculture and Rural Affairs, Bursa

19th Nov. 2009, Day 4 (Thursday)

09:00-09:45	Food labelling (regulations, importance and applications) (B.Çetinkaya)
09:45-10:00	Coffee break
10:00-11:15	Laboratory applications: microbiological analyses (Total viable count, *E.coli*, *S.aereus*) (Group A) nutrition analyses (protein, Vit C and Vit A analyses) (Group B)
11:15-12:30	Laboratory applications: microbiological analyses (Total viable count, *E.coli*, *S.aereus*) (Group B) nutrition analyses (protein, Vit C and Vit A analyses) (Group A)
12:30-14:00	Lunch
14:00-15:15	Laboratory applications: Food chemistry analyses (total sugar, invert sugar, salt, HMF, total acidity) (Group A) Mineral and heavy metal analyses (Ca, Zn, Pb, Cd,) (Group B)
15:15-15:30	Coffee break
15:30-16:45	Laboratory applications: food chemistry analyses (total sugar, invert sugar, salt, HMF, total acidity) (Group B) Mineral and heavy metal analyses (Ca, Zn, Pb, Cd,) (Group A)

20th Nov. 2009, Day 5 (Friday)

09:30-10:30	Laboratory applications: Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group A) Oil/Fats analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group B)
10:30-11:00	Coffee break
11:00-11:45	Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group A)

	Oil/Fats analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group B)
11:45-13:00	Lunch
13:00-14:45	Laboratory applications: Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group B) Oil analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group A)
14:45-15:15	Coffee break
15:15-17:00	UNIDO evaluation
18:30-23:00	Closing ceremony

21-22nd Nov. 2009, (Saturday, Sunday)

Departures

PARTICIPANT LIST FOR THE WELCOME DINNER ON 16 Nov. 2009.

NO	**NAME AND SURNAME**	**TITLE**
1	Dr. Sena Saklar Ayyildiz	Programme Coordinator-Lecturer
2	Mr. Izzet Ahmet Bozbey	Representative form TICA
3	Dr. Ibrahim Sani Ozdemir	Researcher, organisation team
4	Dr. Canan Dogan	Researcher, organisation team
5	Rukiye Sanci	Researcher, organisation team
6	Hatice Semizer	Researcher, organisation team
7	Mr. Samson Girma GABRE	Participant from Ethiopia
8	Mr. Clarkson NYAMBOK	Participant from Kenya
9	Mr. Philip NZAIRE	Participant from Rwanda
10	Ms. Nurana HÜSEYNOVA	Participant from Azerbaijan
11	Mr. Mohammad MOUSA	Participant from Palestine
12	Mr. K.M. Mostafa ANWAR SWAPAN	Participant from Bangladesh
13	Ms. Agnes MNENEY	Participant from Tanzania
14	Ms. Oxana USATII	Participant from Moldova
15	Mr. Iraj AHMADOV	Participant from Tajikistan
16	Mr. Kudret Avcı	Participant from Turkey
17	Mr. Yildiray Istanbullu	Participant from Turkey

PARTICIPANT LIST FOR THE CLOSING CEREMONY ON 20 Nov. 2009.

NO	NAME AND SURNAME	TITLE
1	Mr. Dalibor Kysela	UNIDO representative
2	Enis Tüyeni	Vise President of TUBITAK MRC
3	Assoc. Prof. Dr. Guner Ozay	TUBITAK MRC FI Director
4	Mrs. Ummuhan Yokus	Representative of Turkish Ministry of Industry and Trade
5	Dr. Sena Saklar Ayyildiz	Programme Coordinator
6	Mr. Samson Girma GABRE	Participant from Ethiopia
7	Mr. Clarkson NYAMBOK	Participant from Kenya
8	Mr. Philip NZAIRE	Participant from Rwanda
9	Ms. Nurana HÜSEYNOVA	Participant from Azerbaijan
10	Mr. Mohammad MOUSA	Participant from Palestine
11	Mr. K.M. Mostafa ANWAR SWAPAN	Participant from Bangladesh
12	Ms. Agnes MNENEY	Participant from Tanzania
13	Ms. Oxana USATII	Participant from Moldova
14	Mr. Iraj AHMADOV	Participant from Tajikistan
15	Mr. Kudret Avcı	Participant from Turkey
16	Mr. Yildiray Istanbullu	Participant from Turkey
17	Dr. Ibrahim Sani Ozdemir	Researcher, organisation team
18	Dr. Canan Dogan	Researcher, organisation team
19	Rukiye Sanci	Researcher, organisation team
20	Hatice Semizer	Researcher, organisation team
21	Cesarettin Alaşalvar	Researcher
22	Abdullah Turkmenler	Researcher
23	Hayrettin Ozer	Lecturer/ Researcher
24	Elmas Oktem	Researcher
25	Mehlika Borcakli	Researcher
26	Ferruh Adoglu	Researcher
27	Ebru Pelvan	Researcher
28	Birdem Cetinkaya	Lecturer/ Researcher
29	Halil Kocer	Researcher
30	Vedat Yalçinkaya	Technician
31	Arif Selcuk	Laboratory Technician
32	Basri Cirak	Laboratory Technician
33	Nabi Uygun	Laboratory Technician
34	Gökmen Serdar	Laboratory Technician

35	Yunus Kurtoglu	Laboratory Technician
36	Sefer Yapici	Laboratory Technician
37	Serkan Savsar	Laboratory Technician
38	Bulent Karadeniz	Researcher
39	Mustafa Bozbey	Researcher
40	Senem Yılmaz	Researcher

LECTURE NOTES AND PRESENTATIONS

2 copies of lecture notes, lecture presentations and country presentations are attached of this report.

TÜBİTAK
MAM

INTERNATIONAL WORKSHOP AND STUDY TOUR ON: FOOD LABORATORIES MANAGEMENT AND PRACTICE

UNISWORK VIII

TUBITAK

MARMARA RESEARCH CENTRE

FOOD INSTITUTE

LECTURE PRESENTATIONS

Gebze- Kocaeli, TURKEY

16-20 November 2009

TÜBİTAK
MAM

UNIDO International Training Programme on
Food Laboratory Management and Practice
16–20 November 2009

[redacted]

TUBITAK, Marmara Research Centre

Food Institute

Gebze- Kocaeli, TURKEY

16-20 November 2009

Draft Programme for the UNISWORK VIII

[redacted]

10:00-10:30	
10:30-11:15	
11:15-11:30	Coffee break
11:30-12:30	
12:30-13:30	
13:30-16:30	[redacted]
13:30-13:45	**Ms. Nurana HUSEYNOVA, AZERBAIJAN**
13:45-14:00	**Mr. Mostafa A. SWAPAN, BANGLADESH**
14:00-14:15	**Mr. Samson G. GABRE, ETHIOPIA**
14:15-14:30	**Mr. Clarkson NYAMBOK, KENYA**
14:30-14:45	**Ms. Oxana USATII, MOLDOVA**
14:45-15:00	Coffee break
15:00-15:15	**Mr. Mohammad MOUSE, PALESTINE**
15:15-15:30	**Mr. Philip NZAIRE, RWANDA**

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15:30-15:45	**Mr. Iraj AHMEDOV, TAJIKISTAN**
15:45-16:00	**Ms. Agnex MNENEY, TANZANIA**
16:00-16:15	**Mr. Kudret AVCI and Yildiray ISTANBULLU, TURKEY**
16:15-16:30	**Ms. Annette NABBENGO, UGANDA**
16:30-17:00	[redacted]
17:30-19:30	
[redacted]	
09:00-09:45	
09:45-10:00	**Coffee break**
10:00-10:45	[redacted]
10:45-11:30	
11:30-12:15	
12:15-13:00	
13:00-17:00	
[redacted]	
09:00-18:00	
[redacted]	
09:00-09:45	
09:45-10:00	**Coffee break**
10:00-11:15	**LABORATORY APPLICATIONS:** Microbiological analyses (Total viable count, *E.coli*, *S.aereus*) (GroupA) Nutrition analyses (protein, Vit C and Vit A analyses) (Group B)

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11:15-12:30	**LABORATORY APPLICATIONS:** Microbiological analyses (Total viable count, *E.coli*, *S.aereus*) (GroupB) Nutrition analyses (protein, Vit C and Vit A analyses) (Group A)
12:30-14:00	
14:00-15:15	**LABORATORY APPLICATIONS:** Food chemistry analyses (total sugar, invert sugar, salt, HMF, total acidity) (Group A) Mineral and heavy metal analyses (Ca, Zn, Pb, Cd,) (Group B)
15:15-15:30	Coffee break
15:30-16:45	**LABORATORY APPLICATIONS:** Food chemistry analyses (total sugar, invert sugar, salt, HMF, total acidity) (Group B) Mineral and heavy metal analyses (Ca, Zn, Pb, Cd,) (Group A)
09:30-10:30	**LABORATORY APPLICATIONS:** Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group A) Oil/Fats analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group B)
10:30-11:00	Coffee break
11:00-11:45	**LABORATORY APPLICATIONS:** Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group A) Oil/Fats analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group B)
11:45-13:00	
13:00-14:45	**LABORATORY APPLICATIONS:** Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group B) Oil analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group A)
14:45-15:15	Coffee break
15:15-17:00	UNIDO evaluation
18:30-23:00	

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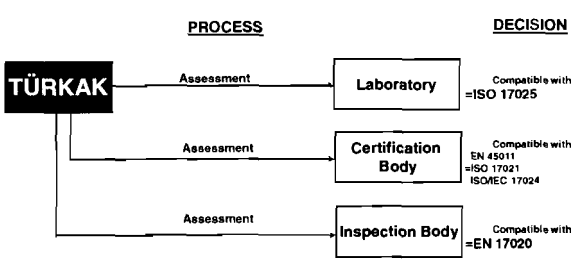
LECTURE 1

INTRODUCTION TO LABORATORY QUALITY SYSTEMS AND LABORATORY MANAGEMENT

Introduction to Laboratory Quality Systems and Laboratory Management

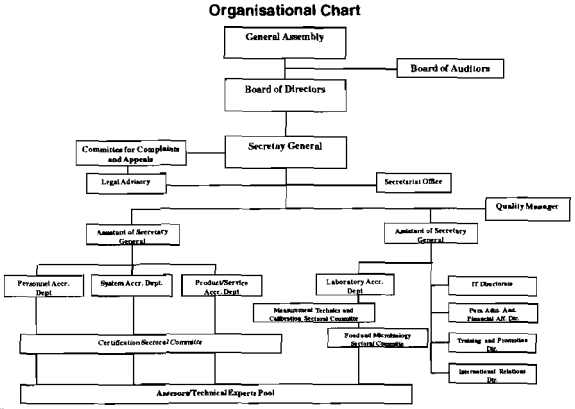
Pınar Yıldızlar AKSU
Turkish Accreditation Agency
Laboratory Accreditation Department
Case Manager, Lead Assessor

What is Accreditation Service?



Accreditation : **evaluation according to nationally and internationally recognized criteria, approval of competence and assessment at regular intervals of laboratories, inspection and certification bodies**

Organisational Chart



Status of TURKAK Accreditation

As at 15 March 2005

Application Department	Accredited
Laboratory Accrd. Dept.	
▪Calibration Laboratories	61
▪Testing Laboratories	239
Personnel Accred. Dept.	9
System Accred. Dept.	38
Product/Service Accrd. Dept.	
▪ Inspection Bodies	44
▪ Product Cert. Bodies	15
TOTAL	406

TURKISH ACCREDITATION AGENCY 4

ISO/IEC 17025

- 1 Scope
- 2 *Normative references*
- 3 Terms and definitions
- **4 Management requirements**
- **5 Technical requirements**

TURKISH ACCREDITATION AGENCY 5

ISO/IEC 17025

- **4 Management requirements**
  - 4.1 Organisation and management
  - 4.2 Quality system
  - 4.3 Document control
  - 4.4 Request, tender and contract review
  - 4.5 *Sub-contracting of test and calibration*
  - 4.6 Purchasing services and supplies
  - 4.7 Service to the client

TURKISH ACCREDITATION AGENCY 6

ISO/IEC 17025
3(5)

- **4 Management requirements**
  - 4.8 *Complaints*
  - 4.9 Control of non-conforming T/C work
  - 4.10 Improvement
  - 4.11 Corrective action
  - 4.12 Preventive action
  - 4.13 Records
  - 4.14 Internal audits
  - 4.15 Management reviews

TURKISH ACCREDITATION AGENCY 7

ISO/IEC 17025
4(5)

- **5 Technical requirements**
  - 5.1 General
  - 5.2 Personnel
  - 5.3 Accommodation and env conditions
  - 5.4 T/C methods incl sampling
  - 5.5 Equipment

TURKISH ACCREDITATION AGENCY 8

ISO/IEC 17025
5(5)

- **5 Technical requirements**
  - 5.6 Measurement traceability
  - 5.7 Sampling
  - 5.8 Handling and transportation of items
  - 5.9 Assuring the quality of T/C results
  - 5.10 Reporting the results

TURKISH ACCREDITATION AGENCY 9

ISO/IEC 17025,
4 Management requirements

Legal identity

The laboratory or the organisation of which it is part shall be an entity that can be held legally responsible

- Examples: Private company, public enterprise, public or local authority
- If the laboratory is part of a legal entity, the accreditation is granted to the legal entity in question

Impartiality, independence and integrity

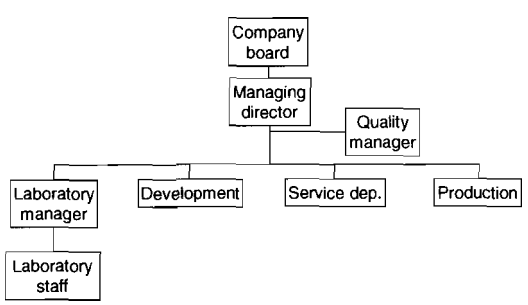
- **Identify potential conflicts of interest**
- Separated from production
- Directly under the central quality department or top management
- Remuneration: No payment per piece of work produced
- If laboratory concerned with e.g. Design: clear separation of *different responsibilities*
- Bankruptcy

Management and organisation

- competent to perform tests concerned
  - personnel aware of area of responsibility
  - supervision
- technical manager and deputy technical manager,
- appointed quality manager
- document showing responsibilities, available and kept up-to-date
- Top management shall ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

TURKISH ACCREDITATION AGENCY 13

Organisation chart



TURKISH ACCREDITATION AGENCY 14

Quality manual and related documentation, 1/2

- Quality policy statement
- organisation and management structure
- relations between management etc. and quality system
- procedures for documentation
- job descriptions
- approved signatories
- procedures for traceability
- scope
- procedures for reviews of all new work
- reference to procedure

TURKISH ACCREDITATION AGENCY 15

Quality manual and related documentation, 2/2

- proc. for handling of objects
- ref. to equipment and standards used
- ref. to proc. for calibr., verif. & maintenance of equipment
- ref. to verification practice
- proc.for feedback and corrective action
- arrangements for departures
- proc. for complaints
- proc. for confidentiality & proprietary rights
- proc. for audit and review

TURKISH ACCREDITATION AGENCY 16

Quality System

- *Top management shall provide evidence of* commitment to the development and implementation of the management system and to continually improving its effectiveness.
- Top management shall communicate to the organization the importance of meeting customer requirements as well as statutory and regulatory requirements.
- Top management shall ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

TURKISH ACCREDITATION AGENCY 17

Document control

- internally generated or from external sources
- a master list identifying the revision status of the documents
- appropriate documents available
- documents periodically reviewed
- uniquely identified
- obsolete documents *suitable marked*

TURKISH ACCREDITATION AGENCY 18

Procedures leading to a contract shall ensure that:

- The requirements including the methods to be used are adequately defined,documented and understood
- the laboratory has the capability and resources to meet the requirements
- the appropriate test and/or calibration method is selected and capable of meeting the clients requirements

Subcontracting

- test and calibration
  - normally not
  - if subcontracting - same requirements
  - inform client in writing
  - retain details
  - register of all subcontractors
  - accredited laboratories ok.
  - not accredited - approval by accreditation body

Outside support services and supplies

- Use only those with adequate quality to sustain confidence in results.
- Procedures to ensure that purchased equipment etc. comply with specified requirements.
- Record of all "approved" suppliers

Co-operation with the client

- Afford clients co-operation
- Ensure confidentiality to other clients

Complaints...

- Documented policy and procedures
- Record all complaints
- Doubt concerning the quality of the results - audit promptly
- determine the root cause(s) of the problem
- ensure that corrective actions taken have been effective

Improvement

- The laboratory shall continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

Procedure for how documents are to be:

- **Registered**(registration identification,edition number)
- **Updated**(revision, recall or approval)

List of persons involved in these activities, showing their responsibilities and authority.

Training

TURKISH ACCREDITATION AGENCY 25

Records

- **Key word: Traceability**
- *maintain a suitable record system*
- complying with any existing regulations
- "all" original observations, calculations etc.
- for an appropriate period
- sufficient information for permitting repetition of test or calibration
- identification of involved personnel
- safely stored, *held secure and in confidence*

TURKISH ACCREDITATION AGENCY 26

Quality audits

- Regular reviews in order to ensure that the quality system is fully implemented in practice.
- Inspection to ensure that the quality manual and its related documents are being complied with at all levels of the work.
- Audit procedures as described in the quality manual.
- The person responsible for quality is responsible for ensuring that the audits are carried out in accordance with the plan.

TURKISH ACCREDITATION AGENCY 27

Quality system reviews

- Previous revisions
- Visits of accreditation bodies
- Results from internal quality audits
- Complaints
- *Resources - personnel, equipment*
- Training
- Improvement
- Plan for introduction of desired changes
- Future planning
- Documentation
- Archiving

TURKISH ACCREDITATION AGENCY 28

Parameters influencing on the result (5.1.1)

- Human factors
- accommodation (premises) and environment
- test / calibration method
- Equipment
- measurement traceability
- sampling
- the handling of test / calibration items

TURKISH ACCREDITATION AGENCY 29

Personnel

- *Education*
- Training
- No formal requirements
- *Annual assessment*
- Technical knowledge
- Experience

TURKISH ACCREDITATION AGENCY 30

Personnel
Education/Training - verification

- No formal requirements on education
- However, some accreditations are based upon certification personnel
- The competence of the laboratory personnel is verified by the technical assessors (annual assessment)
- The effectiveness of the training actions taken shall be evaluated.
- Sample - test procedure
- It is then the management´s responsibility to verify all other personnel

Personnel -
example, criteria for qualifications
Technical manager of the laboratory

- At least M.Sc. In the technical field
- Five years experience in analytical testing
- Be familiar with all analytical methods of the laboratory
- Be the laboratory´s expert on the following instruments:GC, LC
- Have management experience

Personnel
Assessment of the capability to maintain competence 1/2

Procedures for

- introducing new personnel to the administrative and technical procedures of the laboratory
- training personnel to special jobs
- training personnel to meet present and future demands
- qualifying personnel to work independently

Personnel

Assessment of the capability to maintain competence 2/2

Procedures for

- participation in interlaboratory comparisons
- incorporating feedback from interlaboratory comparisons
- internal quality audits to detect non-compliances due to insufficient training, etc.

Personnel records

- Qualifications
- Training
- Experience

of the technical personnel.

Personnel - In the quality system

- Criteria of competence (qualifications) for different appointments within the laboratory (especially for "key persons")
- Criteria for how to verify that the required competence is attained
- Guarantee competent handling of methods/instruments that are not in regular use

Accommodation and environment
1/2

- Eliminate all environmental factors influencing the results
- Evaluate the environmental conditions in the laboratory
- Protect from abnormal conditions
- Certain activities might require:
  - Screened rooms
  - Stabilised mains voltage
  - Special earthing
  - Protection against vibration and noise
  - Protection against dust and humidity

Accommodation and environment
2/2

- Remaining influence parameters must be considered when calculating the total uncertainty
- Continuous control and monitoring of the environment
- Special requirements for site testing
- Controlled access to all test areas
- Adequate measures shall be taken to ensure good housekeeping

Accommodation and environment
Site testing

- Checks/calibrations of equipment not belonging to the laboratory
- Monitoring of environment is especially important
- Check for transport damages
- Is the test method applicable for site testing?

Accommodation and environment
Access to test areas

- Confidentiality and security
- Protect equipment and test samples, e.g. From contamination
- Locked doors

Methods that can be accredited

All standard and in house methods that ensure a well defined result can beincluded in an accreditation. The uncertainty of
measurement must be determined, and the method must be thoroughly documented, and if possible be verified and confirmed against some other method.

ISO/IEC 17025,
5.5 Equipment

The laboratory shall be furnished with all items of equipment required for correct performance of the tests and measurements.

* Properly maintained
* Labelled or marked
* Recorded

Equipment

- Documentation regarding equipment containing at least the following:
  - Name of the item of equipment
  - Manufacturer's name, type identification and serial number
  - Date received and date placed in service
  - Current location
  - Condition when received (new, used, reconditioned etc.)
  - Details of maintenance carried out
  - History of any damage, malfunction, modification and repair.

TURKISH ACCREDITATION AGENCY 43

Equipment, 3/3

- Calibration.
- Controls between calibrations.
- Procedures for taking defective equipment out of service. Labelling.
- Procedures for determining the effect of defective equipment on previous tests.
- Written instructions on the use of equipment including manuals from the manufacturer.
- Calibration labelling.

TURKISH ACCREDITATION AGENCY 44

Common questions about calibration

WHAT should be calibrated

?

HOW IT should be calibrated

HOW OFTEN IT should be calibrated

TURKISH ACCREDITATION AGENCY 45

Calibration system

- Complete documentation (method, equipment, interval, results) for all calibrations made by the laboratory on its own equipment
- System for marking instruments with regard to calibration status.
- Calibration interval for equipment.
- Traceability to national and international reference standards.
- All reference instruments calibrated at national standards laboratory or accredited laboratory.
- Reference instruments checked before and after calibration.
- Reference instruments shall be used only for calibrations.

TURKISH ACCREDITATION AGENCY 46

Calibration system

- At least following shall be recorded:
  - Description of the equipment with unique identification code
  - Place of calibration
  - Calibration method
  - Reference instrument used
  - Influence parameters
  - Results and corrections
  - Measurement uncertainty
  - The person who has done the calibration
  - The date of calibration

TURKISH ACCREDITATION AGENCY 47

Handling of calibration and test items

- A system for identifying the samples or items to be tested or calibrated
- Documents/marking
- Must be possible to identify sample or item with results of measurements made
- Anonymously handling, e.g. to other clients
- Bonded storage where necessary

TURKISH ACCREDITATION AGENCY 48

Handling of calibration and test items

- Prevent damage, e.g. contamination, corrosion or the application of stresses
- Any relevant instructions shall be observed
- Clear rules for the receipt, retention and disposal of samples or items

Handling of calibration and test items

- Upon receipt, the condition of the calibration or test item, including any abnormalities or departures from standard condition as prescribed in the relevant calibration or test method, shall be recorded
- Where there is any doubt as to the item's suitability for calibration or test, the laboratory shall consult the client for further instruction before proceeding

Test comparisons

To investigate the laboratories´ability to perform tests within their scope of accreditation.

Quality control data shall be analysed and, where they are found to be outside pre-defined criteria,planned action shall be taken to correct the problem and to prevent incorrect results from being reported.

Certificates and reports
Contents

- Headings shall be standardised as far as possible
- Corrections or additions after issue: "*Amendment/Addendum to test report serial number* ... (or as otherwise identified)"
- Shall not include any advice or recommendation arising from the results
- Any extrapolation of results from statistically selected test objects to the properties of a lot, batch or production quantity shall be contained in a separate document.

TURKISH ACCREDITATION AGENCY 52

Certificates and reports
Contents

- Results from not accredited test method may be included in the test report. However, they shall cover only the minority of the report and shall be indicated.
- Results from subcontractors may be included. However, the shall cover only the minority of the report and shall be indicated.

TURKISH ACCREDITATION AGENCY 53

Certificates and reports
Contents

- The certificate or report shall include characterisation and condition of the calibration or test item.
- If clients require transmission of results by facsimile, staff shall follow documented procedures.

TURKISH ACCREDITATION AGENCY 54

Guidance Documents In Laboratory Accreditation

- EA Guidelines
  - **EA-4/02 Expressions of the Uncertainty of Measurements in Calibration**
  - **EA-4/07 Traceability of Measuring and Test Equipment to National Standards**
  - **EA-4/09 Accreditation for Sensory Testing Laboratories**
  - **EA-4/10*Accreditation for Laboratories Performing* Microbiological Testing**
  - **EA-4/14 Selection and Use of Reference Materials**
  - **EA-4/15 Accreditation for Bodies Performing non-Destructive Testing**
  - **EA-4/16 EA Guidelines on the Expression of Uncertainty in Quantitative testing**

Guidance Documents In Laboratory Accreditation

- ILAC Guidelines
  - **ILAC G7:1996 Accreditation Requirements and Operating Criteria for Horseracing Laboratories**
  - **ILAC G8:1996 Guidelines on Assessment and Reporting of Compliance with Specification**
  - **ILAC G9:2005 Guidelines for the Selection and Use of Reference Materials**
  - **ILAC G12:2000 Guidelines for the Requirements for the Competence of Reference Materials Producers**
  - **ILAC G13:2000 Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes**
  - **ILAC G17:2002 Introducing the Concept of Uncertainty of Measurement in Testing in Association with the Application of the Standard ISO/IEC 17025**
  - **ILAC G18:2002 The Scope of Accreditation and Consideration of Methods and Criteria for the Assessment of the Scope in Testing**
  - ***ILAC G19:2002 Guidelines for Forensic Science Laboratories***
  - **ILAC G20:2002 Guidelines on Grading of Non-conformities**
  - **ILAC G22:2004 Use of Proficiency Testing as a Tool for Accreditation in Testing**

TÜBİTAK
MAM

UNIDO International Training Programme on
Food Laboratory Management and Practice
16–20 November 2009

LECTURE 2

GOOD LABORATORY PRACTICES

Implementation of the Principles of Good Laboratory Practice

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Outline

- What is GLP? How has it developed?
- The role of the Member States
- The Role of the European Commission

GLP

- GLP: Good Laboratory Practice
- OECD Principles on Good Laboratory Practice
- What is OECD?

The Organization for Economic Cooperation and Development: OECD

- Intergovernmental Organization
- 30 industrialized countries
- Meet to coordinate and harmonize policies
- Discuss issues of mutual concern
- Work together to respond to international problems

OECD Member Countries

Australia	Hungary	Poland
Austria	Iceland	Portugal
Belgium	Ireland	Slovak Republic
Canada	Italy	Spain
Czech Republic	Japan	Sweden
Denmark	Korea	Switzerland
Finland	Luxembourg	Turkey
France	Mexico	UK
Germany	Netherlands	USA
Greece	New Zealand	Norway

Good Laboratory Practice

Definition

- **Quality system** concerned with the organisational process and the conditions under which **non clinical** health and environmental **safety studies** are planned, performed, monitored, recorded, archived and reported.

GLP – Detailed Objectives

- Assurance of quality and validity of test data
- Avoidance of duplicative testing
- Animal welfare
- Time and resource efficiency
- Avoidance of non-tariff barriers to trade

GLP - History

- **1976 : GLP regulation of FDA on non-clinical laboratory studies**
- 1978 : OECD establishes expert group
- 1981 : OECD Council Decision on Mutual Acceptance of Data (MAD) GLP Principles [illegible] Annex [illegible]
- [illegible]
- 1987/88 : EU adopts GLP Directives
- [illegible]
  - [illegible] Procedures for GLP
  - [illegible] Laboratory Inspections and Study Audits
- [illegible] REVISED GUIDANCE FOR GLP MONITORING AUTHORITIES FOR COMPLIANCE MONITORING PROCEDURES FOR GOOD LABORATORY PRACTICE (GUIDES) + New expert group on GLP - Principles
- 1997 : Adoption of Revised GLP Principles
- 1999 : Amendment of EU GLP Directives following OECD
- 2004 : EU GLP Directives codified

Scope of GLP in the EU

	Directives	
Chemical Substances	92/32	
Existing chemicals	Regulation 793/93/EC	
Dangerous preparations	99/45	
Medicinal Products	2001/83	2003/63
Veterinary Medicinal Products	2001/82	
Animal Feed Additives	87/153	94/40
Foodstuffs	89/397	93/99
Pesticides, Biocides	91/414	98/8
Cosmetics	76/768	93/35

Types of research under GLP

- Human health and environmental safety studies,
- carried out for regulatory purposes:
  - toxicity studies
  - mutagenicity studies
  - ecotoxicity studies
  - environmental fate and bioaccumulation studies
  - analytical and clinical chemistry
  - pharmaco– and toxicokinetics
  - physical and chemical properties
  - residue studies
  - studies on effects on mesocosms/ecosystems

GLP in the EC - Legal Framework

- Directive 2004/10/EC
  - on the harmonisation of laws, regulations and administrative provisions relating to the application of the principles of GLP
- Directive 2004/9/EC
  - on the inspection and verification of GLP

GLP: Role of the Member States

Directive 2004/10/EC, art. 1

"Member States shall take all measures necessary to ensure that laboratories carrying out safety studies on chemical products..... comply with the principles of good laboratory practice (GLP).....".

Directive 2004/10/EC replaces Directive 87/18/EEC.

GLP: Role of the Member States

- Article 3.1 of Directive 2004/10/EC:
- Member States shall adopt the measures necessary for verification of compliance with the principles of good laboratory practice. These measures shall include, in particular, inspections and study checks in accordance with the recommendations of the OECD in this area.

EU GLP Monitoring Directives

Dir 2004/9/EC

Article 3.1: "Member States shall designate the authorities responsible for the inspection of laboratories within their territories and for the audit of studies carried out by laboratories to assess compliance with GLP."

The Directive also comprises reporting and internal market (= mutual acceptance of data) requirements.

Furthermore, it requires that the OECD Revised Guides for Compliance Monitoring Procedures for GLP and the OECD Guidance for the Conduct of Test Facility Inspections and Study Audits must be followed during laboratory inspections and study audits.

Directive 2004/9/EC replaces Directive 88/320/EEC as of 11 March 2004.

Monitoring Authorities (EU, 1)

Country	Starting year	No. of inspectors
Austria	[illegible]	2
Belgium	1989	3
Bulgaria	-	-
Cyprus	-	-
Czech Republic	1997/2000	1
Denmark	1980	4
Estonia	-	-
Finland	1990	3
France	1984/1985/1989	6
Germany	1981	15

Monitoring Authorities (EU, 2)

	Country	Starting year	No. of inspectors
	Greece	[illegible]	4
	[illegible]	[illegible]	2
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	25
	Latvia		
	Lithuania		
	Luxembourg		-
	Malta	-	-
	Netherlands	[illegible]	3
EEA	Norway	[illegible]	[illegible]

Monitoring Authorities (EU, 3)

	Country	Starting year	No. of inspectors
	Poland	2003	[illegible]
	Portugal	[illegible]	2
	Romania		
	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
	Spain	[illegible]	[illegible]
	Sweden	[illegible]	3
EEA	Switzerland	1981/1985	[illegible]
	United Kingdom	[illegible]	5

GLP : Role of the Member States

- Practical implementation of GLP Principles and compliance monitoring:
  - adoption of necessary legal measures
  - creation of national compliance programmes
  - inspections and study audits
  - annual reports

States

- Dir 2004/9/EC, Article 4:

  Each year, Member States shall draw up a report relating to the implementation of GLP within their territory. This report shall contain a list of laboratories inspected, the date on which such inspection was carried out and a brief summary of the conclusions of the inspections.

The reports shall be forwarded to the Commission each year, not later than 31 March.

States

- Dir 2004/9/EC. Article 5:
- 1. Without prejudice to Article 6, the results of laboratory inspections and study audits on GLP compliance carried out by a Member State shall be binding on the other Member States.
  2. Where a Member State considers that a laboratory within its territory claiming GLP compliance does not in fact comply with GLP to the extent that the integrity or authenticity of the studies it performs might be compromised, it shall forthwith inform the Commission. The Commission shall inform the other Member States.

States

- Dir 2004/9/EC, Article 6:
-

1. Where a Member State has sufficient reason to believe that a laboratory in another Member State claiming GLP compliance has not carried out a test according to GLP, it may request further information from that Member State and in particular may request a study audit, possibly in conjunction with a new inspection.

Exchange of information - Annual Reports

To be sent each year before April 1 to *European Commission*

Normally copied to all other Monitoring Authorities

Content:
- Identification of test facilities inspected
- Dates of inspections and decisions
- Nature of inspections
- Areas of expertise of facilities inspected
- Compliance status

GLP in the EU - Practicalities

- Role of the Commission inside the EU:
  - ensure uniform application of the GLP principles and compliance monitoring in all Member States
  - facilitate acceptance of data among MS

EU

- What we do to achieve this:
- Regular meetings of Member State experts in the Working Group on Good Laboratory Practice
- Management of lists of inspected laboratories
- (CIRCA website)
- Build up contacts between receiving authorities and monitoring authorities (e.g. EMEA)
- Specific exercises for confidence building: evaluation visits

EU GLP Working Party

1-2 meetings/year

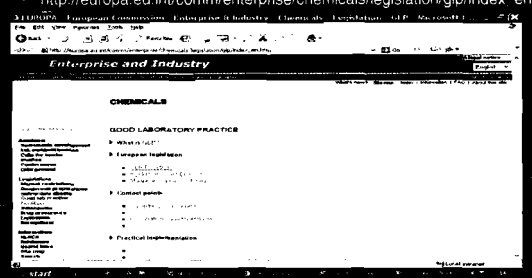
Implementation of GLP Directives in Member States

MRA Negotiations

Mutual Joint Visit Programme

The GLP Web Site

- http://europa.eu.int/comm/enterprise/chemicals/legislation/glp/index_en.ht



GLP in the EU - Foreign Relations

- Role of the Commission outside the EU:
  - correct application of MAD (Mutual Acceptance of Data) Decision between EU and other OECD countries
  - acceptance of data through formal agreements on Mutual Recognition (MRA)

    (Japan, Switzerland, Israel)

EU and its non-OECD

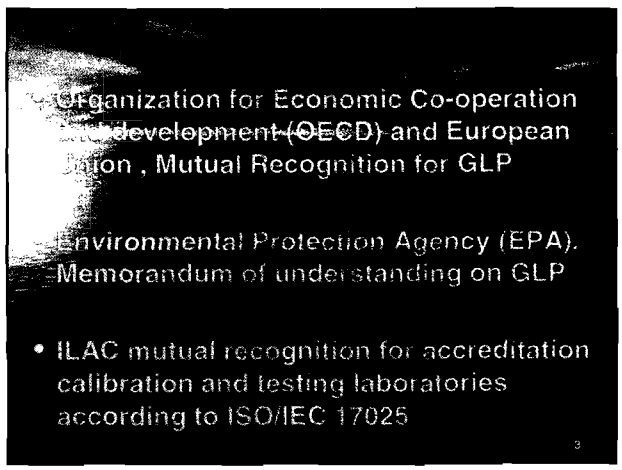
- http://europa.eu.int/comm/enterprise/chemicals/legislation/glp/index.htm

Thank you for your attention

Differences between GLP and ISO 17 025

GLP	ISO 17 025
Objective:	Objective:
Pr (people, animals, environment).	
for regulated area (chemicals, pesticides, human and veterinary medicines, biocides, food and feed additives, cosmetics)	(consumer requirement, etc)

2



EU

Dire

Pa

on the harmonization of laws, regulations and administrative provisions relating to the application of the GLP Principles and the verification of their applications for tests on chemical substances.

4

EU

Di

the

on the inspection and verification of good laboratory practice.

Section 1 : Physical-Chemical Properties
Section 2 : Effect on Biotic Systems
Section 3 : Degradation and Accumulation
Section 4 : Health Effect Methods

5

What

Good Laboratory Practice is a quality system which concerns to the organizational process and the conditions under which non-clinical health and environmental safety studies are planned, performed, monitored, recorded, archived and reported.

The OECD Principles of GLP set out managerial concepts covering the organization of test facilities and the conditions under which non-clinical safety studies are performed. Their purpose is to ensure the generation of high quality and reliable test data (in vitro and in vivo) related to the safety of chemicals and preparations in the framework of the Mutual Acceptance of Data (MAD).

6

What

The International Standard contains all of the requirements that testing and calibration laboratories have to meet if they wish to demonstrate that they operate a quality system are technically competent and are able to generate technically valid results.

The use of International Standard should facilitate cooperation between laboratories and other bodies to assist in the exchange of information and experience, and in the harmonization of standards and procedures

7

The main difference between a laboratory accredited to ISO/IEC 17025 and a test facility working according to the Principles of GLP is the purpose of testing that the laboratories deal with.

GLP studies are:

- Pre-determined experiments agreed upon by the sponsor before commencing the work.

ISO/IEC 17025 accredited tests are:

- Generally short term/immediate test result.

8

Comparison of Schemes

EU

Management
Requirements
4.1 Organization
4.2 Management system

(Technical Management
Quality Manager)

- Test Facility Organization and Personnel
- Quality Assurance Programme
- Study Director's Responsibilities
- Principles of Good Laboratory Practice (GLP)

9

Comparison of Schemes

EN	
Management system	2. Quality Assurance Program
4.14 Internal audits	2.1 General 2.2 Responsibilities of the Quality Assurance Personnel (Well defined Quality Assurance program)

10

Comparison of Schemes

EN	
4.3 Document Control 4.3.1 General 4.3.2 Document approval and issue 4.3.3 Document changes (Established and maintain procedures to control all documents.)	Documented Master schedule for all studies, study plans, and relevant documented SOP's for each activity (Documents, records and its control is in every point of GLP Principles)

11

Comparison of Schemes

EN	
All obsolete documents should be removed. Test results should be archived for *x* years.	Defined person, *procedure* and space for archiving all documents and test items relevant to the study for *x* years.

12

Comparison of Schemes

4.3.3 Document changes

EN	
efined in section 4.3.3.	Planned amendments to the study plan should be documented and signed by the study director. Non planned deviations should be maintained with the study
The laboratory should define how changes are made in documents.	

Comparison of Schemes

E	
EN	
eview of requests nders and contracts	(Not required by GLP)
(Necessary for test and calibration)	

Comparison of Schemes

E	
EN	
ubcontracting of sts and calibrations	(Not required by GLP)
(Defined requirements in the standard)	

Comparison of Schemes

EN	
Purchasing services and supplies	(Compliance with laboratory suppliers.)
(Defined requirements in the standard)	

Comparison of Schemes

EN	
Purchasing services and supplies	(Interaction with the sponsor prior to commencing the study to sign the study plan.)
(Defined requirements in the standard)	

Comparison of Schemes

EN	
Complaints (Laboratory should have a policy and procedure for the resolution of complaints received from customers or the other parties.)	(Not applicable.)

Comparison of Schemes

EN	
Control of nonconforming testing and/or calibration work (On-going quality control.)	(Part of the QAU on-going checks reviewed regularly and internal audits)

Comparison of Schemes

4.12 Improvement Continually improve the effectiveness of the management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review	(Not required)

Comparison of Schemes

EN	
4.11 Corrective action 4.11.1 General 4.11.2 Cause analysis 4.11.3 Selection and implementation of corrective actions 4.11.4 Monitoring of corrective actions 4.11.5 Additional audits	(Defined as amendments to the study)

Comparison of Schemes

Preventive action Needed improvements and potential sources of nonconformities either technical or concerning the management system shall be identified	(Not required.)

Comparison of Schemes

4.13 Control of records 4.13.1 General 4.13.2 Technical records (Original observations (raw data), amending records and computer filing.)	(Ensure the maintenance of the historical file of all the procedures.)

Comparison of Schemes

Internal audits The laboratory shall periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to fulfill the requirements of the management system (this ISO)	

Comparison of Schemes

ISO

4.5 Management reviews

4.5.1 The laboratory's top management shall conduct a review of the laboratory's management system(s), testing and/or calibration activities ...

Comparison of Schemes

ISO

5.4 Technical requirements

5.4.1 General

... factors which control the accuracy and reliability of the ... The extent to which the factors contribute to the total uncertainty of measurement differs between types of test ... and between types of calibration ...

... Uncertainty values are not required ...

Comparison of Schemes

ISO

6.2 Personnel

6.2.1 Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required ...

GLP

1. Test Facility Organization and Personnel

1.1, 1.2, 1.3, 1.4 Management, Study Director, Principal Investigator, Study Personnel

... Current training, knowledgeable in the Principles of GLP ...

Comparison of Schemes

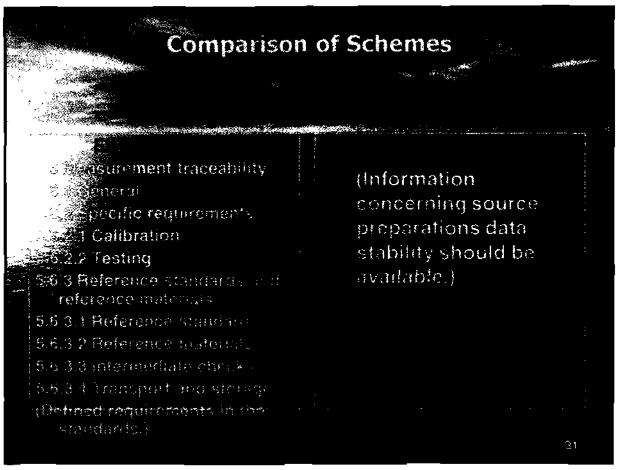
[illegible]	
[illegible] Accommodation and environmental conditions (Sufficient to facilitate correct performance of the tests.)	(Avoid cross contamination; extra emphasis for studies which involve animals.)

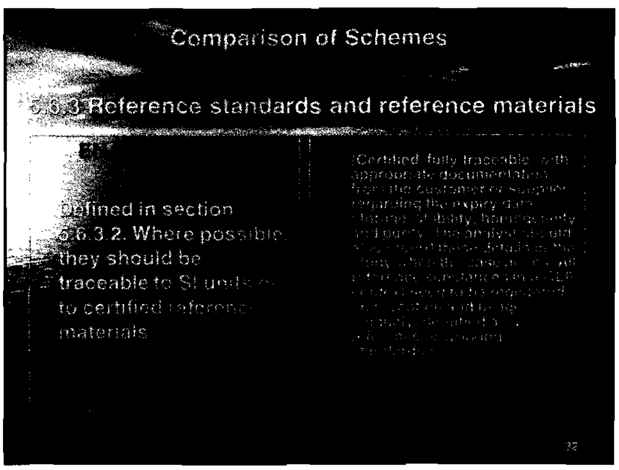
Comparison of Schemes

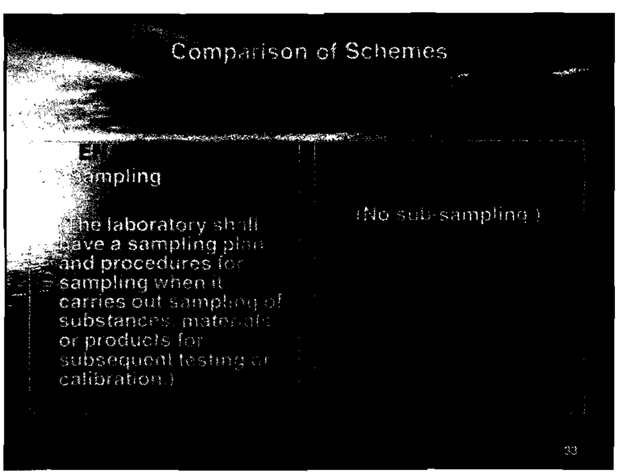
[illegible]	
5.4 Test and calibration methods and method validation 5.4.1 General 5.4.2 Selection of methods 5.4.3 Laboratory-developed methods 5.4.4 Non-standard methods 5.4.5 Validation of methods 5.4.6 Estimation of uncertainty of measurement 5.4.7 Control of data	(All methods have to be validated prior to use in a study.)

Comparison of Schemes

[illegible]	
5.5 Equipment (Defined requirements in the standards.)	(Apparatus used in a study should be periodically inspected, cleaned, maintained and calibrated according to the Standard Operating Procedures.)


Comparison of Schemes
Calibration
Testing
5.6.3.1 Reference standards
(Information concerning source preparations data stability should be available.)
31


Comparison of Schemes
Reference standards and reference materials
they should be
materials
32


Comparison of Schemes
and procedures for
sampling when it
or products for
calibration.)
(No sub-sampling.)
33

Comparison of Schemes

5.8 Handling of test and calibration items The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of test and/or calibration items	(Chain of custody, test of stability throughout the study, effect of storage conditions etc.)

34

Comparison of Schemes

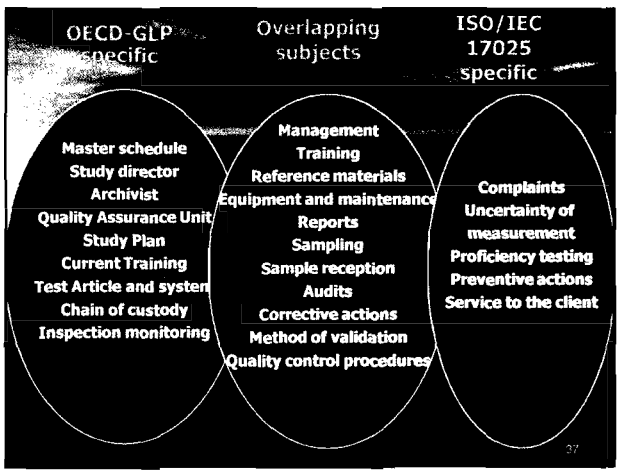
5.9 Assuring the quality of test and calibration The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken	(On-going predetermined and ad hoc quality checks of the test item, study and facility.)

35

Comparison of Schemes

5.10 Reporting the results General Test reports and calibration certificates Test reports Calibration certificates Opinions and interpretations Testing and calibration results obtained from subcontractors Electronic transmission of results Format of reports and certificates Amendments to test reports and calibration certificates	(Identified in GLP.)

36


OECD-GLP specific
Overlapping subjects
ISO/IEC 17025 specific
Master schedule
Study director
Archivist
Quality Assurance Unit
Study Plan
Current Training
Test Article and system
Chain of custody
Inspection monitoring
Management
Training
Reference materials
Equipment and maintenance
Reports
Sampling
Sample reception
Audits
Corrective actions
Method of validation
Quality control procedures
Complaints
Uncertainty of measurement
Proficiency testing
Preventive actions
Service to the client
37


THANK YOU
38

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LECTURE 3

FOOD TESTING LABORATORY ACCREDITATION (GENERAL PRINCIPLES, ISO17025:2005 REQUIREMENTS)

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Food Testing Laboratory Accreditation – ISO17025:2005 Requirements

Hayrettin ÖZER

UNIDO – November 2009

Overview

- Background
- What is Accreditation
- ISO/IEC 17025
- Management requirements
- Technical requirements

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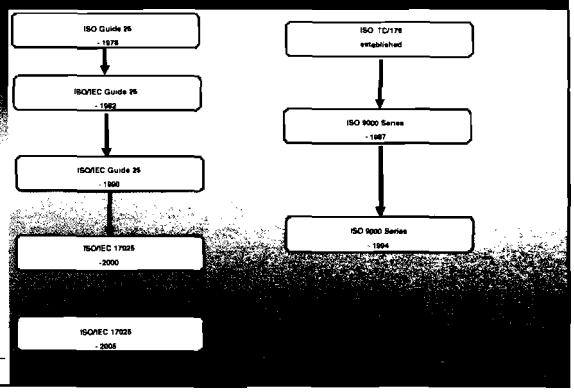
BACKGROUND



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What is Accreditation

- ISO/IEC 17025 contains the criteria necessary for a laboratory to implement in order for it to perform its test work competently
- Accreditation is independent and formal recognition of the competence a laboratory to perform specific tests etc.
- **The criteria that a laboratory must comply with to be internationally acceptable is ISO/IEC**

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ISO/IEC 17025

1. **These are the base requirements necessary for a laboratory to be able to implement and operate to demonstrate it is competence**
   - Cost (of compliance and/or Accreditation) is cost of achieving consistently good results
   - Cost of not having consistently good results could be much higher
2. **17025 focus is on:**
   - Management requirements
   - Technical requirements

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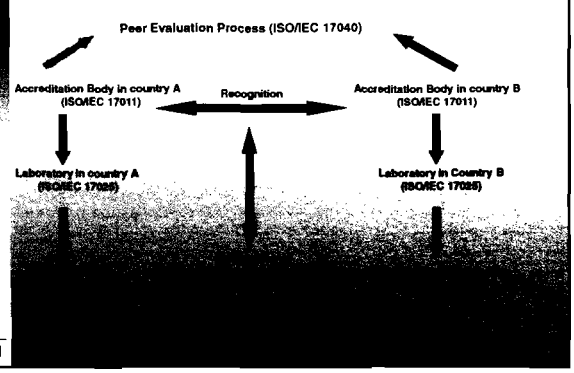
ILAC MLA (Mutual Recognition Arrangement)



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Recognition and Trade

It is generally agreed by the WTO that the biggest obstacle to trade is Non Tariff Technical Barriers

It is in the interest of all to overcome these and to develop a means of recognizing the technical infrastructure and output from one

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ISO/IEC 17025 – Some issues

Scope:

- Includes sampling
  It covers standard methods, non standard methods and laboratory developed methods
- Includes 1st, 2nd and 3rd Party laboratories
- NOT INTENDED TO BE USED AS A BASIS FOR CERTIFICATION OF LABORATORIES
  - Causes confusion if not adhered to
  - Regulators may specify additional requirements unique to their needs
  - Must principles of ISO 9001

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ISO/IEC 17025 – Some issues

4.1 Management requirements

- Legal entity
- Conflict of interest defined and relationship documented (Part of larger org.)
- Impartiality (4.1.5 b) and freedom from commercial/financial pressures which might influence judgement for 3rd party activities (Note 2)
- Have policies in place to avoid involvement in activities that would negatively affect confidence in its competence, impartiality, judgement or operational integrity

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ISO/IEC 17025 – Some issues

4.2 Management System

- *Must have implemented a MS appropriate for its scope and activities* (documented to extent necessary to assure quality of result) Understood by all in org. and communicated to all (4.2.2 d)
- System must be documented 4.2.2
- Top management must demonstrate commitment

4.3 Document control

- Approval and issue – Reviewed and approved (uniquely identified)
- Document changes – by original function. Altered text highlighted

ISO/IEC 17025 – Some issues

4.4 Review of requests, tenders and contracts

- Have procedures in place for this (Records of review must be available)
- *Have necessary resources*
- Differences resolved before acceptance
- Can be oral or documented (contract) (Note)
- For review of simple or routine results (date and initials of person doing test sufficient) – Note in 4.2.2
- Repetitive tests – Review at initial stage only as long as unchanged

4.5 Subcontracting

- Use of a competent subcontractor (could be one that complies with ISO/IEC [illegible]

ISO/IEC 17025 – Some issues

4.7 Customer service

- The laboratory shall obtain feedback from customers and analyze for possible improvement.

4.8 Complaints

4.9 Control of nonconforming testing/calibration work

- Policies – procedures implemented shall include
- Responsibilities and authority
- Evaluation
- [illegible]

ISO/IEC 17025 – Some issues

4.10 Improvement
- Shall continually improve effectiveness of MS through analysis

4.11 Corrective action
- *Shall designate and give authority for implementing Corrective Action*
- Shall start with a cause analysis (analysis of all potential causes)
- Identify all potential corrective actions (select and implement)
- Shall monitor corrective actions effectiveness

4.12 Preventative Actions
- Needed improvements and potential sources of non-conformities (NC) shall be addressed

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ISO/IEC 17025 – Some issues

4.13 Control of Records
- Readily retrievable
- Protect and back up needed

4.14 Internal audits
- Done periodical – All elements of system
- Done by trained and qualified staff where ever permitted by persons independent of activities to be audited

4.15 Management Review
- Periodic, at least once per year

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ISO/IEC Technical Requirements

5.2 Personnel
- Competent qualified appropriately on basis of education, training, experience and demonstrated skills
- Persons who give opinions and interpretations of test results should have additional qualifications
- Policy to identify training needs
- Effectiveness of training shall be calculated
- Authorize specific staff for specific work

5.3 Accommodation and Environment
- Appropriate and Adequate

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ISO/IEC Technical Requirements

5.4 Test and calibration methods and validation

- *Instructions on use of all equipment and handling of items*
- Deviation from methods documented and justified authorized and accepted by customer
- Standards do not need to be supplemented/ rewritten
- Selection of method – meets customer needs and appropriate
- Can use international, regional, national or published by reportable technical organizations/journal or specified by manufacturer
- Laboratory developed methods
  - when necessary, approved by customer and validated
- Non standard test methods validated

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ISO/IEC Technical Requirements

5.4 Test and calibration methods and validation (continued)

- Validation of Methods
  Defines validation as "Particular requirements for a specific intended use are fulfilled"
- Laboratories validate non standard methods, laboratories developed, standard methods used outside intended scope.
  - Record validation
- Estimation of Uncertainty
  - Procedure to estimate Uncertainty of Measurement (UOM)
  - Where methods includes and defines UOM then it is established (ISO 5725 Guide to expression of UOM)
- Control of data

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ISO/IEC Technical Requirements

5.5 Equipment

- Needs all equipment for tests. Allows use of outside equipment
- Calibration schedules
- Operated by authorized persons
- Maintenance and storage
- Equipment labelled
- Safeguarded from adjustments which could invalidate it

5.6 Measurement traceability

- Calibrated before use
- Calibration traceable to SI System

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ISO/IEC Technical Requirements

5.7 Measurement traceability

- "A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to this International Standard, for the *calibration concerned is sufficient* evidence of traceability of the calibration data reported."
- Testing laboratories
- Reference Standards and Reference Materials
  Calibration of Ref. Standards – traceability (only used for this purpose)

  Ref. Materials traceable to SI Units if possible or CRM
  Intermediate checks

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ISO/IEC Technical Requirements

5.8 Handling of test and calibration items

- Procedures for transport, receipt, handling
- Protection, storage, Disposal
- Identifying test items

5.9 Assuring the quality of test and calibration results procedures for monitoring tha validity of tests and calibrations

5.10 Reporting of results

- Accurately and clearly unambiguously and objectively

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ISO/IEC Technical Requirements

Test reports and calibration certificates

- Identifies all information needed
- Where applicable UOM for best reports
- UOM for calibration certificates
- *Evidence that measurements are traceable* (calibration)
- When statement of compliances is made UOM shall

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ISO/IEC Technical Requirements

Opinions and Interpretation

- Document basis upon which interpretation is used
- Clearly marked as such
- Not confused with inspections and Product certification
- Subcontracted results clearly identified
- Calibration Certification issued to the contracting laboratory

Reference

- ISO 17025:2005, "General requirements for the competence *of testing and calibration laboratories*"
- S.M. Curtain, 2007, "presentation on ISO 17025:2005", CASCO/DEVCO workshop, Tunisia

Thank you for your attention !

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LECTURE 4

QUALITY CONTROL IN FOOD TESTING LABORATORIES, REFERENCE MATERIALS AND PROFICIENCY TESTING

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Quality Control in Food Testing Laboratories, Reference Materials and Proficiency Testing

Nihat ÖZCAN
17.11.2009

Outline

- Quality: Terms & Definitions
  - Quality Assurance
- Quality and Quality Control
  - Quality Control System
  - *Quality Control Characteristics*
  - Quality Control of tests
  - Quality Control strategies
  - Quality Control Methods
- Reference Material & Certified Reference Material
  - Definitions
  - Requirements of RM & CRM
  - Choice of CRM
  - Testing & Interlaboratory Comparision

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Quality: Terms & Definitions

- Quality(Q):Degree to which a set of inherent characteristics fulfils requirements
- Quality Assurance(QA):Describes the overall measures that laboratory uses to ensure the quality of its operation
- Quality Contol(QC) Describes the individual measures which relate to the monitoring and control of analytical operations

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Quality: Terms & Definitions

Typically QA might include;

- A quality system
- Suitable laboratory environment
- Educated, trained and skilled staff
- Training procedures and records
- Equipment suitably maintained and calibrated
- ***Quality control procedures***
- Documented and validated methods
- Traceability and measurement uncertainty
- Checking and reporting procedures
- Preventive and corrective actions

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Quality and Quality Control

Quality Control: The operational techniques and activities that are used to fulfil requirements for quality.

Quality control of analytical methods; provides the information needed to ensure that procedures, equipment, and personnel are performing at the levels of precision and accuracy required by the intended use of the data.

"The laboratory shall have quality procedures for [illegible]"

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Quality and Quality Control

Quality Control Characteristics

Quality control system is established by laboratory management to improve the quality of its results.

The system provides:

- Early warning to analysts when methods or equipment begin to develop a bias or show deterioration of precision;
- The protection and retrievability of data (results); traceability and control of samples as they are processed [illegible]

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Quality and Quality Control

- Involve the operators or analysts who actually perform the work to the greatest possible extent.
- Use the simplest, most direct statistical procedures that will provide the necessary degree of control. This means that graphical or simplified arithmetic procedures are preferred.
- **Perform the quality control measurements as early in the measurement process as possible. This prevents waste of analytical effort if the method is not initially in**

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Quality and Quality Control

- Provide specific action limits and describe exactly what must be done when these limits are exceeded.
- For each method (for each sample type), choose a control material that is;
  - Stable and homogeneous
  - measured values within the range of interest
  - **Where possible, the control material should be similar to the samples to be analyzed.**
- **In situations where satisfactory control material cannot**

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Quality and Quality Control

- Give analysts specific instructions concerning their response to an out-of-control condition.
- Provide for a periodic in-depth review by supervision and management of the overall effectiveness of the laboratory quality control system.

  Operating experience may indicate that
  - **methods should be added to, or dropped from the program,**
  - **That the frequency of specific control samples should**

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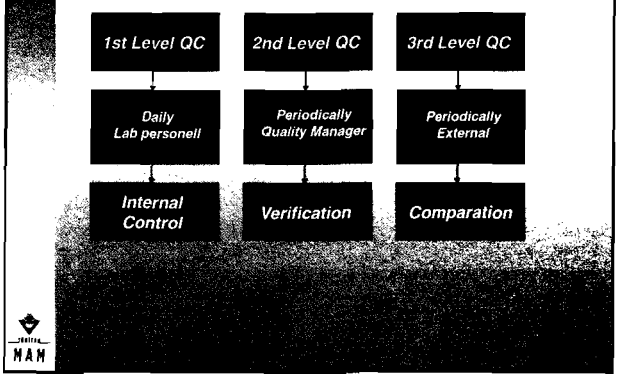
Quality and Quality Control



Quality and Quality Control

1st Level QC

- Blank
- Calibration Standarts
- Control Samples
- Replicate Analysis
- Standart addition Methods, internal standart
- Spiked samples

Quality and Quality Control

2nd Level QC

- Blind samples
- Reference material

3rd Level QC

- Interlaboratory comparison
- Proficiency Tests

Quality and Quality Control

1st Level QC

- Blank

  The use of various types of blanks enable the analyst to ensure that calculations made for the analyte can be *suitably corrected to remove any contributions to the* response which are not attributable to the analyte

- Calibration Standarts

  Standards and chemical calibrants placed at intervals in an analytical batch enable checks to be made that the response of the analytical process to the analyte is stable.

- Replicate Analysis

  [illegible]

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Quality and Quality Control

2nd Level QC

- Blind samples

  Blind analysis is effectively a form of repeat analysis and provides a means of checking precision. It *consists of replicated test portions* placed in the analytical batch, possibly by the laboratory supervisor, and is so-called because the analyst is not normally aware of the identity of the test portions or that they are replicates. Thus the analyst has no preconceived ideas that the particular results should be [illegible]

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Quality and Quality Control

Laboratory management should set and justify an appropriate level of quality control, based on; risk assessment, reliability of the method, the criticality of the work, and the feasibility of repeating the analysis if it doesn't work correctly.

For routine analysis, a level of internal QC of 5% has been identified as reasonable, *i.e.*, 1 in every 20 samples analysed should be a QC sample.

For robust, routine methods lower level of QC may be reasonable

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Quality and Quality Control

The $\bar{X}$ - and *R*-chart method

The control sample is run two or more times during the run, batch, or shift. The average is plotted on the $\bar{X}$ -chart and the absolute value of the difference between the high and low values, the range, is plotted on the *R*-chart. If the average falls between the upper and lower control limits and the range falls below the upper control limit, the process is considered to be in control.

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Quality and Quality Control

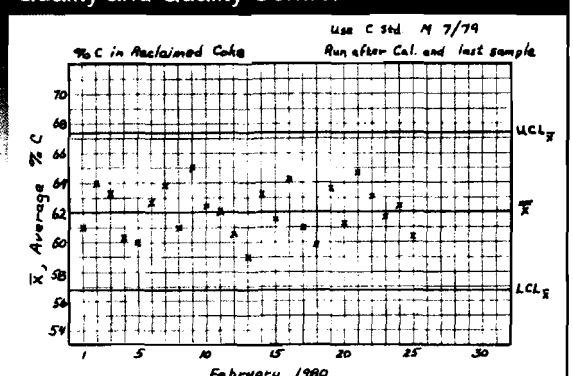

FIG. 1 Control Chart for Averages

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Quality and Quality Control

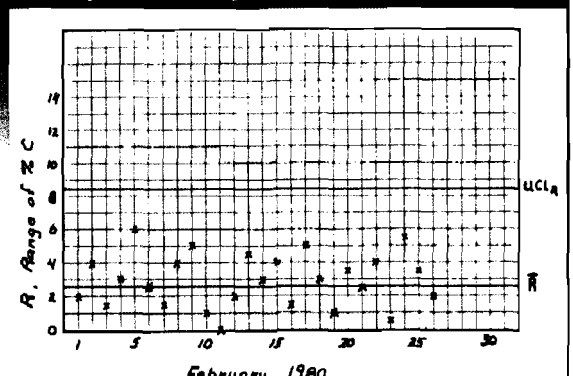

FIG. 2 Control Chart for Ranges

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Quality and Quality Control

The *X*-chart method (control chart for individuals)
useful for measurements that are made on a frequent or continual basis and methods or instruments for which the usual mode of failure produces relatively large shifts in results.

It is sensitive to sudden and relatively large changes in the analytical process
but it is not as sensitive to small changes as the $\bar{X}$ - and *R*-chart method.

Each time the control material is analyzed, its value is

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Quality and Quality Control

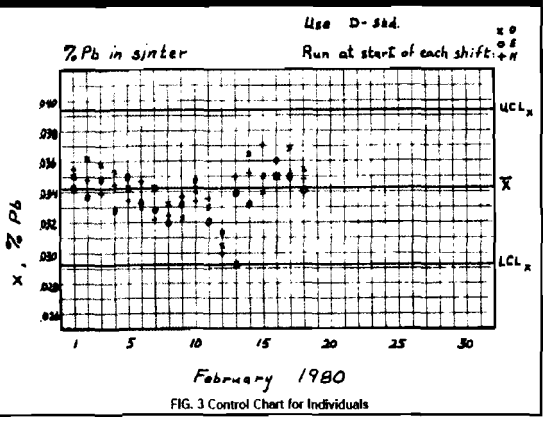

FIG. 3 Control Chart for Individuals

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Quality and Quality Control

A combination of this two methods constitutes a useful strategy. A fixed number of control sample runs are made during a period that samples are being analyzed (such period could, for example, be a shift or a day in a continuous analysis process). Each individual value is plotted on the *X*-chart as the measurement is completed. Their average value and range are plotted on the $\bar{X}$ - and *R*-charts.

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Quality and Quality Control

Comparison with certified reference materials (CRMs)

this method is frequently the only strategy that can be employed for infrequently used analytical methods or for nonroutine sample types. If an CRM is run along with the samples, comparison of the measured value against the known value of the standard provides a measure of confidence in the sample assays. Lacking an CRM, any previously analyzed material may be used.

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Reference Material & Certified Reference Material

Reference Materials(RM)

A material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

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Reference Material & Certified Reference Material

Certified Reference Material (CRM)

a reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

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Reference Material & Certified Reference Material

Requirements of RM & CRM

A CRM must be suitable for the exacting role it performs in storing and transferring information on measured property values. The following technical criteria apply to the fitness-for-purpose of CRMs in general:

- *the CRM itself and the property value(s) embodied in it* should be stable for an acceptable time-span, under realistic conditions of storage, transport and use;
- **the CRM should be sufficiently homogeneous that the property value(s) measured on one portion of the batch should apply to any other portion of the batch within**

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Reference Material & Certified Reference Material

- the property value(s) of the CRM should have been established with an uncertainty sufficient to the end use(s) of CRM;
- clear documentation concerning the CRM and its established property value(s) should be available. The property value(s) should have been certified, so **the documentation should include a certificate**

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Reference Material & Certified Reference Material

Uses of Certified Reference Materials

One laboratory

The test procedure constitutes a check of precision and/or trueness of a measurement method as applied by one particular laboratory. The laboratory uses a CRM to check its measurement process, for any particular reason, at any time.

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Reference Material & Certified Reference Material

Uses of Certified Reference Materials
Interlaboratory programme
In this case the test procedure is performed by a number of laboratories as part of an organized programme. The purpose of this programme is to establish the performance characteristics of a measurement method, against which a typical laboratory can compare its own performance.

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Reference Material & Certified Reference Material

Choice of CRM
Relevance to the measurement procedure
The user of the CRM must decide what properties of the CRM are relevant to his measurement procedure, taking into account the method of certification, the statement on intended use and instructions for the correct use of the CRM on the certificate.

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Reference Material & Certified Reference Material

- **Level.** The CRM should have properties at the level appropriate to the level at which the measurement process is intended to be used, e.g. concentration.
- **Matrix.** The CRM should have a matrix as close as possible to the matrix of the material to be subjected to the measurement process, e.g. carbon in low-alloy steel or carbon in stainless steel.

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Reference Material & Certified Reference Material

- **Form.** The CRM may be in any physical state and form, e.g. solid, gas, etc. It may be a test piece or *amanufactured article* or a *powder.* It *may need* preparation. It shall be used in the same form as the sample to be measured.

- **Quantity.** The quantity of the CRM shall be sufficient for the entire experimental programme, including some reserve if it is considered necessary. Avoid having to obtain additional new batches of the CRM later in a given measuring process.

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Reference Material & Certified Reference Material

- **Stability.** Wherever possible the CRM should have stable properties throughout the experiment. Three situations can exist:
  - the properties are stable and no precaution is necessary;
  - the certified value of the properties may be influenced by storage conditions, in which case the container should be stored, both before and after its opening, in the way described on the certificate;

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Proficiency Testing & Interlaboratory Comparision

Interlaboratory Comparision

Interlaboratory comparisons mean organization, performance and evaluation of tests on the same or similar items by two or more laboratories in accordance with predetermined conditions.

Comparasions are organised at all scientific levels, but the objectives, protocols and participants vary. In certification trials, the measurements are used to assign values to reference materials.

In method validation studies (collaborative trials),

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Proficiency Testing & Interlaboratory Comparision

Proficiency Testing(PT)

PT schemes also known as external quality assessment (EQA) schemes or laboratory performance studies are one means os assessing the quality of routine measurements.

PT schemes are the most common and perhaps the most important, type of interlaboratory comparisons.

The primary aim of proficiency testing is to provide a quality assurance tool for individual laboratories

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Proficiency Testing & Interlaboratory Comparision

Advantages of PT

Participation in PT enables you to compare your results with those from laboratories. It can also provide you with:

- Regular, objective and independent assessment of the quality of your routine analyses
- Feedback that stimulates improvement of the technical work
- Comparative information about method and instrument performance

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Proficiency Testing & Interlaboratory Comparision

Limitations of PT

Ideally, PT samples are similar in nature to routine samples, and sufficiently homogeneous and stable not to influence the evaluation of participants performance. Due to practical aspects, PT samples are sometimes processed, e.g. Stabilised and/or freeze-dried.

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Proficiency Testing & Interlaboratory Comparision

Appropriate PT Schemes

The huge number of analytes, and the large variety in the way tests are performed, means that it is not always possible to find a scheme that meets a laboratory's exact requirements. Before signing up for a scheme, carefully check that the test materials, and the analytes and their levels fit your routine measurements. Is the frequency appropriate and does the provider's report give you sufficient information?

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Proficiency Testing & Interlaboratory Comparision

PT in Measurement Quality

Correct measurements require both internal and external tools. During the validation step, the performance of the method is established. Subsequent use of control charts will show if the measuremnts are under statistical control. Many laboratories choose to accredit their services, thereby agreeing to implement a quality management system and accepting regular external audits of their work. Participation in PT is an effective

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Proficiency Testing & Interlaboratory Comparision

PT Results

Bacause PT schemes provide an overview of the analytical quality for specific applications, the results are increasingly used by laboratories custumers, and by accreditation and regulatory bodies. PT helps identifying measurement problems, which have a direct impact on trade, environmental monitoring and health and safety

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Proficiency Testing & Interlaboratory Comparision

PT Providers

TÜGAP TÜBİTAK MRC Food Instiute: www.mam.gov.tr
IMEP JRC IRMM: http://irmm.jrc.ec.europa.eu
LGC Standards Proficiency Testing: www.lgcpt.com
TÜBİTAK UME: www.ume.tubitak.gov.tr
FAPAS The Food and Environment Research Agency www.fapas.com
NIST National Institute of Standarts and Technology www.nist.gov

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SORULAR??

Thank You

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LECTURE 5

PRINCIPLES OF METHOD VALIDATION AND MEASUREMENT UNCERTAINTY IN CHEMICAL ANALYSIS

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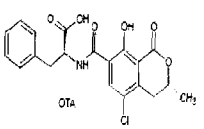
Principles of method validation and measurement *uncertainty in chemical analysis*



Hayrettin ÖZER

November 2009

Overview

- What is Uncertainty
- Types of the Uncertainty
- What is method validation?
- Validation stages?
- Method Performance Parameters (TYPE A Uncertainties)
- How do you assess fitness-for-purpose?
- Quality Controls

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What is Uncertainty?

- **Uncertainty** → **Suspicion**
- **Uncertainty of Measurement** → **Suspicion on the trueness of the measurement**

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What is Uncertainty?

An estimate attached to a test result which characterises the range of values within which the true value is asserted to lie (EurochemCITAC, QUAM 2000)

An example: *If you do not know whether it will rain* tomorrow, then you have a state of uncertainty. If you apply probabilities to the possible outcomes using weather forecasts, you have quantified the uncertainty

Uncertainty of measurement

Parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand.

Uncertainty is an expression of dispersion of measurement results and it can not be removed.

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Types of Uncertainty

Uncertainty of measurement comprises, in general, many components:

Type A: Those can be evaluated from the statistical distribution of the results of series of measurements and can be characterised by standard deviations. (Uncertainties from Validation Parameters)

Type B: can be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information. (Uncertainties from calibration certificates, certificates of reference materials, etc.)

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What is Validation?

ISO 17025 definition: The *confirmation* by examination and the provision of objective evidence that particular requirements for a specific intended use are fulfilled.

Systematical test/measurements and [illegible] of a method to

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Why is method validation necessary?

- Ethical
  - *establish fitness-for-purpose on customer's behalf*
  - good science
- Commercial
  - "due care" in product liability/responsibility
- **Regulatory**
  - **legal requirements**
  - **consistent application of method**

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When do you validate a method?

- Before using any method
  - ➢ Verify own ability to match published data in your own laboratory conditions
  - ➢ Verify suitability for analytical requirements
- During method development
- **Change of application / working environment / analysts**
- **If you notice changes in your results during your Quality Control (QC) checks**

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Who validates a method?

- The analyst
  - In-house development and validation of new methods
  - Verification of the performance of previously validated methods
- **The laboratory**
  - **method development and validation section**

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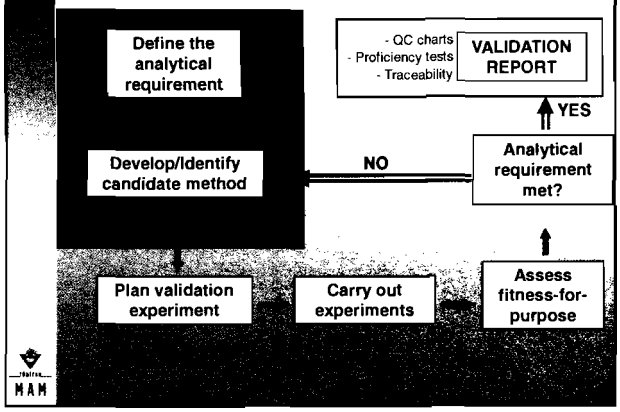
VALIDATION STAGES



Technical Sufficiency

- Tools have to be identified to determine the quality of the results
  - ➢Internal quality control tools
  - ➢External quality control tools

Technical Sufficiency

- Internal quality control tools
  - ➢Quality control of equipments
  - ➢Quality control of measurements
- External quality control tools
  - ➢Interlaboratory tests (comparison tests)
  - ➢Proficiency tests

Method Validation/Performance Parameters

(TYPE A Uncertainties)

Providing evidince that the method produces results that are fit-for-purpose

- Bias, recovery, accuracy
  -how close are the results to the "right" answer?
- Precision (repeatibility, reproducibility)
  - how close are the results of replicate measurements made on the same sample?
- Working & Linear Range(LOD, LOQ, Linearity)
- Specificity/Selectivity

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Bias, recovery, accuracy

- **How close are the results to the "right" answer.**
- **You have to calculate your recovery by spiking or you have to use referance (RM)/certified referans material (CRM) for calculation of bias**

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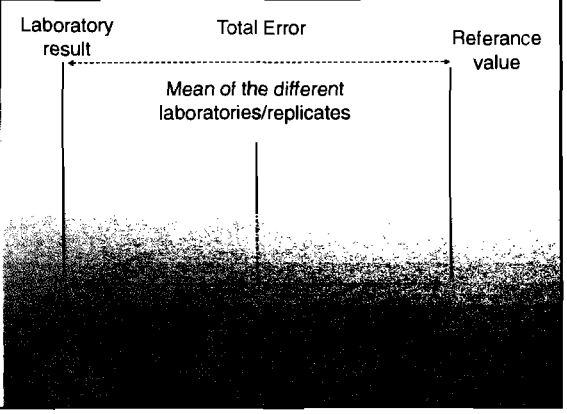
ERROR & BIAS



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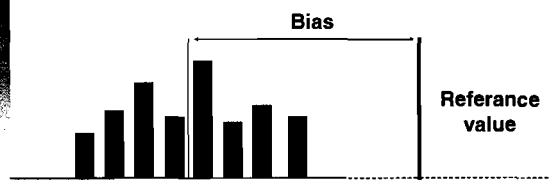
BIAS



- Replicate analysis of referance material
- Difference between mean value of analytical results and referance value

RECOVERY

- The methods that you use don't always measure the component that you are interested in
- Generally other components effect the results possitively or negatively

RECOVERY

Possible to calculate recovery in 2 ways

1- By spiking the sample with a known amount of the component

2- By using CRM

RECOVERY

1- SPIKING:

Known amount of pure standard is added to the sample, then you have 3 values:

- Result of the sample
- Concentration of the standard added
- Result of spiked sample

RECOVERY

The formula to calculate the recovery:

Recovery(%) = 100x(C1-C2)/C3

C1= Concentration of the spiked sample

C2= Concentration of the sample

C3= Concentration of the standard

RECOVERY

To have statisticaly meaningful result;

- Recovery has to be calculated for at least 2 levels of concentration
- At least 6 replicates have to be performed for recovery calculation for each level of concentration

$$s = \sqrt{\left(\frac{\sum_{i=1}^{n} (xi - \bar{x})^2}{n-1}\right)}$$

$$RSD = \frac{s}{\bar{x}}$$

RSD of the mean

$$RSD_{\bar{x}} = \frac{RSD}{\sqrt{n}}$$

RECOVERY

2- BY USING RM/CRM

- CRMs/RMs are used (Preferably CRMs)
- In these samples, there is known amount of component that you are interested in
- Results of the experiment and the reference value given in the certificate are

PRECISION

- How close are the results of replicate measurements made on the same sample?
- Trueness of the results are not important
- Expressed as standard deviation/Relative standard deviation
- Repeatibility and Reproducibility

PRECISION

Repeatibility,

- 6-10 replicates of whole method have to be run
  for the same sample
  by using same instrument
  in the same laboratory
  in a short time (1-3 days)
  by same analyst

$$s = \sqrt{\left(\frac{\sum_{i=1}^{n} (xi - \bar{x})^2}{n-1}\right)}$$

$$RSD = \frac{s}{\bar{x}}$$

Suggested value for RSD from repeatibility related to the concentration of measurand:

(Pure&Appl.Chem 62 (1990) 149-162)

Concentration	RSD (%)
100 g/kg	2
10 g/kg	3
1 g/kg	4
100 mg/kg	5
10 mg/kg	7
1 mg/kg	11
100 µg/kg	15
[illegible]	21

PRECISION

Reproducibility;

Intra-reproducibility	Inter-reproducibility
Different analyst	Different analyst
Different equipment	Different equipment
Same Laboratory	Different Laboratory
In a long time	In a long time

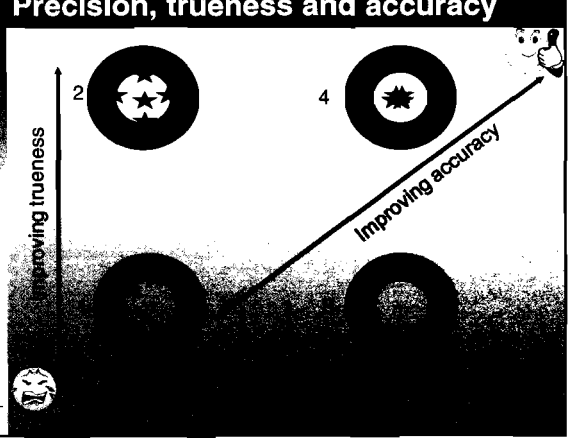
- At least 6-10 replicates of whole method are [illegible]

$$RSD = \sqrt{\frac{\sum\left[(a_i - b_i)/\bar{x}_i\right]^2}{2n}}$$

$$s = \sqrt{\left(\frac{\sum_{i=1}^{n}(xi - \bar{x})^2}{n-1}\right)}$$

$$RSD = \frac{s}{\bar{x}}$$

Precision, trueness and accuracy



Limit of detection-LOD

- Minimum concentration that can be detected by the method in the laboratory condition has to be determined.
- Limit of detection is the concentration which can be analyzed qualitatively but which can not be quantified.

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CALCULATION of LOD

- Standard deviation (SD) of blank response
  - 10 replicates of the whole method are necessary
  - LOD=3 x SD
- Standard deviation of lowest spike
  - 10 replicates are necessary
  - LOD=3 x SD
- Successive dilution
  - 10 replicates are necessary

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LOD, Successive Dilution

Concentration (ppb)	Replicate	Positve/negative result
50	10	10/0
40	10	10/0
30	10	10/0
20	10	7/3
15	10	5/5
10	10	1/9
5	10	0/10

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Limit of quantification-LOQ

- LOQ is the lowest level at which uncertainty is acceptable
- The lowest level that you can quantified.
- Use the same data of LOD
  - LOQ= 10xSD (In some references, 5 or 6 is used as a factor instead of 10)

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LOD & LOQ

1	29,75
2	29,23
3	29,60
4	28,33
5	28,98
6	29,50
7	29,07
8	30,20
9	29,10
10	28,58
Mean	**29,23**
Sd	**0,552**
LOD	**1,66**
LOQ	**5,52**

LOD= 3xsd

LOQ= 10xsd

LOD= 3x0,55=1,66 ppb

LOQ= 10x0,55=5,52 ppb

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Working Range and Linearity

- Working range has to be determined in quantitative methods. The results of the analysis must be within the this working range
- The lowest level of the working range is LoQ. The highest level of the range is up to equipment used. Usually highest level is the response of the highest standard used in the calibration curve.

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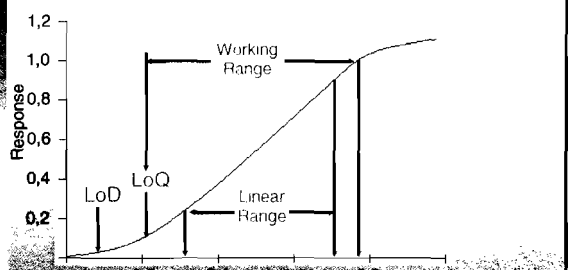
Working & Linear Range



SPECIFICITY / SELECTIVITY

- How selective is the method?
- Is there any interferences in the sample for the method?
- During method development
- **Spiking of different component at different concentrations**

Robustness / Ruggedness

- During method development
- Identfy the parameters which can effect the results (e.g. Temparature for moisture analysis in biscuit)
- **Change the parameters (e.g. Change the temperature from 103C to 95 and then to 110)**

Limited validation

- **Precision**
  - Repeatability
  - Reproducibility
- **Accuracy**
  - Bias
  - Recovery
- **Working & Linear range**
  - LoD & LoQ
  - Linearity

Method performance parameters

Performance criteria for Aflatoxins (Both for aflatoxin B1 and Total Aflatoxins)*

Recovery-Aflatoxin M1	0,01-0,05	60-120
	>0,05-1,0	70-110
	>1,0	50-120
Recovery-Aflatoxin B1, B2, G1,G2	1-10	70-110
	>10	80-110

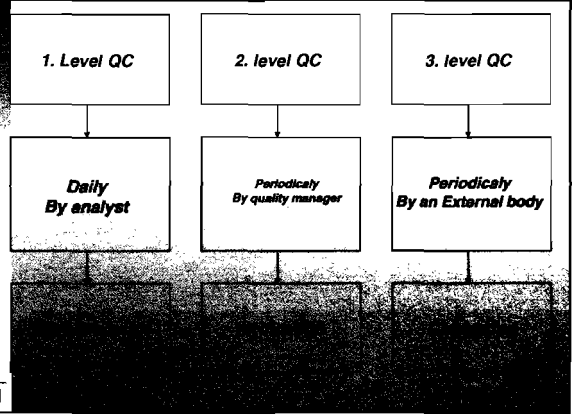
Performance criteria for Ochratoxin A*

<1	≤40	≤60	50-120
1-10	≤20	≤30	70-110

Fitness-for-purpose

- **Analyse data from method performance parameters**
- **Are target values achieved?**
  - **YES: method is fit-for purpose**
  - **NO: more development required**
- **Method is validated by the declaration**

Quality Control (QC) of Measurements



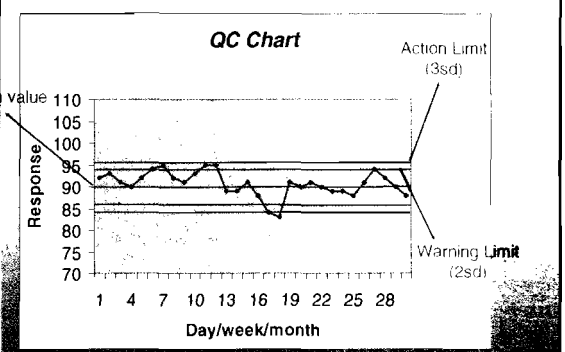
Quality Control of Measurements

- **1 level QC**
  - ➢Blank
  - ➢Calibration standards
  - ➢Control samples or referance material
  - ➢Parallel analysis
  - ➢**Spiking of standard, using an internal standard**
  - ➢Cross check with another method

Quality Control Charts

- Quality Control (QC) charts are the most important tools those can be used for checking the performance of the method
- Qualtiy Control charts have to be prepared for whole of the method

Using of Quality Control Charts



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Quality Control of Measurements

- Evaluation Criterias

IF

- ➢ 10 sequential responses are on the same side of mean
- ➢ 2 sequential responses are out of 2σ (Warning limit)
- ➢ 1 response is out of 3σ (Action limit)
- ➢ [illegible]

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Quality Control of Measurements

- **2. level QC (by quality manager)**
  - ➢ Blank sample
  - ➢ Referance material
- **3. level QC**
  - ➢ Comparisions between the laboratories
  - ➢ Join to Proficiency Testing organized by an

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Keep in mind (Method Validation)

- Method validation is required to produce meaningful data
- Both in-house and standard methods require validation
- Validation should be a planned activity
  - Parameters required will vary with application

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Thanks for your attention

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16–20 November 2009

LECTURE 6

PRINCIPLES OF METHOD VALIDATION AND MEASUREMENT UNCERTAINTY IN MICROBIOLOGICAL ANALYSIS

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Principles of method validation & Measurement uncertainty in microbiological analysis

UNIDO
International Training Programme
November 16-20, 2009
Food Institute

N. Aslı Kısıklaya ÖNCÜ (MSc.)

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- What is Uncertainty?
- What is method validation?
- Concepts of Uncertainty?
- Spiking.
- Introduction to uncertainty in microbiological analysis components.
- Uncertainty Measurement Prediction.

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- No measurement is perfect.
- It has associated uncertainty arising from many factors.
- Uncertainty of measurement according to ISO (Anon 1995) is a"Parameter, associated with the result of a measurement, that characterises the

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- **Uncertainty is an expression of dispersion of measurement results and it can be removed.**

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- Why do have to know uncertainty in Microbiological analysis?
- **Evaluating the results.**
- **Evaluating the performance of the labortatory test.**
- **According to ISO/IEC 17025 "Laboratory shall at least attempt to identify all the components of uncertainty and make a**

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Method Validation

- <u>ISO 17025 definition:</u> **The confirmation by examination and the provision of objective evidence that particular requirements for a specific intended use are fullfilled.**
- **The laboratory shall validate non-standardized methods, laboratory designed/developed methods, standardized methods used outside their intended range and amplifications of**

Method Validation

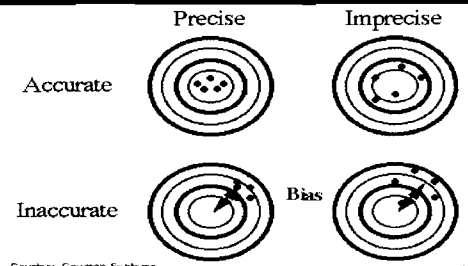
- The validation of microbiological test methods should reflect actual test conditions.
- This may be achieved by using naturally contaminated products or products spiked with a predetermined level of contaminating

Concept of Uncertainty

What is the real value of the result?

- Is this result correct or not ?

Concepts of Uncertainty



Courtesy Gayman Systems

You can correct for Bias
You can NOT correct for Imprecision

Concepts of Uncertainty

- Accuracy, is a qualitative concept; can be defined as the correctness of a result *"The closeness of agreement between a test result or a measurement result and the true value"*
- Bias, is the difference between the expectation of the test results and an accepted reference value and is a measure of total systematic, but not random, error.

"Bias is a measure of *Trueness*"

Concepts of Uncertainty

- Precision, is defined as the closeness of agreement between independent test results obtained under stipulated conditions.
- Precision depends only on the distribution of random errors and does not relate to a true value or a specified value.
- Independent test results means results obtained in a manner not influenced by any

SPIKING (Inoculation)

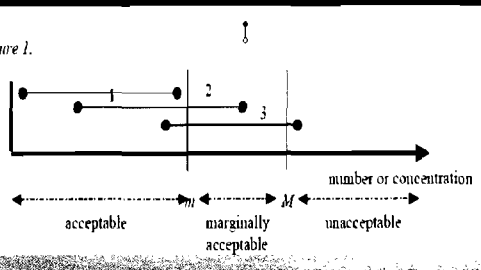
Spiking: Products spiked with a predetermined level of contaminating organisms.

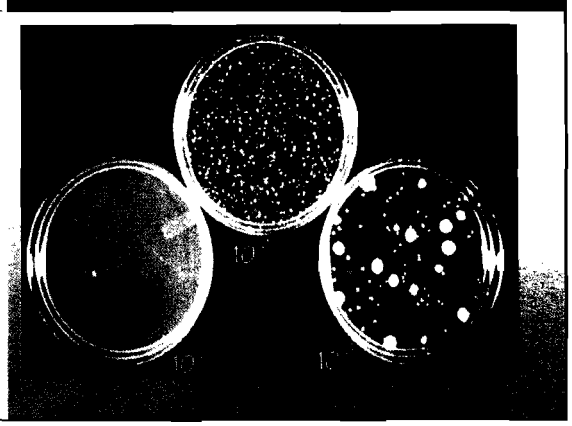
Inoculation of product suspensions with ATCC microorganisms

Quantitative analysis

- Spiking level; should be determined in the appropriate level intervals specified in the analysis ?
- m:100 and M: 1000 cfu/g. For instance; acceptable limits of this spiking level should be between 100-10.000 cfu /g.
- For estimation of uncertainty; the results also should contain above 1 or 2 log of range

Figure 1.





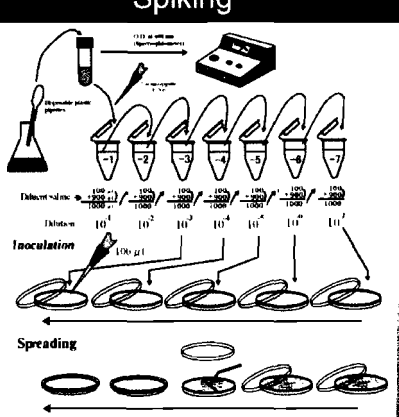
Qualitative analysis

- At least two different spiking level should be performed.
- Low Level: To identify the limit of detection (LoD)
- High Level: To show that you really can find it.
- Appropriate level can be obtrained from literature and /or validation reports.
- For instance; assuming a perfect test, when sampling 25 g of food the smallest number of colony forming units (cfu) that can be detected is 1. This is equivalent to a concentration of 1 cfu/25 g or 0.04 cfu/g, which is known as the [illegible]

Spiking sample for Qualitative analysis of *Salmonella*

- The highest level of spiking for 25 gram should be 100-200 Colony forming unit.
- If it is possible try to use different ATCC microorganism. (Different strains)
- If appropriate, as the competitive flora, add *Escherichia coli*, *Proteus* spp.

Spiking



Concepts of Uncertainty

- The total uncertainty of a test result typically consists of several components. In microbiology, at least three factors are always involved:

a) The uncertainty of the inoculum volume

b) Random scatter due to particle statistics

c) The uncertainty of reading the result.

d) Uncertainty of dilution is frequently a

Concept of Uncertainty

Standard Deviation: Is a measure of the dispersion of a collection of numbers, it is defined as the rooth-mean-square (RMS) deviation of the values from their mean, or as the square root of the variance.

$$s = \sqrt{\left(\frac{\sum_{i=1}^{n}(xi-\bar{x})^2}{n-1}\right)}$$

Concepts of Uncertainty

Arithmetical mean, x mean of a list of numbers is the sum of all of the list divided by the number of items "n" in the list.

$$\bar{x} = \sum_{i=1}^{n} x_i$$

Concepts of Uncertainty

Reproducibility & Repeatibility

Measures of repeatability and reproducibility are the corner stones of estimation of analytical uncertainity

Concepts of Uncertainty

Reproducibility: is a measurement of the differences among the analyses which were performed under the same conditions.(Same method, same sample/test)

- Same operator, same analysist
- Same equipment
- Same conditions
- Same time (With short intervals)

Repeatibility

Repeatibility: is a measurement of the differences among the analyses which were performed under the different conditions. Same method, same sample/test)

- Different operator (Different laboratory)

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Introduction to uncertainty in microbiological analysis components.

Where Uncertainty comes from?

- Sampling, sub-sampling /Primary dilution.
- Homogeneity of materials (homogeneity, not homogenous)
- Permanent random errors.
- Operator/time (training, experience, ability)
- Deviation (Systematic error)

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Introduction to uncertainty in microbiological analysis components.

Uncertainity components come from Equipments?

- Scale (Human errors in weighing)
- Thermometer
- Bagmixer blenders
- Autoclave
- Diluents

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Introduction to uncertainty in microbiological analysis components.

Uncertainity components come from Operators?

- Sub-sampling
- Dilution (Pipetting)
- Treatment period.
- Petri dish.
- Reading of Petri dish(Uncertainty of reading)
- Biochemical Identification

Introduction to uncertainty in microbiological analysis components.

Other Uncertainity components ?

- Quality of media (it is so important)
- The consequence of changing brands of commercial media.
- The use of the incorrect ingredients in a culture medium.
- Water quality (Detergent residues)
- Cleaning of glass apparatus.
- Storage of media before in use and during in use.

Uncertainty Measurement Prediction.

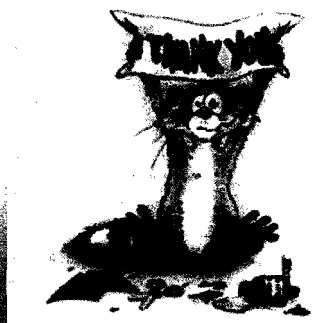
ISO 19036?

- Quantitative Uncertainity Measurement Manual.
- ISO 19036 provides an easy way for calculating uncertainty in microbiological analyses.
- According to ISO 19036; on the basis of initial estimates of uncertainty should

Quality Control and Quality Assurance Measurements.

- Internal Quality Control:
  - ➢Reference Samples.
- External Quality Control:
  - ➢Interlaboratory tests (comparison tests)

Thank you for your attention!



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16–20 November 2009

LECTURE 7

FOOD LABELLING (REGULATIONS, IMPORTANCE AND APPLICATIONS)

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Food Labelling

Regulations Importance Applications

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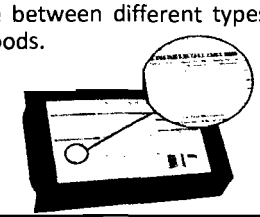
19.11.2009

Food Labelling

- Consumers today expect a great deal of information about the food products they purchase.
- Providing this information on food labels helps consumers to make informed choices, and can help people to choose between different types, brands and flavors of foods.



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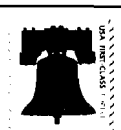
Why label food?



- **Protect public health and safety**
  - by health and safety information
    - e.g. allergen labelling
- **Provide adequate information for informed choice**
  - By basic product *Information*
    - e.g. common name, ingredients, net quantity, shelf life, country of origin
- **Prevent misleading or deceptive conduct**
  - e.g. description of the product
- **Vehicle for food marketing, promotion and advertising**

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Food Regulations

- **Codex Alimentarious**
- **The European Commission (EC)**
- **FDA (US)**
  - *Federal Food, Drug, and Cosmetic Act (FD&C Act) Chapter IV: Food*
  - *Fair Packaging and Labeling Act of 1966*
- **FSA (UK)**

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Food Regulations

TURKEY

Turkish Food Codex

Communiqué on Rules for General Labelling and Nutritional Labelling of Foodstuffs (2002-58)

This communiqué has been prepared within the framework of compliance with European Union in accordance with the Commission Directives:

•2000/13/EEC on "Approximation of the Laws of the Member States Relating to Labelling, Presentation and Advertising of Foodstuffs"

• 90/496 on "Nutrition Labelling for Foodstuffs"

•80/232/EEC on "Approximation of the Laws of the Members States Relating to the Ranges of Nominal Quantities and Nominal Capacities Permitted for Certain Prepackaged Products".

Codex Alimentarious

- A collection of internationally adopted food standards, guidelines, codes of practice and other recommendations.
- The only worldwide intergovermental Food Standard Organization consisting of
  - 180 member countries and one member organisation (EC)
- Observers
  - United nations organizations
  - International scientific organisations
  - Consumer organisations
  - Food industry and trade

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Codex Alimentarious

- The Codex Alimentarius Commission was created in 1963 by FAO and WHO to develop food standards, guidelines and related texts such as codes of practice under the Joint FAO/WHO Food Standards Programme.

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Codex Alimentarious

Main Purposes

- To protect the health of consumers
- To ensure fair practices in the food trade
- To coordinate all food standarts work undertaken by international and non-governmental organisations.

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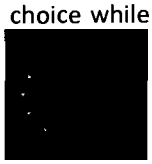
Codex Alimentarious

Current Official Standarts
General Standard for the Labelling of Prepackaged Foods
General Standard for the Labelling of Food Additives when sold as such
Standard for Labelling of and Claims for Prepackaged Foods for Special Dietary Use
General Guidelines on Claims
Standard for Labelling of and Claims for Foods for Special Medical Purposes
Guidelines on Nutrition Labelling
Guidelines for the Production, Processing, Labelling and Marketing of Organically Produced Foods
Guidelines for Use of Nutrition and Health Claims
General Guidelines for Use of the Term Halal

The European Commission (EC)

- In the European Union, rules are put in place on the labelling of foodstuffs to enable European consumers to get comprehensive information on the contents and the composition of food products. Labelling helps consumers to make an informed choice while purchasing their foodstuffs.



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The European Commission (EC)

- Directive 2000/13/EC
  - the labelling, presentation and advertising of foodstuffs
- Council Directive 90/496/EEC
  - nutrition labelling for foodstuffs into one instrument

MAM

Directive 2000/13/EC

- of 20 March 2000 on the approximation of the laws of the Member States relating to the labelling, presentation and advertising of foodstuffs.

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Other Directives of Food Labelling

- Directive 2002/67/EC
  - labelling of foodstuffs containing quinine, and caffeine
- Directive 2003/89/EC
  - Allergen labelling
  - amending Directive 2000/13/EC as regards indication of the ingredients present in foodstuffs
- Directive 94/35/EC
  - sweeteners for use in foodstuffs
- Regulation (EC) 1830/2003
  - Labelling of GMO Foods

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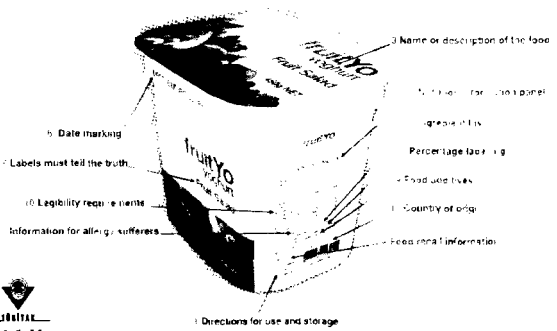
Mandatory food labelling requirements	Voluntary* information sometimes provided
Name	Nutrition information (if no claims made)
List of ingredients	Nutrition signposting
Quantity of certain ingredients (QUID) *e.g. pork (10%)*	Guideline Daily Amounts
Net quantity (weights & measures)	Claims such as 'no artificial additives'
Date of minimum durability ('best before' or 'use by')	Graphical and pictorial information
Any special storage conditions	Graphical and pictorial information
The name and address of manufacturer/ packer/ seller	Vegetarian/ vegan labelling
Place of origin (if failure to do so might mislead)	May contain (e.g. nuts) labelling
Instructions for use (if failure to do so might mislead)	Assurance schemes
Alcoholic strength by volume (drinks over 1.2% only)	Free range e.g. eggs

Mandatory food labelling requirements	Voluntary* information sometimes provided
Allergen information (in the ingredient list)	Method of slaughter (e.g. Halal)
Quinine labelling	Marketing terms e.g. fresh, pure, natural
High caffeine content warning (drinks containing over 150mg/l of caffeine)	Number of servings
Sweeteners labelling	Environmental impact e.g. dolphin friendly
Polyol warning ('excessive consumption may produce a laxative effect)	Country of origin (where not required)
PKU warning ('contains a source of phenylalanine')	Customary or descriptive names
Packaging gases ('packaged in a protective atmosphere')	Quality type claims e/g/ '100% chicken breast'
Raw milk labelling	Special offer competitions
GMO labelling	Production methods (e.g. organic)
Irradiated food labelling ('irradiated' or 'treated with ionising radiation')	Logos
Nutritional Panel (where a nutritional claim is made, e.g. low in fat)	Brand information * Based on information provided voluntarily in the

Consumer information on food labels



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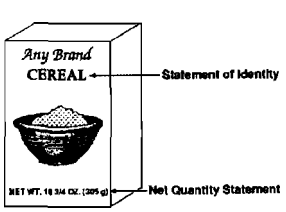
The Name of The Food

- ***The name of the food must be clearly displayed on all prepacked foods.***
  - it is not sufficient to just say 'cheese', because there are so many different types of cheese.
- ***clear description of the food***
  - "M&M's" – milk chocolate covered peanuts in a crisp coloured shell
- ***any processing the food has undergone***
  - *dried* apricots- *salted* peanuts- *smoked* mackerel.
- ***describe the differences between apparently similar products.***
  - a yogurt labelled 'fruit yogurt' must be flavoured with real fruit
  - a yogurt labelled 'fruit flavoured yogurt' can be flavoured with artificial flavourings

MAM

Net Quantity (Weights&Measures)

- The weight or volume of prepacked foods must be shown on the label.
- The symbol e indicates an average quantity i.e. the average pack is at least the weight declared.
- Comparing the weight with the price of different brands enables consumers to make choices between brands based on value for money.



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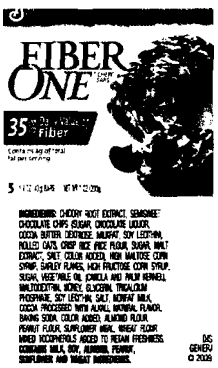
List of Ingredients

- Ingredients must be listed in descending order of weight, according to the amounts used to make the **food**.
- The ingredient names must be given in the language of the country where the **food** is to be sold.
- All additives must be stated on the ingredients list.



MAM

Allergen Information (in the ingredient list)

- Directive 2003/89/EC
- Establishes a list of ingredients, known to be likely to trigger adverse reaction in sensitive individuals.
  - 12 food ingredients;
  - milk, eggs, peanuts, nuts, fish, crustaceans, soya, wheat, celery, mustard, sesame and sulphur dioxide – to be clearly labelled on food products.
- This is particularly helpful to people with food allergies and food intolerances, who may need to avoid these specific food ingredients.

MAM

GM food and irradiated foods

- Regulation (EC) 1830/2003
- The EU recognises the consumers' right to information and labelling as a tool for making an informed choice. Since 1997 Community legislation has made labelling of GM food mandatory for:
  - products that consist of GMO or contain GMO;
  - products derived from GMO but no longer containing GMO if there is still DNA or protein resulting from the genetic modification present;

MAM

Date and storage conditions

How long a product will keep,
How the product should be stored

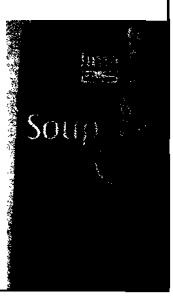
- 'Date marking' provides an important safeguard against foods which may be unsafe to eat.
- Following storage instructions
  - prevent food from spoiling too quickly
  - helps to ensure that food looks and tastes its best when eaten.

• Perishable foods that spoil quickly, have a 'Use by' date.
• 'Use by' provides clear instruction that a food should be used by the end of the date shown on the label.

MAM

Preparation instructions

- Where relevant, instructions on how to prepare and cook a food must be given on the label. These instructions help to ensure that a food tastes its best when eaten, and that it will be thoroughly heated to a core temperature of 75°C to minimize the risk of food poisoning.



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Place of origin

- 'Place of origin' must be included on the **label** if it is unclear where the **food** has come from.
  - Greek yogurts are made in France and it might mislead the consumer if this is not properly labelled.

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Nutrition Labelling

- Nutrition labelling can help consumers to make healthy choices and can provide clear consistent messages to consumers.
- Nutrition labelling is only required by EU Law where a nutritional claim about a product is made (e.g. low fat, rich in calcium),
- but as consumers' knowledge about health and diet grows, many manufacturers are choosing to display nutrition information on **food** labels.

MAM

Nutrition Labelling

- Directive 90/496/EEC
  - Nutrition labelling for foodstuffs
- This directive ensures that nutrition information is presented in a standard way, allowing consumers to easily compare the nutrient content of one food with another.

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Nutrition Labelling

- The nutrition information order;
  - Grup 1
    - Energy value
    - Protein, Carbonhydrate, Fat
  - Grup 2
    - Energy value
    - Protein, Carbonhydrate, Sugars, Fat, Saturates, Fibre, Sodium

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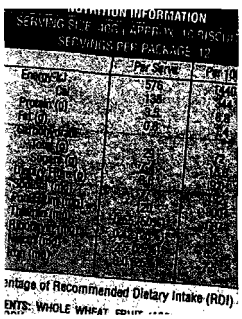
Nutrition Labelling

- Nutrition information must always be given as values per 100g or per 100ml of food.
- Values for a portion or serving can be given as well, provided that the number of servings contained in the pack is shown on the label.



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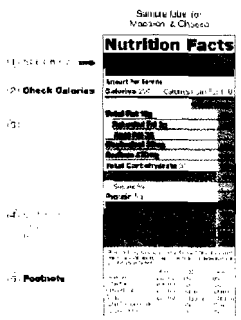
Nutrition Labelling

Reference Daily Intake (or Recommended Daily Intake) (RDI)

is the daily dietary intake level of a nutrient which was considered (at the time they were defined) to be sufficient to meet the requirements of nearly all (97–98%) healthy individuals in each life-stage and sex group.

The RDI is used to determine the Daily Value which is printed on food label.



Nutrition and health claims

- Food manufacturers often want to draw attention to the amount of nutrients present in a food, outside of the nutrition information panel on the label.
- Nutrition and health claims provide an opportunity for manufacturers to highlight any specific nutritional benefits a food many have, beyond the provision of nutrients for general health and well being.

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Nutrition and health claims

- **Regulation (EC) 1924/2006**
  - ***on nutrition and health claims made on foods***
- ensure that any claim made on a food label in the EU is clear, accurate and based on generally accepted scientific evidence.
- enable consumers to make informed and meaningful choices when it comes to food and drinks.

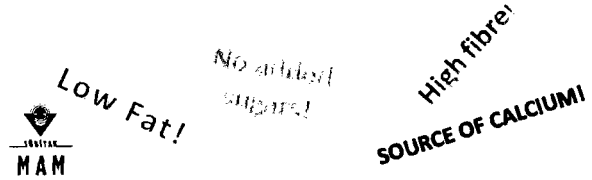
MAM

Nutrition and health claims

- **Regulation (EC) 1924/2006**
  - ***on nutrition and health claims made on foods***
- It applies to all nutrition and health claims made in commercial communications, i.e. Labelling, presentations or advertising of foods and supplements,
- Aims to provide a high level of consumer protection whilst allowing the EU market to function effectively.

Nutrition Claims

- For a company to make a nutrition claim on their product, the specific claim must be listed in the Annex to the regulations.
- Examples listed in the Annex include:
- Conditions are specified for each claim
  - Foods claiming to be 'low fat' must have no more than 3g fat per 100g



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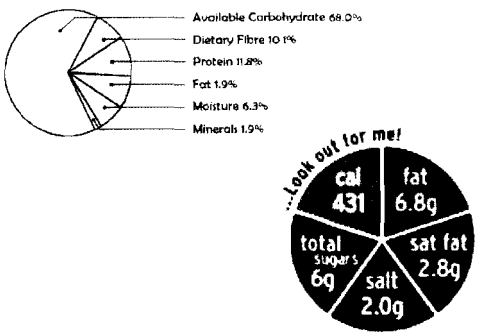
Expression/Presentation



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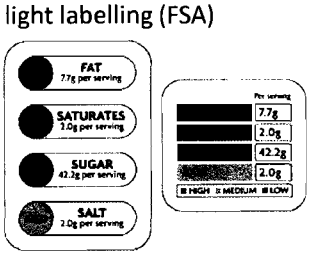
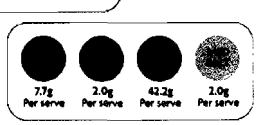
Expression/Presentation

Traffic light labelling (FSA)





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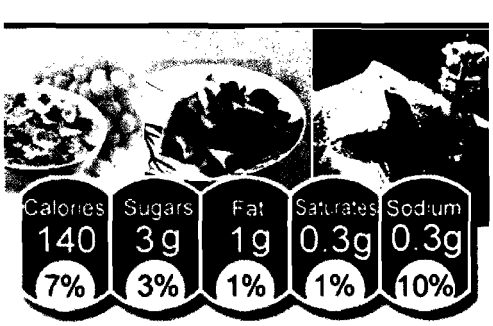
Expression/Presentation

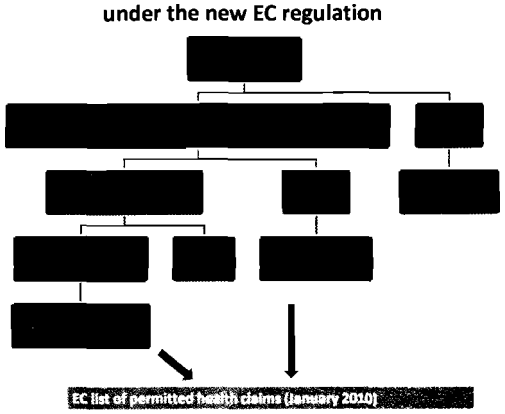


Health Claims

- any claim that states, suggests or implies that a relationship exists between a food category, a food or one of its constituents and health
- two essential components:
  - (1) a substance
  - (2) a disease or health-related condition
  - There should be information about any population groups that should avoid eating the food
- A list of permitted health claims will be available from Jan 2010

IRON HELPS MAKE RED BLOOD CELLS, WHICH CARRY OXYGEN AROUND THE BODY

The process of authorising a nutrition or health claim under the new EC regulation



Health Claims

- ***FDA 21 Code of Federal Regulations § 101.14***

Requirements for Health Claims Made in Labeling

Approved Claims	Requirements for the Food	Claim Requirements	Model Claim, Statements
Sodium and Hypertension	- Low sodium	Required terms: - "Sodium", "High blood pressure" Includes physician statement (Individuals with high blood pressure should consult their physicians) if claim defines high or *normal blood pressure*	Diets low in sodium may reduce the risk of high blood pressure, a disease associated with many factors.

Where do manufacturers get the nutrition information for their products from?

- Nutrition information for use on food labels can be obtained from two main sources.

1. **By direct chemical analysis**

   The 'gold standard' for manufacturers to obtain nutrition information for their products is to chemically analyse samples of their products in a laboratory.

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Where do manufacturers get the nutrition information for their products from?

2. **Using food composition data to inform food labelling**

- Food composition databases(FCDBs) contain information on the nutrient content of a range of different foods and ingredients.
- Typically, FCDBs provide values for the amount of nutrients that 100g of a particular food or ingredient contains.
- Datas derived by chemical analyses of the foods and then compiled into databases.
- Manufacturers may then use the nutrient values from these official databases to calculate the nutrition information for their products

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Turkish Food Composition Database

www. .com

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Food Composition Databases

- US : http://www.nal.usda.gov/fnic/foodcomp/search/
- Denmark: http://www.foodcomp.dk/v7/fcdb_search.asp
- France: http://www.afssa.fr/TableCIQUAL/
- Finland: http://www.fineli.fi/index.php?lang=en
- Japan: http://database.food.sugiyama-u.ac.jp/PC_ASIA/search2.php
- Austria:http://www.foodstandards.gov.au/monitoringandsurveillance/nuttab2006/onlineversionintroduction/onlineversion.cfm
- FP6 Eurofir Project: http://www.eurofir.org/eurofir/Links.asp

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Energy

Total food = moisture + fat + protein + available carbohydrates + ash + fiber

- **The Atwater general factor system**
  - The energy values are
  - (4.0 kcal/g) for protein
  - (9.0 kcal/g) for fat
  - (4.0 kcal/g) for carbohydrates
  - (7.0 kcal/g) alcohol
- **The extensive general factor system**
- **The Atwater specific factor system**
- **Net metabolizable energy system**
- **Hybrid systems**

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Protein

- **Kjeldahl-Wilforth-Gunning method**
- Mostly derived from N-content

  ⬇

  conversion factors to express as « crude protein »

- **Dumas method**
- **Spectroscopic methods**
- **Near-infrared**

Foodstuffs	CF
Wheat, whole flour	5.83
Rice	5.95
Soya	5.71
Milk and cheese	6.38
Rye, barley, and oats	5.83
Seeds (sesame, safflower)	5.30
Almonds	5.18
Other foods	6.25

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Fats

"Fat = sum of fatty acids expressed as triglyceride equivalents"

- **Methods for Fat Determination**
- **Gravimetric measurements**
  - Chloroform/methanol (Folch; Bligh & Dyer)
  - Soxhletmethods
  - Acid hydrolysis (WeibullStoldt; Schmidmethods)
  - Alkaline hydrolysis (Mojonnier; Roese-Gottlieb methods)
  - Other (water saturated butanol; hexane-isopropanol)
- ***Volumetric methods***
  - Babcock procedure
  - Gerber procedure
  - Creamatocrit
- **Other methods**

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Carbohydrates and fibre

- **Carbohydrates**
  - Major proximate constituents
  - Major energy source
  - Free or interlinked monosaccharide units
  - Degree of polymerisation
    - Sugars
    - Oligosaccharides
    - Polysaccharides
  - *Glycemic Carbohydrates*
  - Non-glycemicUnavailable
- **Dietary fiber**
  Sum of plant polysacharides and lignin that are not hydrolysed by the endogenous secretions of the human digestive tract

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Carbohydrates and fibre

- **Total and available carbohydrate**
- **Total carbohydrate:**
  - By difference: 100 – (weight in grams [protein + fat + water + ash +alcohol] in 100 g of food)
  - By direct analysis: weight in grams (mono- + disaccharides + oligsaccharides + polysaccharides, including fibre)
- **Available carbohydrate:**
  - By difference: 100 – (weight in grams [protein + fat + water + ash +alcohol+ fibre] in 100 g of food)
  - By direct analysis: weight in grams (mono- + disaccharides +oligosaccharides + polysaccharides, excluding fibre)*
- * May be expressed as weight (anhydrous form) or as the monosaccharide equivalents (hydrous form including water).

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Food, Consumer Surveys

- Surveys help to protect and inform consumers, judge the effectiveness of regulation, monitor trends and assess risks.
- Surveys are important because they carry out checks on retail foods and can therefore alert the goverment to potential food safety issues. However, a food survey that has entirely negative results is just as important as one that shows up a potential problem.

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Consumer awarness and use of food labels

- Country basis
  - Trainings
  - Label aplications
  - Regulation
  - Media
  - Associations
  - Nutrition
- Development level of the country
- Turkish consumers

Literature

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2. http://www.cantest.com/files/CANTEST%20webinar%20-%20Nutritional%20Labelling%20Regulations%20in%20effect.pdf
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Thank you...

TÜBİTAK
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INTERNATIONAL WORKSHOP AND STUDY TOUR ON: FOOD LABORATORIES MANAGEMENT AND PRACTICE

UNISWORK VIII

TUBITAK

MARMARA RESEARCH CENTRE

FOOD INSTITUTE

COUNTRY PRESENTATIONS

Gebze- Kocaeli, TURKEY

16-20 November 2009

TÜBİTAK
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UNIDO International Training Programme on
Food Laboratory Management and Practice
16–20 November 2009

UNIDO International Training Programme on
Food Laboratory Management and Practice

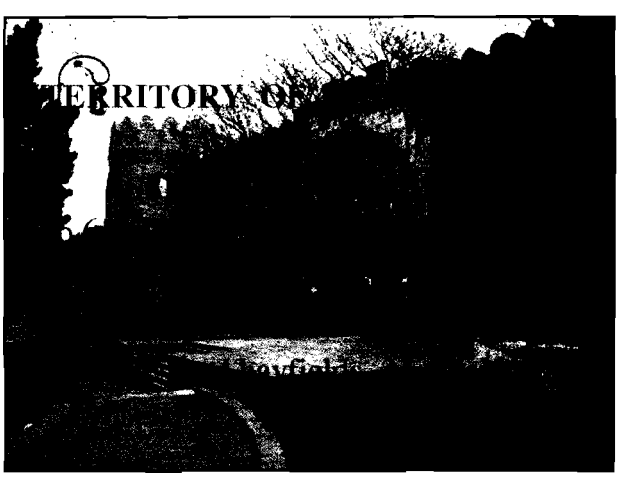
TUBITAK, Marmara Research Centre
Food Institute
Gebze- Kocaeli, TURKEY

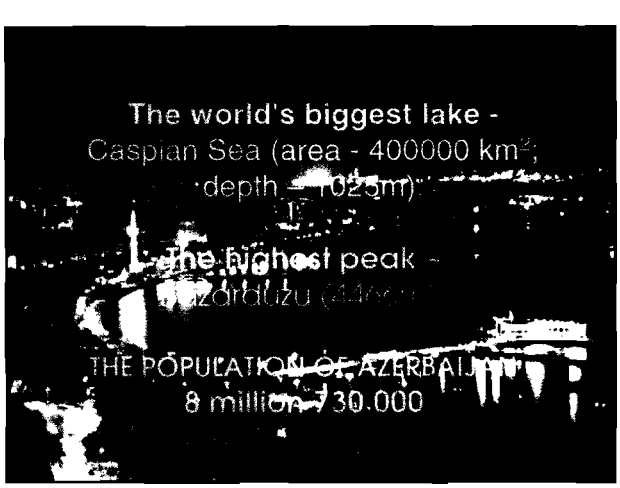
UNISWORK VIII

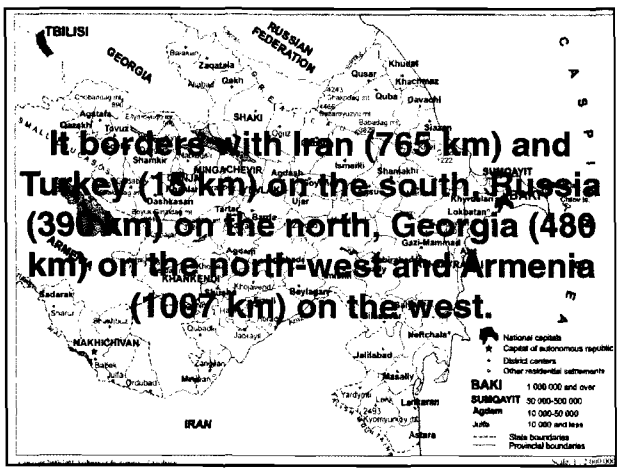
COUNTRY PRESENTATIONS

13:30-13:45	**Ms. Nurana HUSEYNOVA, AZERBAIJAN**
13:45-14:00	**Mr. Mostafa A. SWAPAN, BANGLADESH**
14:00-14:15	**Mr. Samson G. GABRE, ETHIOPIA**
14:15-14:30	**Mr. Clarkson NYAMBOK, KENYA**
14:30-14:45	**Ms. Oxana USATII, MOLDOVA**
14:45-15:00	Coffee break
15:00-15:15	**Mr. Mohammad MOUSE, PALESTINE**
15:15-15:30	**Mr. Philip NZAIRE, RWANDA**
15:30-15:45	**Mr. Iraj AHMEDOV, TAJIKISTAN**
15:45-16:00	**Ms. Agnex MNENEY, TANZANIA**
16:00-16:15	**Mr. Kudret AVCI and Yildiray ISTANBULLU, TURKEY**
16:15-16:30	**Ms. Annette NABBENGO, UGANDA**

Nurana Huseynova
Chief microbiologist
State Office on Control of Consumer Market
Customer Commodities Expertise Bureau



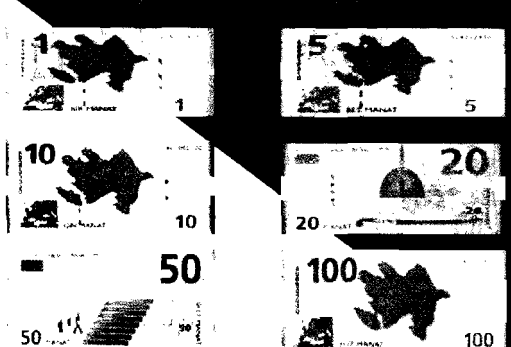


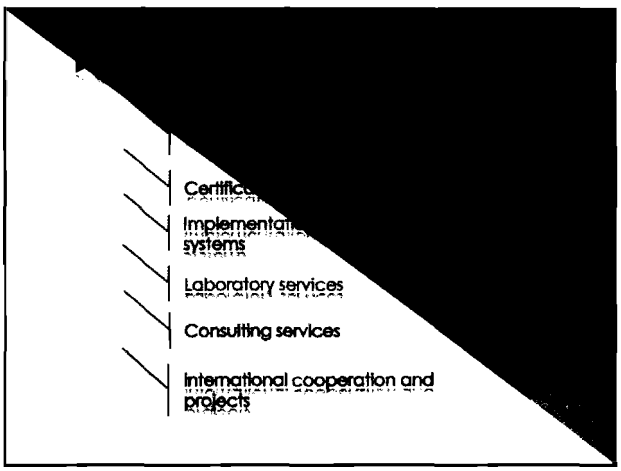


It borders with Iran (765 km) and Turkey (15 km) on the south, Russia (390 km) on the north, Georgia (480 km) on the north-west and Armenia (1007 km) on the west.

Azerb

- Genealogically, belongs to the Turki and, together with close Turkish, Turkmen and Gaga languages, forms the southweste of Turkic languages.




Certific
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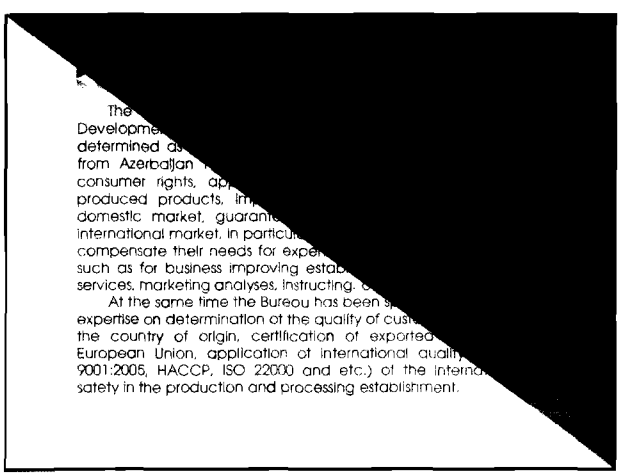
systems
Laboratory services
Consulting services
International cooperation and
projects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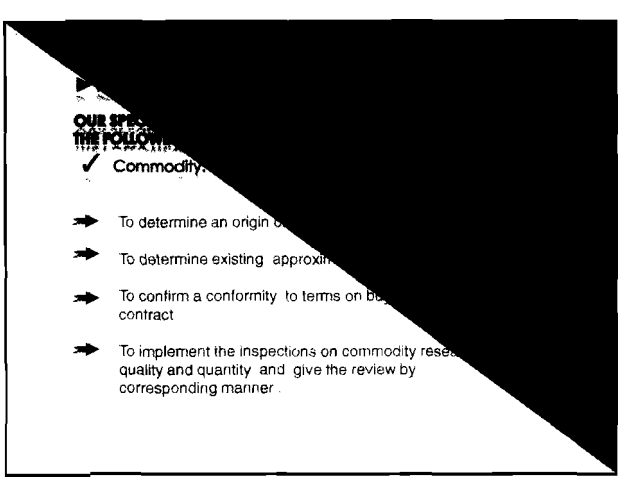
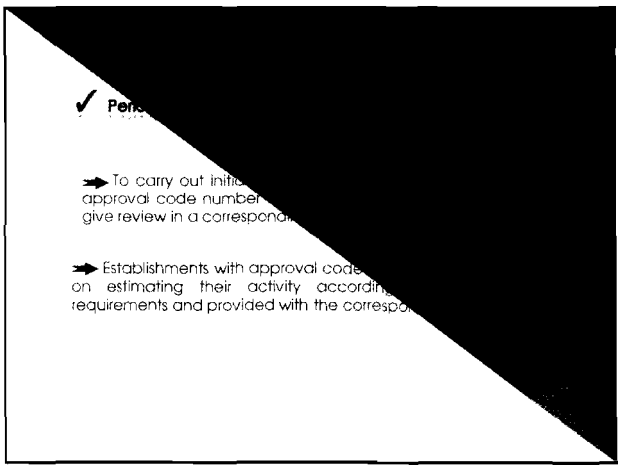
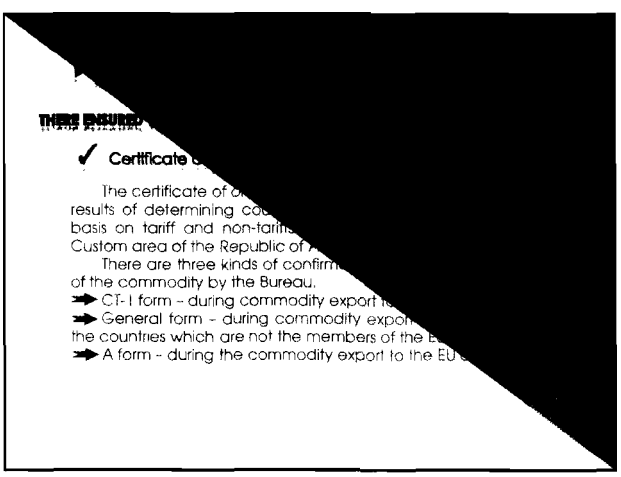
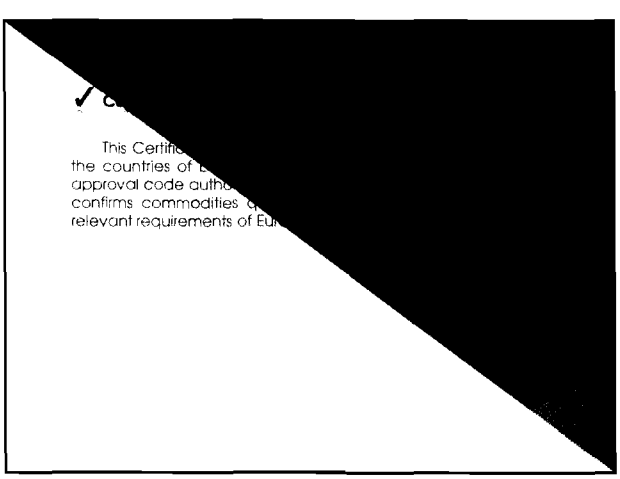
from Azerbaijan
consumer rights, ap
produced products, in
domestic market, guarant
international market. In particu
compensate their needs for expe
such as for business improving estab
services, marketing analyses, instructing.
At the same time the Bureau has been
expertise on determination of the quality of cust
the country of origin, certification of exported
European Union, application of international quali
9001:2005, HACCP, ISO 22000 and etc.) of the interna
safety in the production and processing establishment.


OUR SPE
THE FOLLOW
✓ Commodity
➡ To determine an origin o
➡ To determine existing approxi
➡ To confirm a conformity to terms on b
contract
➡ To implement the inspections on commodity rese
quality and quantity and give the review by
corresponding manner .


✓ Per
➡ To carry out initia
approval code number
give review in a correspond
➡ Establishments with approval code
on estimating their activity accordi
requirements and provided with the corresp


✓ Certificate
The certificate of
results of determining co
basis on tariff and non-tarif
Custom area of the Republic of
There are three kinds of confirm
of the commodity by the Bureau.
➡ CT-1 form – during commodity export
➡ General form - during commodity expo
the countries which are not the members of the E
➡ A form - during the commodity export to the EU


✓
This Certifi
the countries of
approval code autho
confirms commodities
relevant requirements of Eu

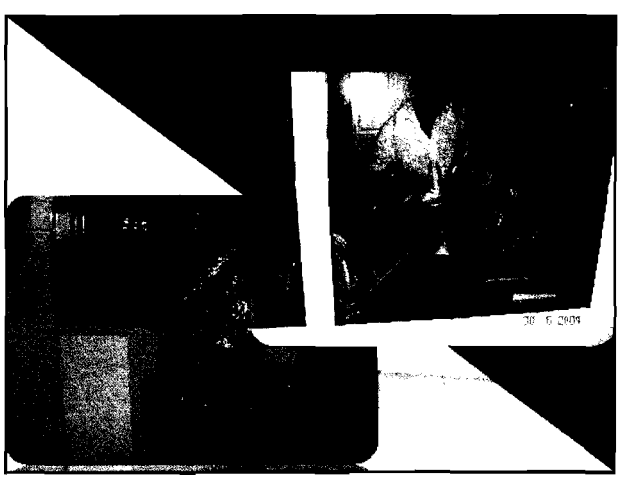
Systen
well as
Managemen
Environmental
(ISO 17025).

✓ **Food safety management**
Implementation of HACC
foodstuff production and proce
support of food safety. Implementa
control product safety at all stages of
processing.

✓ **Quality management standard (ISO 9001-20**

✓ **Environmental management standard (ISO 14001)**

✓ *Laboratories accreditation (ISO 17025)*



In
with long-te
Our labora
meat products, fis
dairy products, cer
confectionery, fruit juice
testing for determining
parameters, provide giving revi

✓ **Microbiological parameters:**

➡ Total viable organisms

➡ Escherichia coli

➡ Pathogenic group (salmonella, S.aureus)

➡ Yeast and mould fungi, etc.

- Di
- Fattines
- Sugar
- Humidity
- Falling figure
- Gluten quality, quantity etc.

✓ **Safety parameters**

- Heavy metals
- Nitrates and nitrites
- Fusel oil, aldehydes
- Mycotoxins (aflotoxins, ochratoxin A, patulin etc.) determine and give corresponding review.

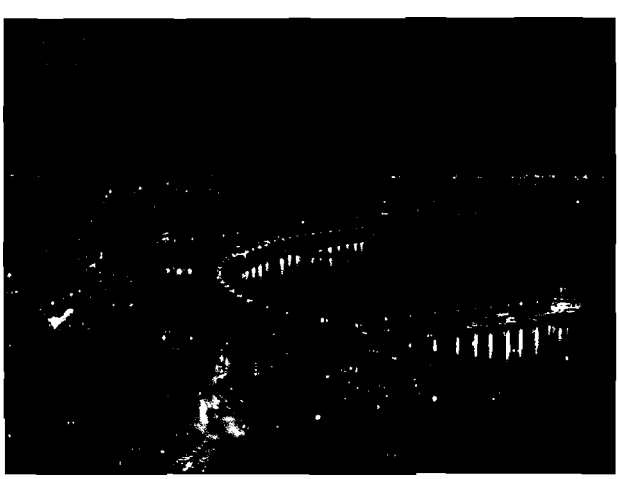
Our Bu

- All kinds o
- To make practic
- To improve company
- To prepare marketing and bu
- To enhance products quality
- To regulate conflict situations etc

The Bure
and internationa
protection, suppor
control establishments
ensuring projects etc. We
organizations: Health and
General European Commission
Agro center, GTZ Germany, TIKA Tur
for food safety and quality (IIFSQ)
projects together with these organizations.

The main purpose of executing such pr
protect consumer rights, to manage improve stu
food safety and expertise, to guaranty system form
foodstuff quality and safety.

We invite all the interested parties to participate
realization of project with great pleasure.



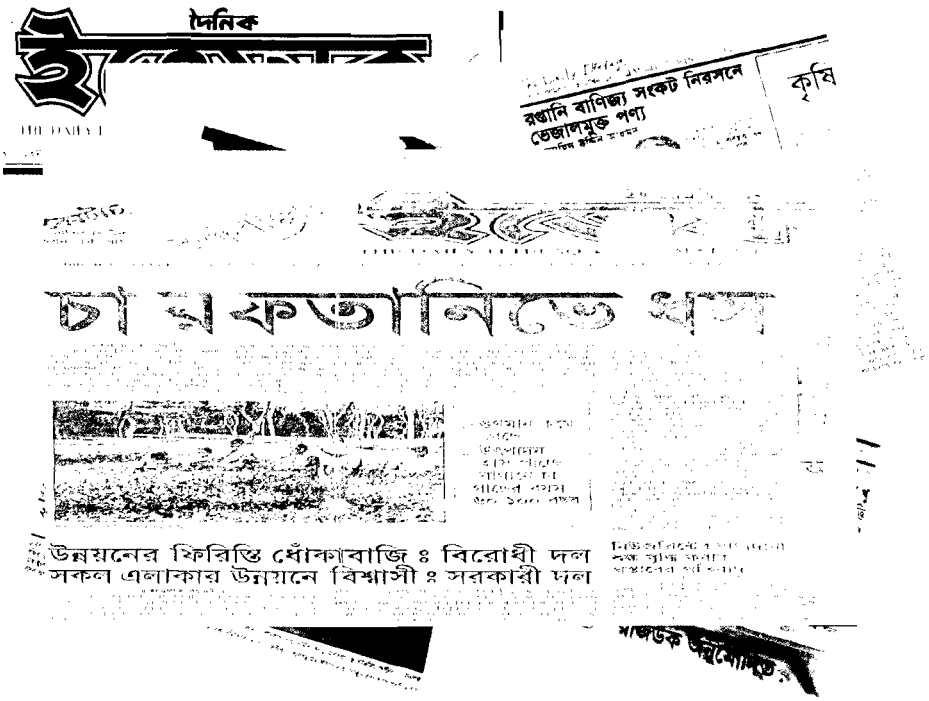
UNISWORK-VIII International Programme on Food Laboratories Management Practice

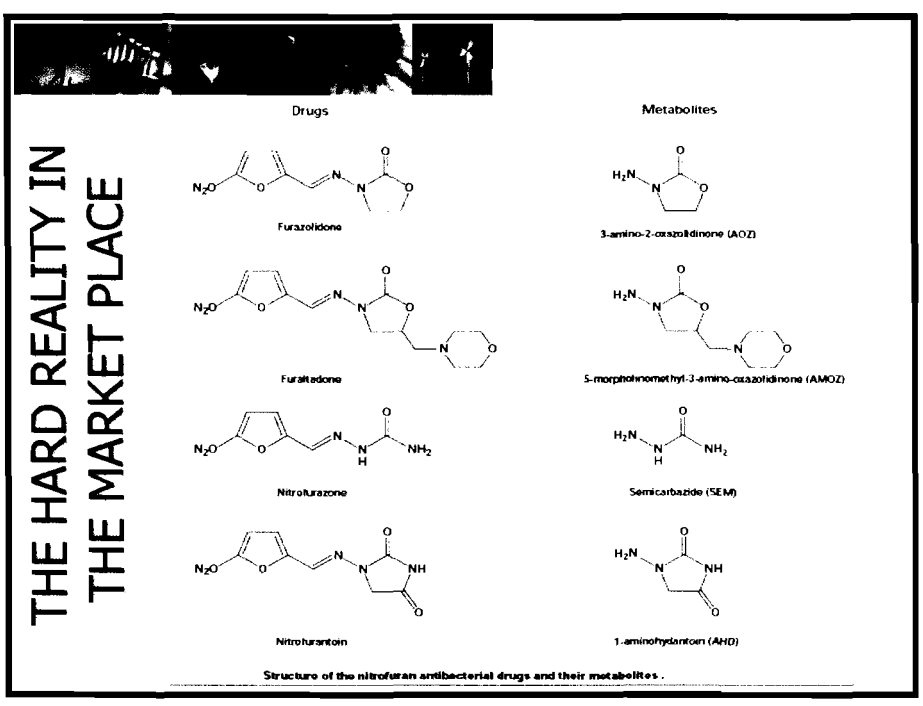
Jointly Organized by
TUBITAK- Marmara Research Centre (MRC), Food Institute (FSTRI)
United Nations Industrial Development Organization (UNIDO)

In Cooperation with
The Scientific & Technical Research Council of Turkey (TUBITAK)
Turkish International Cooperation & Development Agency (TIKA)
Turkish Ministry of Industry and Trade

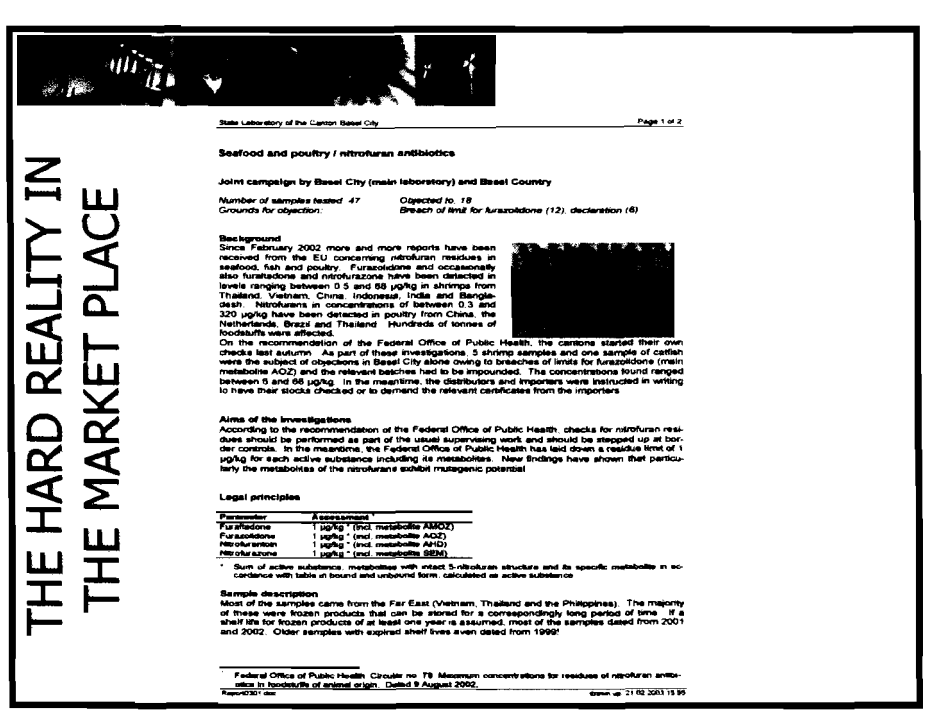
STATE OF FOOD QUALITY & CONFORMITY ASSESSMENT IN BANGLADESH

KM Mostafa Anwar
National Project Coordinator
Quality Management System Component
Bangladesh Quality Support Programme
United Nations Industrial Development Organization


দৈনিক
রপ্তানি বাণিজ্য সংকট নিরসনে
তেজালমুক্ত পণ্য
কৃষি
উন্নয়নের ফিরিস্তি ধোঁকাবাজি ঃ বিরোধী দল
সকল এলাকায় উন্নয়নে বিশ্বাসী ঃ সরকারী দল

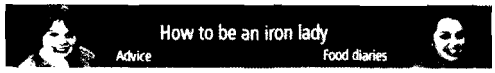


Structure of the nitrofuran antibacterial drugs and their metabolites .



THE HARD REALITY IN THE MARKET PLACE

FOOD STANDARDS AGENCY



> Homepage > News Centre > Press releases > Agency acts on products from SE Asia showing further illegal drug residues

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Agency acts on products from SE Asia showing further illegal drug residues

Friday, 15 March 2002

The Food Standards Agency has received the results of tests on a number of products of animal origin from China and South East Asia. The Agency began these tests as a result of concerns about a lack of control on the use of veterinary drugs in China. The latest results show further residues of illegal and unacceptable drug residues.

Illegal residues of the veterinary medicine chloramphenicol were found in samples of Royal Jelly, normally sold in capsule form, at similar levels to those recently found in honey. Independent scientific experts agreed that there was only an extremely low risk to public health through the consumption of products with residues at these low levels.

Given the low risk, the Agency is not advising against the consumption of Royal Jelly, but is calling for a withdrawal of Chinese and other Royal Jelly products, unless they meet legal requirements.

Further test results have also been received on two samples of Chinese rabbit meat. One is clear of all veterinary medicines and the other tested positive for residues of a pesticide. The levels were extremely low and while undesirable are not illegal and pose a negligible public health risk.

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TODAY'S FEATURES
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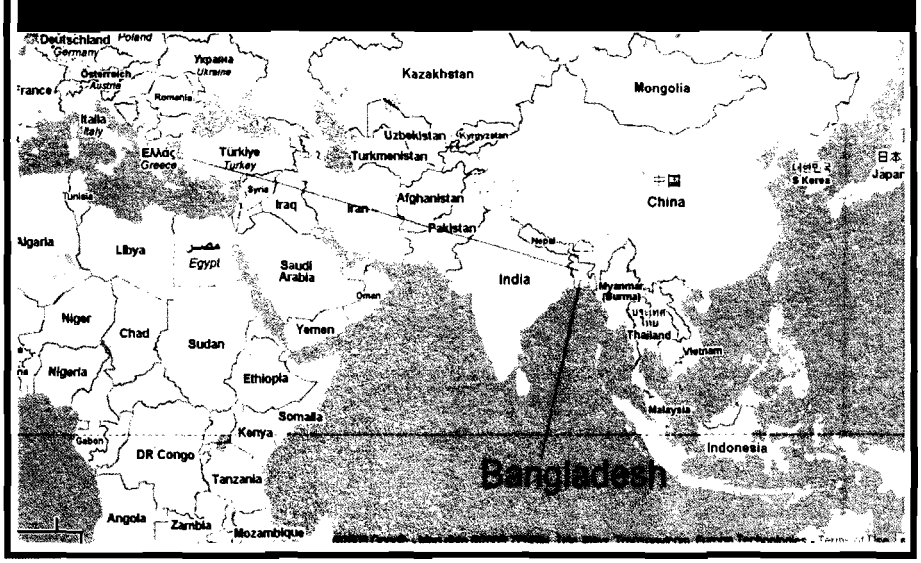
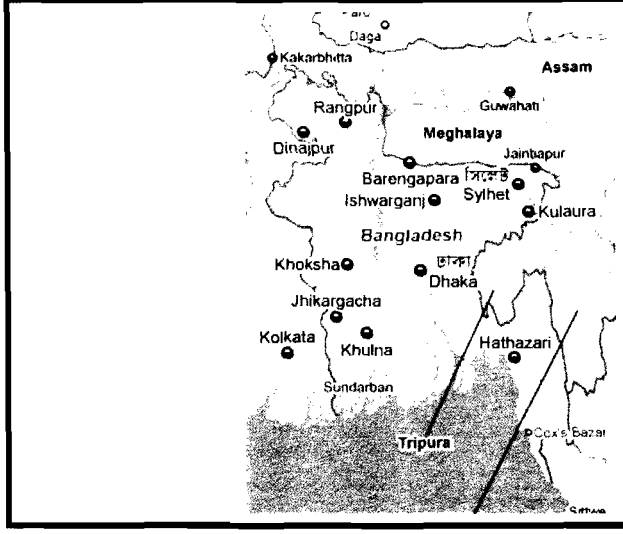
FOOD AND WEIGHT

- Bangladesh: Nature and Economy
- State of Regulatory Regime for Food Safety & Quality Control
- State of Technical Infrastructure for Standards, Testing & Certification
- Summary of Testing & Certification Requirements for Foods & Agro-based Products
- Challenges for Foods & Agro-based Industries: Setting up Acceptable System of Standards, Testing, Certification for Quality, Safety, Health & Environmental Compliance
- Way forward to Develop National Food Quality Assurance Infrastructure



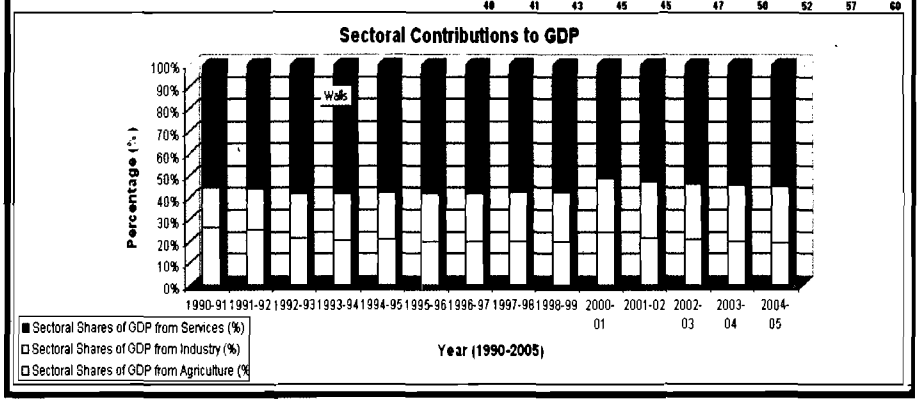


- Region: South of the Southeast Asia
- Position: Latitude: 20°34' - 26°38' N, Longitude:88°01' - 92°41' E
- Area: 147 570 sq. km.
- Climate: Sub-tropical monsoon with six distinct seasons. Temperature in winter: 7°C – 13°C in summer: 24-31°C. Max temp recorded in summer 37°C or 41°C. Monsoon July – October, 80% rainfall in monsoon. In monsoon the average rainfall 1429 - 4338 mm whereas in winter rainfall is ~100 – 200 mm or less.
- Population estimated (June 2009): 144.2 million
- Density of Population: 720/sq.km. (the highest in the world)

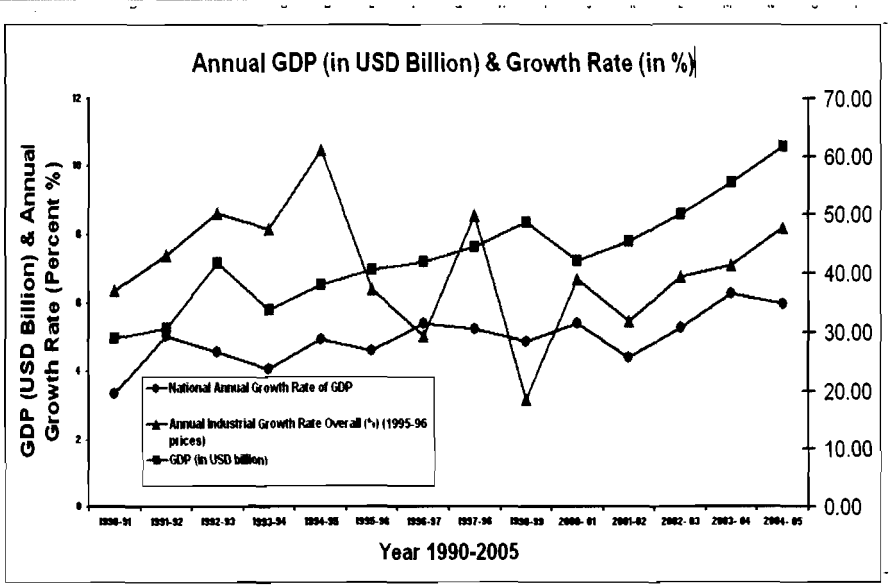
- GDP in Current Price: 89.51 billion USD
- GDP Growth Rate (estimated in June 2009): 5.9%
- Agriculture Contribution to GDP: ~ 20.6%
- Industry Contribution to GDP: ~ 26.0% of GDP
- Service Sector Contribution to GDP: 49.7%
- Export (Jul 08-March 09): 11.63 billion USD
- Import (Jul 08-March 09): 15.47 billion USD
- Per Capita National Income : 690 USD
- Per Capita GDP: 621 USD

BANGLADESH : FACT SHEET

	1990-91	1991-92	1992-93	1993-94	1994-95	1995-96	1996-97	1997-98	1998-99	2000-01	2001-02	2002-03	2003-04	2004-05
Population (in million)	111.00	113.3	115.5	117.7	119.9	122.1	124.3	126.5	128.2	128.1	131.00	133.40	136.2	137.00
GDP (in USD billion)	29.00	30.65	41.70	33.00	38.13	40.57	42.02	44.40	46.02	52.25	48.53	50.00	55.43	61.70
Sectoral Shares of GDP from Agriculture (%)	28.57	28.57	21.60	20.00	21.17	20.32	20.22	18.90	20.20	24.00	21.00	21.00	20.20	19.30
Sectoral Shares of GDP from Industry (%)	17.56	18.64	20.52	21.30	21.00	21.04	22.21	22.5	22.34	24.40	29.50	29.30	29.50	29.10
Sectoral Shares of GDP from Services (%)	53.30	52.70	57.70	58.03	57.24	57.04	57.57	57.12	57.37	51.00	52.00	53.70	54.30	54.00
National Annual Growth Rate of GDP	2.34	5.69	4.57	4.69	4.53	4.52	5.30	5.22	4.00	6.11	4.42	5.20	6.27	6.90
Annual Industrial Growth Rate Overall (%) (1995-96 price	6.26	7.30	6.62	6.10	10.40	6.41	6.05	6.54	3.10	6.60	5.40	6.70	7.1	6.10
Large (%)	6.62	7.24	9.00	6.25	11.44	5.57	3.97	5.25	4.10	6.05	4.6	6.50	6.06	6.3
SME (%)	7.28	7.40	7.70	7.06	8.1	6.20	7.75	6.77	6.75	7.62	7.69	7.21	7.45	7.52
						40	41	43	45	45	47	50	52	57	60



LOOK AT BANGLADESH



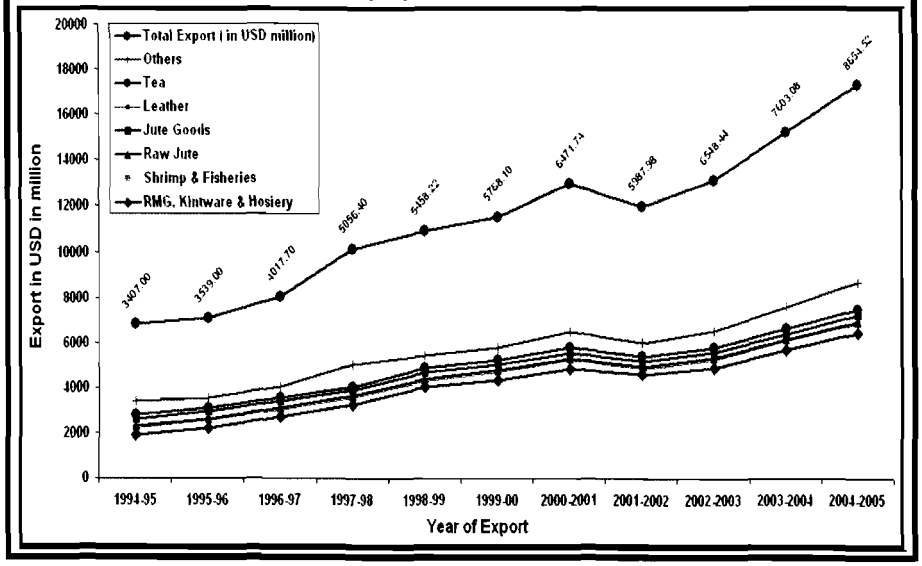
LOOK AT BANGLADESH

EXPORT SCENARIO OF BANGLADESH INTERMS OF PRINCIPLE GOODS (1994-2005) (in USD million)

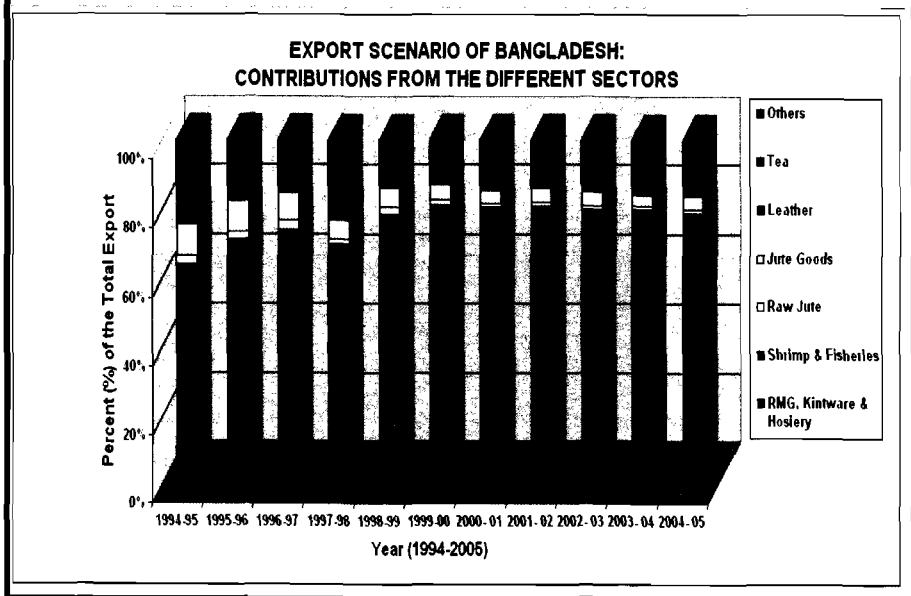
Principle Goods	1994-95	1995-96	1996-97	1997-98	1998-99	1999-00	2000-01	2001-02	2002-03	2003-04	2004-05
RMG, Kintware & Hosiery	1873.3	2210.9	2688.4	3253.7	4020.1	4352.4	4060.1	4583.90	4912.10	5686.09	6417.67
Shrimp & Fisheries	321.2	323.7	297	285.1	274.3	343.8	363.2	276.11	321.81	390.25	420.74
Raw Jute	79.1	72.7	101.7	83	111.82	72.1	67.10	61.13	82.46	79.7	96.19
Jute Goods	305.6	312.6	313.5	269.8	303.8	265.9	233.6	243.5	257.10	245.6	306.53
Leather	201.6	149.6	125.7	106	168.2	195.1	253.93	207.33	191.23	211.41	220.93
Tea	32.7	30.7	32.3	47.4	33.6	15.9	21.50	17.30	15.47	15.81	15.84
Others	593.5	438.8	459.1	1011.4	546.4	522.9	672.15	598.73	768.19	974.22	1176.62
Total Export (in USD million)	3407.00	3539.00	4017.70	5056.40	5458.22	5768.10	6471.74	5987.90	6548.44	7603.08	8654.52
Total Export (in BDT million)	136970	144520	171550	229410	245620	247420	306400	302120	337890	437100	519271.2
USD Conversion Rate with BDT	40	41	43	45	45	43	47	50	52	57	60

LOOK AT BANGLADESH

EXPORT SCENARIO OF BANGLADESH: 1994-2005

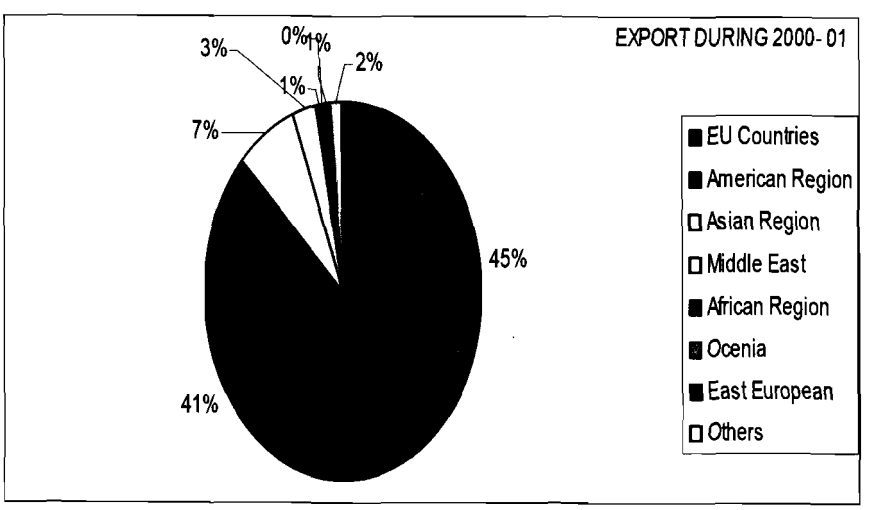


LOOK AT BANGLADESH



LOOK AT BANGLADESH

EXPORT IN DIFFERENT REGIONS OF THE WORLD



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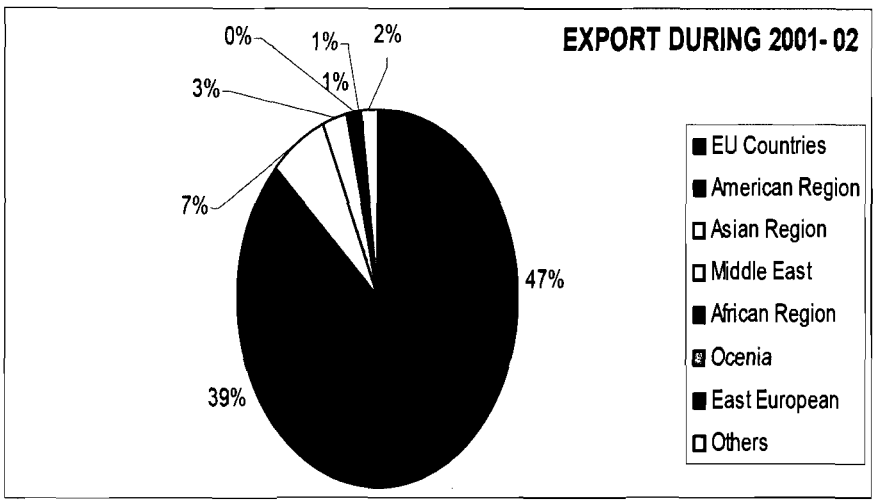
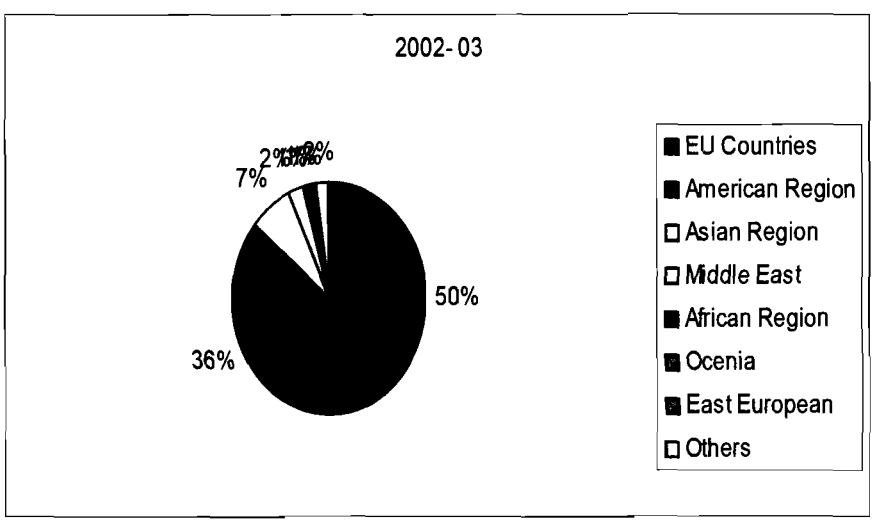
EXPORT IN DIFFERENT REGIONS OF THE WORLD



LOOK AT BANGLADESH

EXPORT IN DIFFERENT REGIONS OF THE WORLD



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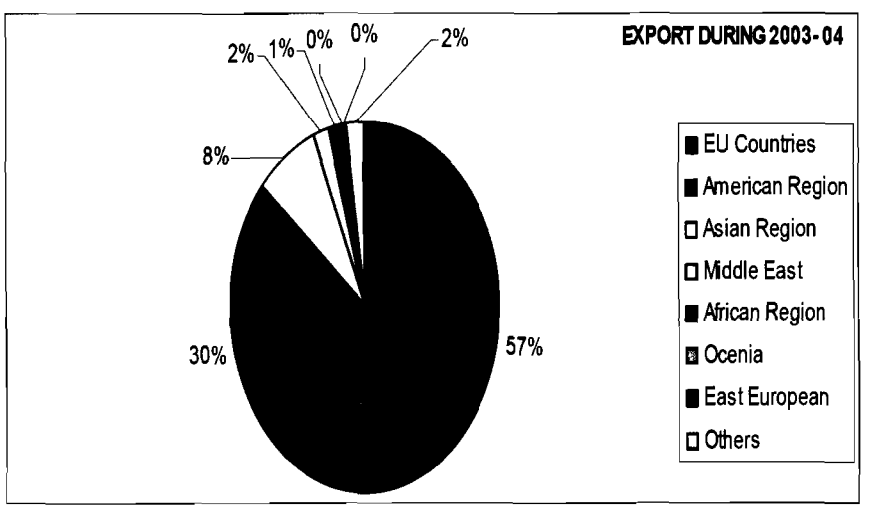
EXPORT IN DIFFERENT REGIONS OF THE WORLD



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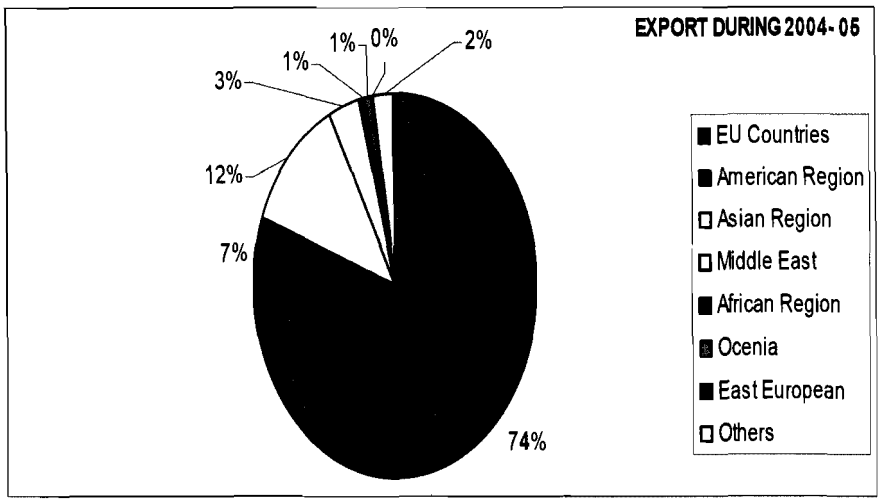
EXPORT IN DIFFERENT REGIONS OF THE WORLD



STATE OF REGULATORY REGIME

Quite a Large number of Laws and Regulations Empowered More than 13 Different Ministries & Departments but Enforcement for Assuring Food Quality & Safety is REALLY scanty. NO CETRAL COORDINATING & REGULATORY BODY TO LOOK AFTER THE NATIONAL FOOD QUALITY ASSURANCE. Setting up a **Bangladesh National Food Administration (BNFA)** to be a food regulatory authority has been proposed by the author for many years (Anwar et.al., 2003)

CONSTITUTION OF BANGLADESH

- Article 15 of the Bangladesh Constitution states that it shall be a fundamental responsibility of the State to secure provision of the basic necessities of life, including food.
- Article 18(1) of the Constitution states that the State shall raise the level of nutrition and improve public health as among its primary duties.

[Both the Articles imply food safety requirements for consumers and the State must ensure it through enactment of appropriate laws and regulations. However, these provisions of the Constitution cannot be enforced by the Court]

REGULATORY REGIME

YEAR	LEGISLATIONS RELATED TO FOOD SAFETY & QUALITY
1860	**Sections 273-274, Bangladesh Penal Code, 1860**
1950	**Fish Protection & Conservation Act, 1950**
	Agricultural Products Market Act, 1950
1956	**The Food or Special Courts Act, 1956**
	The Food Grain Supply (Prevention of Prejudicial Activity) Ordinance, 1956
	The Food Grain Supply (Prevention of Prejudicial Activity) Ordinance, 1956
1957	**The Essential Commodity Act, 1957**
1958	**The Essential Commodity Act, 1958**
1959	**The Bangladesh Pure Food Ordinance, 1959**
1964	**The Essential Commodity Act, 1964**
	Agricultural Produce Market Act, 1964 (revised in 1985)
1966	**Destructive Insects and Pests Rules (Plant Quarantine), 1966 (amended up to 1989)**
1967	**The Bangladesh Pure Food Rules, 1967**

REGULATORY REGIME

YEAR	LEGISLATIONS
1971	**The Pesticide Ordinance, 1971**
1974	**The Special Power Act, 1974**
1977	**The Paurashava Ordinance, 1977 (Ordinance No. XXVI of 1977), Part IV, Chapter III Articles of Food and Drink**
1983	**The Dhaka City Corporation Ordinance,1983 (Ordinance No. XL of 1983), Part IV, Chapter III Articles of Food and Drink**
1983	**The Animals Slaughter (Restriction) and Meat Control (Amendment) Ordinance, 1983**
	Marine Fisheries Ordinance, 1983
	Marine Fisheries Rules, 1983
	Fish & Fish Products (Inspection & Quality Control Ordinance, 1983
1985	**The Bangladesh Standards and Testing Institution Ordinance, 1985 (has been amended as BSTI (amendment) Act, 2003)**
	The Pesticide Rules, 1985
	Agricultural Product Market (Revised) Act, 1985

REGULATORY REGIME

YEAR	LEGISLATIONS
1990	The Essential Commodity Act, 1990
1995	Fish Protection & Conservation (Amendment) Act, 1995
1997	Fish & Fish Products (Inspection & Quality Control) Rules, 1997 (Amended in 2008)
2005	Pure Food Act, 2005
2006	Bangladesh Accreditation Act, 2006
2007	Packaged Commodities Rules, 2007
2009	The Consumers' Right Protection Act, 2009
	SOME POLICIES & ORDERS FROM THE GOVERNMET
2006	**National Food Safety Policy, 2006**
2006	**Export Policy Order 2006-2009**
2006	**Import Policy Order 2006-2009**

NATIONAL REQUIREMENTS

❑ Phytosanitary certificate: Plant Protection Wing (PPW) of Department of Agricultural Extension, Ministry of Agriculture only regulatory body to issue the Certificate

❑ Health certificate: 'as *an evidence of its fitness for human consumption'*, based on the tests:

- Poisonous contaminant test: As, Pb, Cr, Mercury, Cd etc.
- Pathogen test: E. Coli, Salmonella, Shigella, Pseudomonous, St. Aureus, etc.
- Total microbial count: Total bacteria, fungus etc.

✓Certificates Accepted from: Institute of Food Science & Technology (BCSIR) within Ministry of Science, Institute of Public Health (IPH), Institute of Nutrition & Food Science of Dhaka University, International Centre for Diarrheal Diseases & Research, Bangladesh ICDDR,B)

NATIONAL REQUIREMENTS

❑ Conformity Certificate: Compliance with 54 BDS Standards/ Technical Regulations for foods mandatory. Certification Mark License from Bangladesh Standards & Testing Institution (BSTI) MUST be obtained by local producers as well as by the importers

❑ Nutrition Information Test: Energy, Protein, Carbohydrate, Fat, Cholesterol, Sugar, Dietary Fiber, Calcium, Sodium, Iron, Mineral, Magnesium, Phosphorus, Thiamine, Potassium, Riboflavin, Nicotine, Vitamin-A, Vitamin-B1, Vitamin-B6, Vitamin-12 etc. should be declared on the product labels

✓Tests Accepted from: Institute of Food Science & Technology (BCSIR) within Ministry of Science, Institute of Public Health (IPH), Institute of Nutrition & Food Science of Dhaka University

NATIONAL REQUIREMENTS

❑ Radioactivity Test: Level of radioactivity contamination is checked at the Port of Entry (POE) in cereal, corn, milk and milk-based products

✓Test Accepted ONLY from: Bangladesh Atomic Energy Commission (BAEC) within Ministry of Science

❑ Irradiation to food & agro-based products: Appropriate dose of ionizing radiation to agro-based food products applied for preservation of food, control of sprouting agricultural products, e.g. potato, onion vegetables etc, and control of food borne diseases. It destroys or inactivates organisms that cause spoilage thereby extending shelf-life of certain foods.

✓Applied ONLY by: Bangladesh Atomic Energy Commission (BAEC) within Ministry of Science

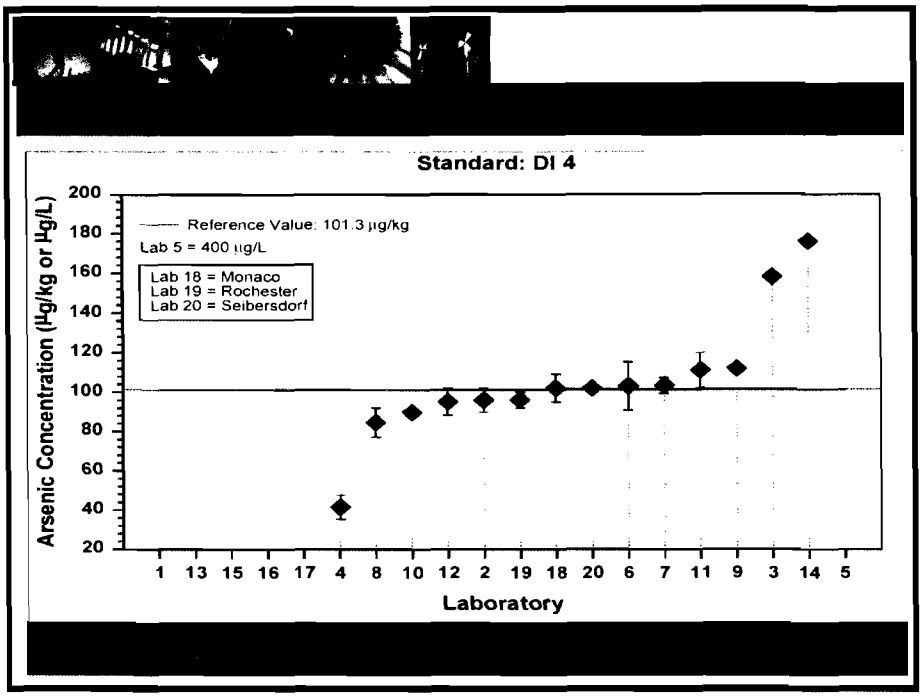
EU & INTERNATIONAL MARKET REQUIREMENTS & CHALLENGES FOR CREATING THE TECHNICAL ARRANGEMENT In Assuring Food Quality, Safety, Health & Environment

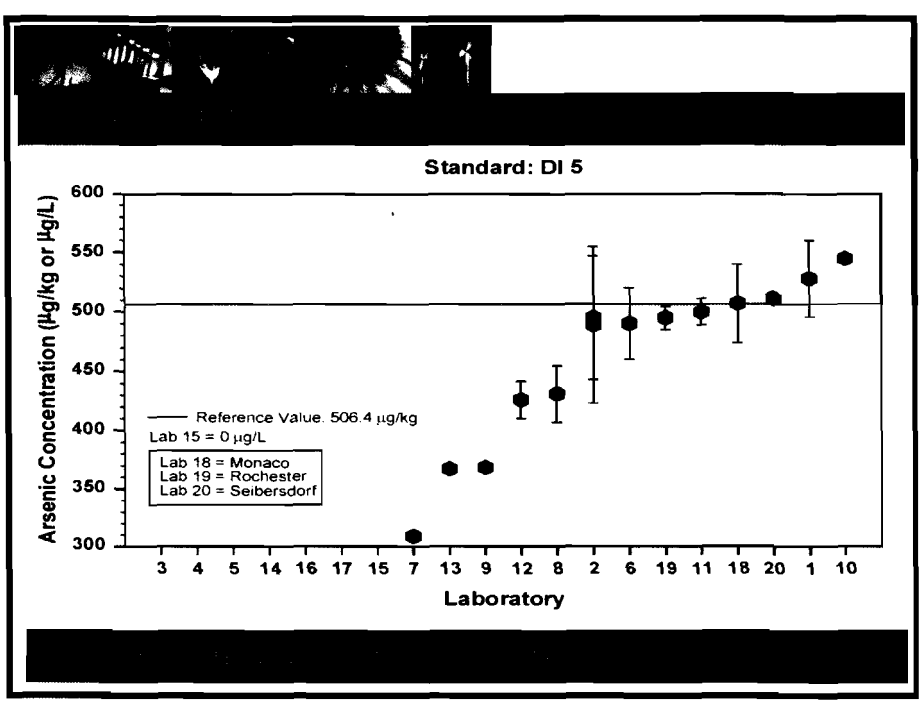
CHALLENGES?

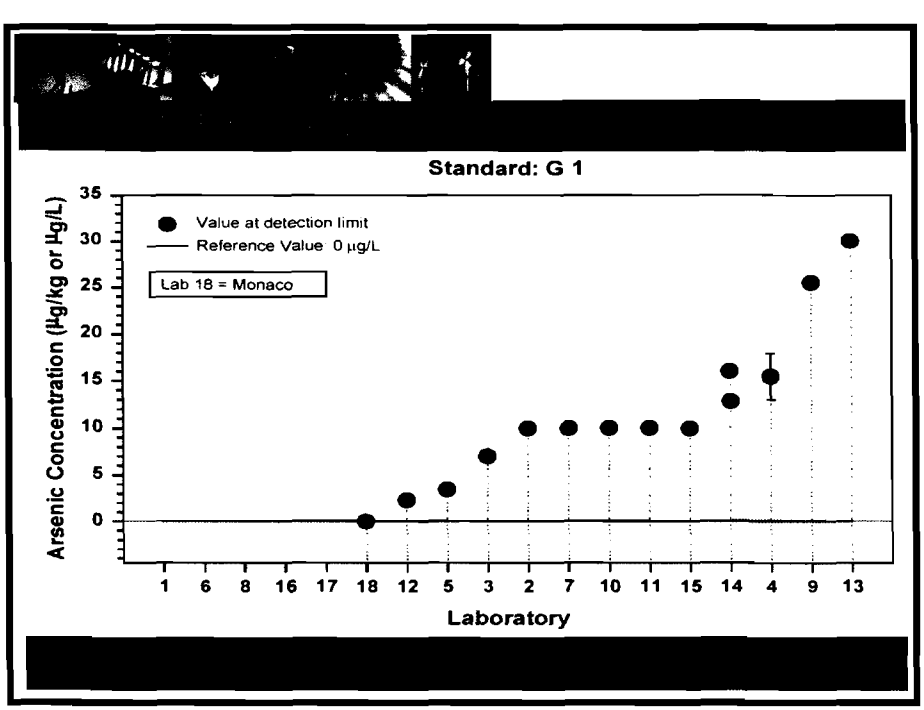
- Non acceptance of test results from the national laboratories due to lack of confidence and lack of accreditation. Poor understanding on ISO/IEC 17025
- Costly re-testing of products in the laboratories of importing countries and at the Port of Entry (increases lead time, food products may spoil)
- Non acceptance of certification mark of the national product certification bodies e.g. BSTI in case of exporting: not having the product certification system as per ISO/IEC Guide 65
- Not meeting the requirements of packaging and labeling

CHALLENGES?

- Absence of national calibration laboratories with traceability to (SI): BSTI NMI established under technical assistance action BQSP from EU-UNIDO-NORAD started providing a limited number of calibration services from August 2009. Not yet accredited. One calibration lab is NOT enough to cover ALL fields of measurements
- No proper institutionalized arrangements for organizing proficiency testing (PT) / Inter-laboratory Comparison (ILC) on regular basis to support accreditation. ICSL – BCSIR under Ministry of Science establishing the nation's first Designated National Reference Laboratory aiming to create these services in Metrology in Chemistry
- Difficulty in obtaining Certified Reference Materials CRMs. ICSL – BCSIR under Ministry of Science establishing the nations first Designated National Reference Laboratory aiming to produce and disseminate matrix matched CRMs to provide traceability in chemical measurements


Standard: DI 4
Arsenic Concentration (µg/kg or µg/L)
Reference Value: 101.3 µg/kg
Lab 5 = 400 µg/L
Lab 18 = Monaco
Lab 19 = Rochester
Lab 20 = Seibersdorf
200
180
160
140
120
100
80
60
40
20
1 13 15 16 17 4 8 10 12 2 19 18 20 6 7 11 9 3 14 5
Laboratory


Standard: DI 5
Arsenic Concentration (µg/kg or µg/L)
Reference Value: 506.4 µg/kg
Lab 15 = 0 µg/L
Lab 18 = Monaco
Lab 19 = Rochester
Lab 20 = Seibersdorf
600
550
500
450
400
350
300
3 4 5 14 16 17 15 7 13 9 12 8 2 6 19 11 18 20 1 10
Laboratory


Standard: G 1
Arsenic Concentration (µg/kg or µg/L)
Value at detection limit
Reference Value: 0 µg/L
Lab 18 = Monaco
35
30
25
20
15
10
5
0
1 6 8 16 17 18 12 5 3 2 7 10 11 15 14 4 9 13
Laboratory

CHALLENGES IN EXPORTING FOOD & AGRO-BASED PRODUCTS

- Quality Management System (ISO 9000)
- Environment Management System (ISO 14000)
- Hazard Analysis & Critical Control Point HACCP
- Food Safety Management System (ISO22000)
- Social Accountability Standard SCR ISO 26000
- Occupational safety standards (OHAS) etc.
- EU Directive for Traceability EC 178/2002 – 2005
- EU Directives for Residue Control in Foods 96/23/EC, 2003/657/EC
- Compliance with Standards BRC, GAP

CHALLANGES IN WATER & ENVIRONMENTAL QUALITY : HAZARDS ENTERING INTO FOOD CHAIN

❑ Arsenic, Chromium, Mersury, Cd, Lead, Barium and other heavy metals

❑ Residual Pesticides and other Persistent Organic Pollutants are now contaminating the ground water and both surface water.

❑ Thousands of Hospitals, medical diagnostic laboratories, Clinics are releasing hospital garbage and chemicals

❑ Industrial Wastes and poly-bags, plastics/ pvc and non-biodegradable materials are released

e.g. SHRIMP & FROZEN FOODS TO EU MARKET

❑ Suffering for absence accredited testing, certification and inspection System: to meet the stringent EU Regulations 96/23/EC for controlling
  - Veterinary Drug Residues
  - Antibiotics
  - Pesticide Residues
  - As, Cr, Cd. Lead, Mercury and other trace metals
  - Microbial Contaminations

❑ Packaging & Labeling Requirements

❑ Environmental & Social Compliance

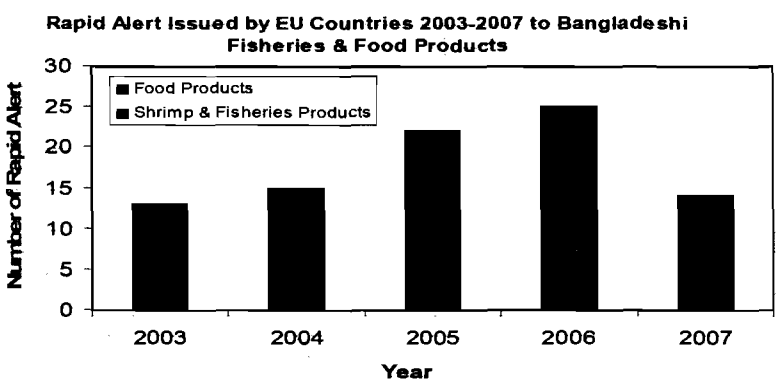
❑ SPS Requirements: EU Rapid Alerts System (RASFF) issued a quite large number of notifications from 1997. Many consignments: shrimp, fisheries, foods, drinks and agro-based rejected

EXAMPLES OF REQUIREMENTS FOR EU MARKET (96/23/EC, 2003/657/EC):

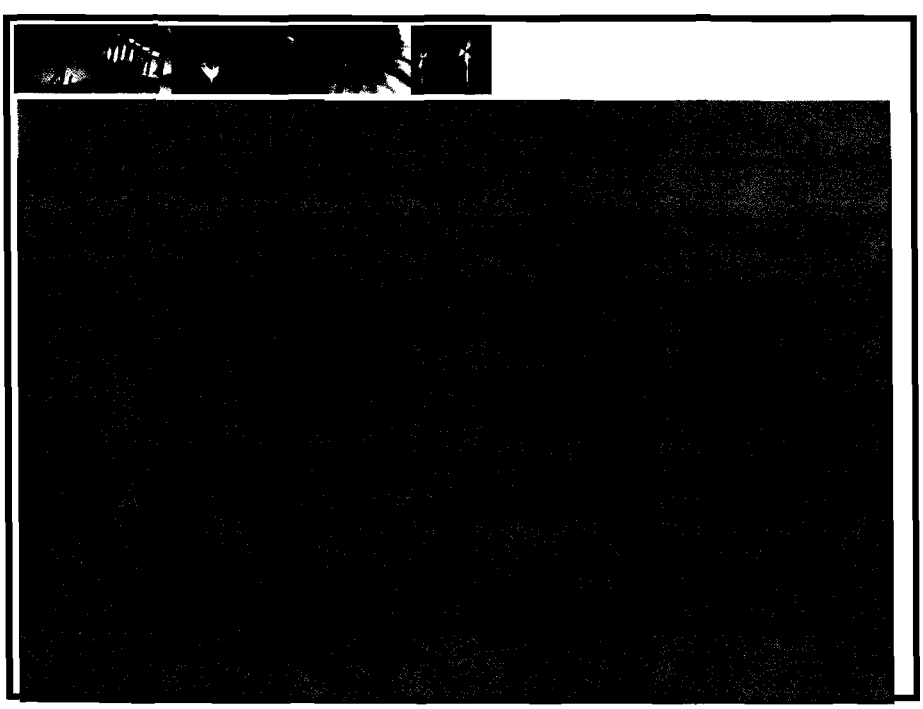
- For Food Products:
  - No Nitrofuran metabolites: SEM AOZ AMOZ AHD (ban veterinary drugs)
  - No Chloramphenicol CAP (ban antibiotics), crystal violate,malachite green
  - Maximum Residue levels (Aldrin, DDT, Chlordane, Endrin, HCH, HCB)
  - Maximum Cadmium, lead, Arsenic, mercury levels
  - Polychlorinated Biphenyls (PCB), Terphenyles (TCB)
  - Pathogenic micro-organisms: salmonella, protozoa
  - Irradiation of Food
  - Packaging Requirements
- Other System Requirements
  - HACCP, ISO 14000, ISO 9000, ISO 22000, ISO 26000, OSHA
  - International label for fish from -Marine Stewardship Council: Eco-labeling
  - Environmentally Sound Production (ESP

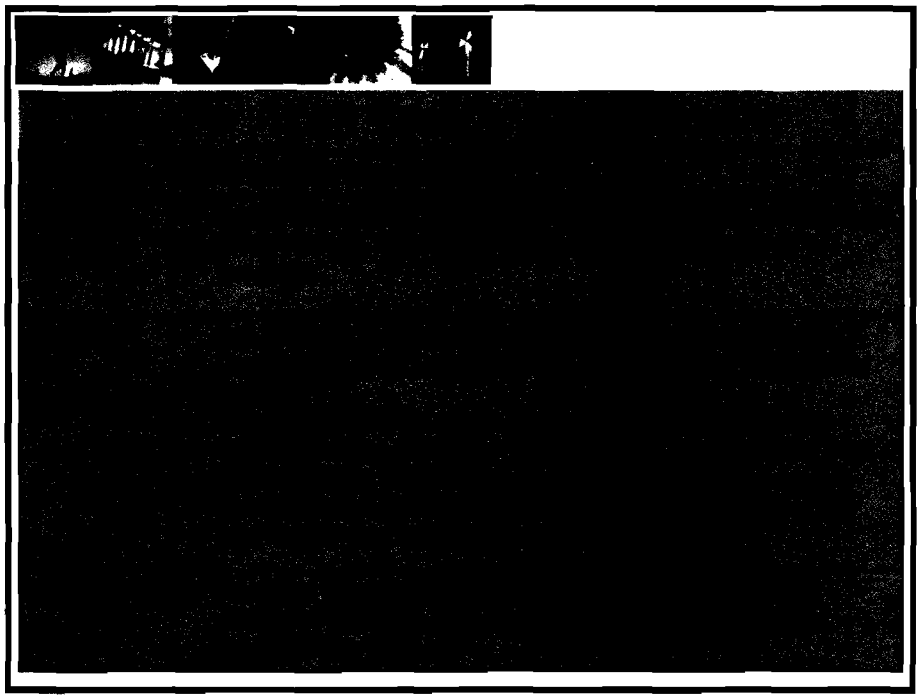
EU RAPID ALERT NOTIFICATIONS (2003-2007)

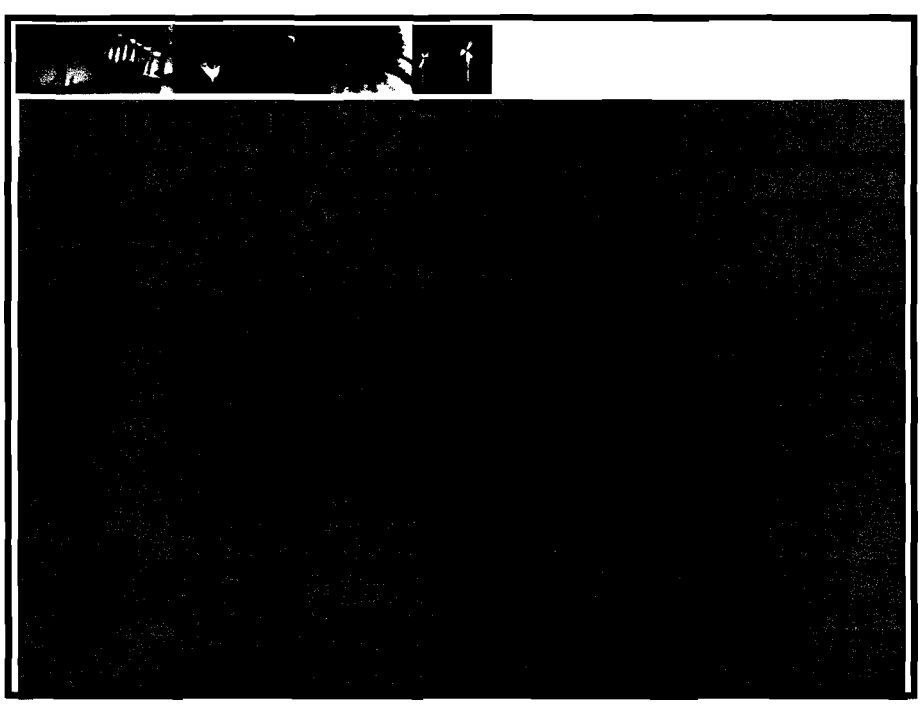
Shrimp & Fisheries Products	11	11	18	23	5
Food Products	2	4	4	2	9
Total Rapid Alert Issued	13	15	22	25	14

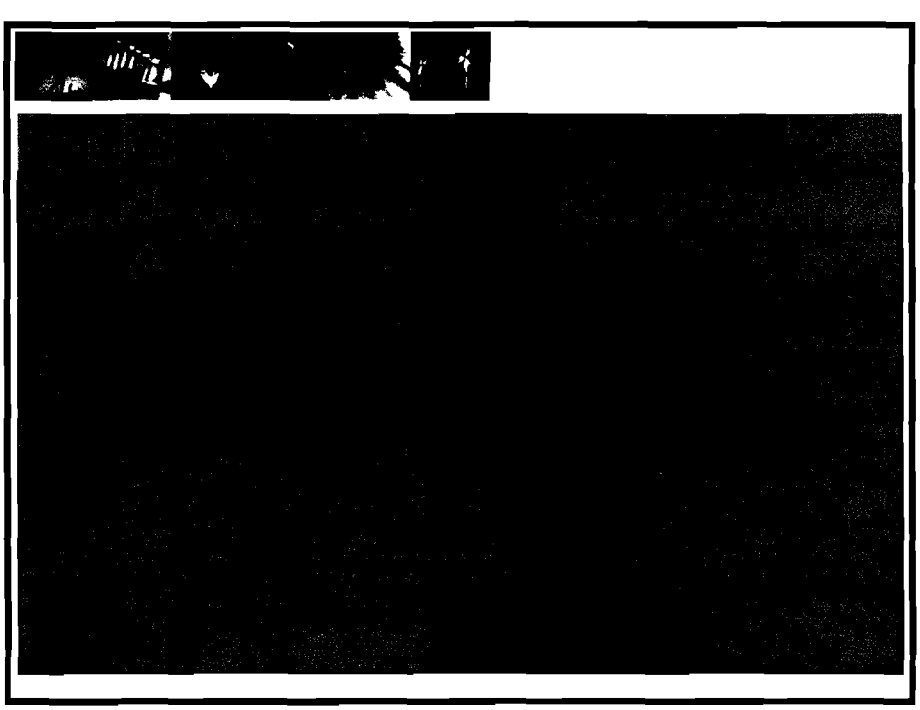


Note: * Weekly Rapid Alert Reports began in June 2003







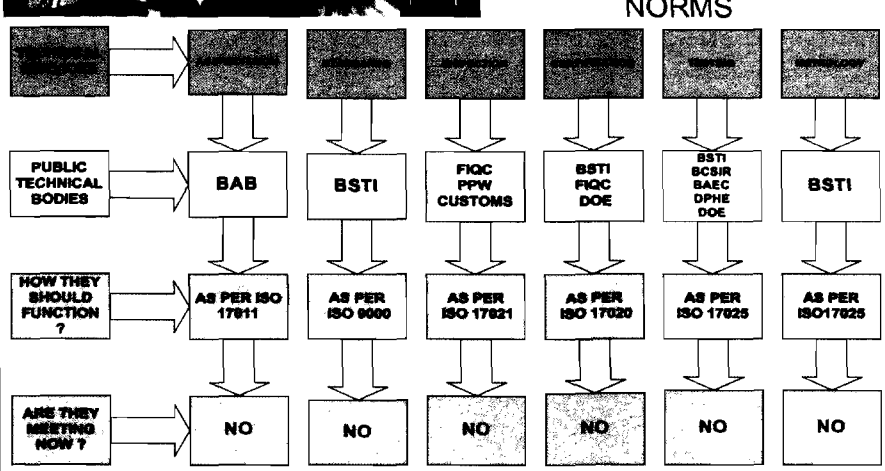


TECHNICAL ARRANGEMENT:

Standards Regime & Conformity Assessment Infrastructure are at infancy state and NOT yet in compliance with international standards and North

Body	National	International	Total
Accreditation	1 (BAB)	>3 (NABL, UKAS & other)	> 4
Standards	1 (BSTI)	0	1
Metrology	BSTI (NMI) & Legal Metrology	0	1
Product Certification	4 (BSTI CM, FIQC, PPW etc.)	~2 (SGS, BV)	~6
Management System Certification	1 (BSTI MSC)	~ 14 (URS, BVQ, Orion, DNV, TÜV etc.)	~15
Inspection	3 (FIQC, PPW: Quarantine, Boiler Inspt.)	~6 (SGS, BVI, Lloyd etc.)	~9
Food Quality Testing & Calibration	>300 (1 calibration lab ICSL BCSIR): BSTI, BCSIR, BAEC, IPH, DU, BUET, FIQC, DPHE, SRDI, BFRI, BLRI etc.	4 (SGS, BV, ITSL, etc.	> 300

STATUS TO INTERNATIONAL NORMS



The Conformity Assessment Bodies IN FOOD QUALITY CONTROL are NOT FULLY in compliance with International Standards & Requirements

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **STANDARDS: BSTI Bangladesh Standards & Testing Institution**
  - Officially the only national standards body (Ordinance XXXVII of 1985 and subsequent amendments in 2001)
  - The *custodian of the national physical standards* and responsible for disseminating the traceability in measurements upto SI Units (Weights and Measure Ordinance 1982 and subsequent amendments).
  - Enforcing the legal metrology and to control the pre-packaged commodities

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **STANDARDS:** Bangladesh Standards & Testing Institution
  - Conducting tests for product certification marking (CM)
  - BSTI is the full member of ISO, Affiliate member of IEC, participating SAARC Regional Standards Organization SARSO, holding the TBT NEP and also the national focal point for CODEX.
  - More than 3000 BDS standards developed, adopted and or harmonized as national standards. But implementation of voluntary standards is almost absent
  - ~54 BDS Standards (out of 151) are indeed to be mandatory technical regulations for Certification Mark Licensing
  - More than 150 CODEX standards have been harmonized

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **IN REALITY BSTI**
  - Is not a truly functional national standards body. Instead, BSTI acts as a regulator to control a large number (151) items/ products under mandatory certification marking (CM) scheme as such to issue/control 13000 CM Licenses.
  - It centralized a huge responsibilities in paper without effective implementation due to limited capacity.
  - Not having proper technical arrangement. Not having the updated knowledge and no research within the institution

STATE CONFORMITY ASSESSMENT INFRASTRUCTURE

- **IN REALITY BSTI**
  - Is rigid in bringing change within the organization in line with the best practices. Lack of transparency and governance almost paralyzed the organization.
  - Is not effectively participating in the international activities in standards, metrology etc.
  - Reluctant to cooperate/collaborate with other national expert technical institutions to develop the partnership based national infrastructure in metrology/calibration with traceability up to SI Units

- **ACCRDITATION**
  - **Bangladesh Accreditation Board BAB newly formed (by Act 29, 16 July 2006) at its infancy stage: It will take some time to be fully functional and to enter into ILAC MRA. EU funded UNIDO implemented Bangladesh Quality Support Programme BQSP providing technical assistance to operationally BAB. Long way to go.**
  - **BAB become Associate Member of APLAC in 2007.**
  - **Norwegian Accreditation (NA) has been engaged by UNIDO for supporting BAB to assist up to ILAC MRA.**
  - **Various training courses on ISO 17025 are conducted in the last 3 years to develop the assessor pool for BAB and laboratory quality managers and internal auditors.**

- **ACCRDITATION**
  - Huge awareness /training activities operational by BQSP UNIDO
  - BAB Quality system as per ISO/IEC 17011 is expected to be deployed.
  - Only 14 multinational testing/inspection/Certification bodies (SGS, BVQ, BVI etc.) at present received accreditation from NABL India, UKAS, RvA

- **METROLOGY**
  - **BSTI is mandated to maintain the national physical standards, to provide traceability upto SI Units.**
  - **BSTI is also responsible to enforce legal metrology (Weights and Measure Ordinance 1982 and subsequent amendments) to control the selected measuring instruments for trade and commerce. BSTI is also to control the pre-packaged commodities under legal metrology.**
  - **EU- UNIDO Bangladesh Quality Support Programme BQSP has supplied equipment (USD1.12million) for BSTI Metrology and imparting technical assistance (2006-2009) to establish the physical metrology lab to provide calibration in mass, temperature, volume, density, length, viscosity, pressure, frequency and time. Appropriate environment control system established. Accreditation would be as per ISO/IEC 17025.**

- **METROLOGY**
  - At this beginning stage no primary standards are being established.
  - But Calibration capacity developed under BQSP is not adequate and BSTI Metrology wing does not have technical/ institutional capacity at this moment to implement the metrology programme throughout the country to meet the existing industrial demand which is huge indeed.
  - Only under legal metrology 10.3 million (!) measuring equipment are to be calibrated each year which is an impossible task for BSTI alone.

- **METROLOGY**
  - BSTI does not have experience in chemical metrology and also is reluctant to buildup a partnership based system for metrology in chemistry program in the country with other expert technical institutions who have already experience in measurements in chemistry in different fields.
  - BSTI is participating Asia Pacific Metrology Program (APMP).
  - Bangladesh is neither a signatory to the Meter Convention, nor at least the associate member of BIPM as such not yet entering to CIPM MRA.
  - Existing Technical and financial capacity is severely limiting BSTI to take part to BIPM activities

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **TESTING & CALIBRATION**
  - More than 10,000 testing laboratories (including medical diagnostic) operating in the country mostly in public sector
  - Nearly 70 laboratories are involved in testing foods, drinks, water, pharma and agro-based products without accreditation
  - (01) One laboratory within BCSIR, Ministry of Science just received accreditation to test 04 chemistries of drinking water
  - Only a few (less than 15) are in private sector
  - SGS, BVQ, ITSL are multinational accredited labs not testing foods

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **TESTING & CALIBRATION**
  - ***Instrumentation & Calibration Service Laboratory ICSL BCSIR:*** For the first time to initiate Metrology in Chemistry (MiC): national accredited CRM program, to operate regular national accredited PT /ILC program and also to provide accredited instrumentation calibration services, the first National Reference Laboratory for Chemical Measurements is under construction by the Government within ICSL BCSIR (Ministry of Science Information & Communication Technology). This project would be primarily focused on foods & pharmaceuticals applications only. ICSL BCSIR is willing to be the "Designated National Reference Laboratory for Chemical Measurements in Food & Pharma" by the Government as such to be a partner of NMI activities. This project is being implemented in July 2008-June 2012.

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **TESTING & CALIBRATION**
  - Within the other national expert institutions several similar Designated Reference Laboratories for other chemical measurements should be established following the decentralized partnership/network based model upon assessing the national needs framing the National Strategy for Developing Chemical Metrology Infrastructure.
  - ICSL BCSIR & Ministry of Science and Information & Communication Technology prepared the first ***National Strategy for Chemical Metrology 2009-2021***

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **FOOD PRODUCT CERTIFICATION**
  - BSTI (Bangladesh Standards & Testing Institution): Regulates 54 I food items under mandatory product certification marking scheme (CM).
  - Product Certification for Voluntary food standards: yet to be developed
  - FIQC (Fish Inspection & Quality Control) under Deptt of Fisheries DoF: Controls Fisheries Products also competent authority by EU

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **FOOD PRODUCT CERTIFICATION**
  - Plant Protection Wing of Deptt. of Agriculture: provides SPS certifications
  - BAEC (Bangladesh Atomic Energy Commission BAEC): provides certification ionizing radiation emitting devices
  - BCSIR (Bangladesh Council of Scientific & Industrial Research): certifies Arsenic Mitigation Technologies to be used to treat drinking water

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **MANAGEMENT SYSTEM CERTIFICATION**
  - Around 14 multinationals are operating to provide management, environmental, system and social compliance certifications for ISO9000, ISO14000, ISO22000, HACCP, WRAP, SA8000 etc.
  - These above are all accredited by ILAC/IAF Members : NABL, NABCB, UKAS, RvA, DnV
  - BVQ, SGS, Orion Register, URS, RdeR, Moodi International, AJA, SAI Global etc.

STATE OF CONFORMITY ASSESSMENT INFRASTRUCTURE

- **INSPECTION**
  - Around 04 accredited multinationals are operating to provide inspection services BVI, SGS, LLoyed etc.
  - FIQC is mandated for fish inspection
  - Local govt. /sanitary inspectors are also operating throughout the country for food control under Pure Food Ordinance 1959.
  - Plant Protection Wing, Department of Agriculture for quarantine

ROAD MAP TO DEVELOP THE NATIONAL FOD QUALITY ASSURANCE SYSTEM

ROAD MAP TO DEVELOP NATIONAL FOOD QUALITY ASSURANCE SYSTEM

- **Establishing a Bangladesh National Food Administration BNFA to be the central national regulatory authority.** The Food Safety Policy incorporate a central regulatory body instead of existing non-functional National Food Safety Advisory Council which is neither having any enforcing arm nor even secretariat. Legal framework for national food quality assurance should be revised to separate regulatory and testing conformity assessment roles to avoid the potential conflict of interest existing now.

ROAD MAP TO DEVELOP NATIONAL FOOD QUALITY ASSURANCE SYSTEM

- Developing, disseminating, implementing Guidance Documents for Food Industries to meet the technical, regulatory and quality requirements in line with the international and national food safety, quality, conformity standards and best practices.
- Establishing Food Packaging Research, Testing & Training Institute within BCSIR under Ministry of Science and Information & Communication Technology
- Establishing Food Quality Testing Laboratories Accredited as per ISO/IEC 17025 in Dhaka, Chittagong & Jessore (three main Ports of Entry POE) for exported and imported foods

ROAD MAP TO DEVELOP NATIONAL FOOD QUALITY ASSURANCE SYSTEM

- **Introducing Traceability System through developing organizational framework from the food products producers' level to consumers/exporter's level within the food products value chain.**
- **Establishing a Centre for Risk Analysis within BCSIR under Ministry of Science with a view to support the effective activities in food standards setting, quality and safety management system**
- **Strengthening the National Conformity Assessment Infrastructure: Standards, Metrology, Testing, Certification & Accreditation in line the *international best practices and with WTO TBT & SPS Agreements***
- **Creating Awareness on Food Quality, Safety, Standards, HACCP, GHP, GMP, GLP, FSMS**

For Further Communications

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National Project Coordinator, QMS Component,
Bangladesh Quality Support Programme (BQSP)
United Nations Industrial Development Organization UNIDO

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Email: mostafa_anwar@yahoo.com

Thank You
Any Question?

UNIDO International Training Program on Food Laboratory
Management and Practice

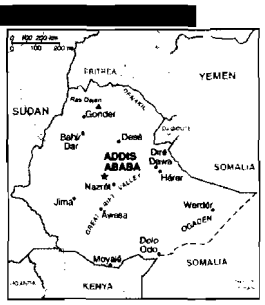
Gebze – Kocaeli, TURKEY
16-20 November 2009

BY Samson Girma
Head, Testing officer and Researcher
FOOD AND DRINKING WATER MICROBIOLOGY LABORATORY
Ethiopian Health and Nutrition Research Institute

Ethiopia

Geography of Ethiopia

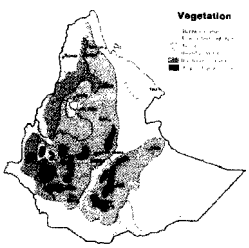
- Ethiopia is the oldest independent country in Africa
- Ethiopia is 1,127,127 square kilometers (435,071 sq. mi) in size.
- Ranked 26th in the world.
  - Land: 1,119,683 km²
  - Water: 7,444 km²
- Area - comparative
  - Slightly less than twice the size of Texas



Geography Continue

nd use

- Arable land: 12%
- Permanent crops: 1%
- Permanent pastures: 40%
- Forests and woodland: 25%
- Other: 22% (1993 est.)



Geography Continue (Highlands)

Population of Ethiopia

- Population = 77 million (ranked 15th, US = 300 million)
- 80 different ethnic groups in Ethiopia
- Oromo are the largest ethnic group in Ethiopia at 32.1%.
- The Amhara represent 30.2%.
- Tigray people are 6.2% of the population.
- Other ethnic groups are as follows: Somali 6.0%, Gurage 4.3%, Sidama 3.4%, Welayta 2%, Afar 2%, Hadiya 2%, Gamo 1%

Languages of Ethiopia

- The official language of Ethiopia is Amharic, a Semitic language which is spoken by about 27 million people.
- The second largest language in Ethiopia is the Oromo language, a Cushitic language spoken by about 30% of the population.
- English is the most widely spoken foreign language and is taught in all secondary schools.
- Amharic was the language of primary school instruction, but has been replaced in many areas by local languages such as Oromifa and Tigrinya.

Culture in Ethiopia

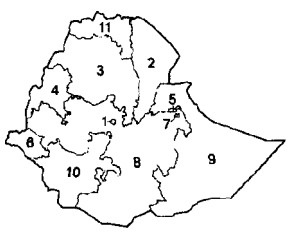
- Proud of their heritage, respectful to elders.
- Traditional Ethiopian cuisine employs no pork of any kind, as both Muslims and Ethiopian Orthodox Christians are prohibited from eating pork.
- Ethiopia has some of the finest athletes of the world, middle-distance and long-distance runners.

Government

- Before 1996, Ethiopia was divided into 13 provinces.
- Ethiopia now has a tiered government system consisting of a federal government, ethnically-based regional states, zones, districts (woredas), and neighborhoods (*kebele*).
- Ethiopia is divided into 9 ethnically-based administrative regions and 2 chartered cities: Addis Ababa and Dire Dawa
- Subdivided into 68 zones
- It is further subdivided into 550 districts
- Article 39 of the Ethiopian Constitution further gives every regional state the right to secede from Ethiopia.

Government Continue

- The subdivisions of Ethiopia are:
  - Addis Ababa (chartered city)
  - Afar
  - Amhara
  - Benishangul-Gumaz
  - Dire Dawa (chartered city)
  - Gambela
  - Harari
  - Oromia
  - Somali
  - Southern Nations, Nationalities, and Peoples Region
  - Tigray



Economy

- GDP = $55 billion, 73rd.
- Ranked 173 out of 177 countries in income per capita.
- $100 USD per year
- Highly reliant on agriculture (coffee being main: 65%-75%)
- Ethiopia's agriculture is plagued by periodic drought, soil degradation caused by overgrazing, deforestation, high population density

Major Exports

- Coffee
- Oilseeds
- Cereals
- Cut flowers
- Live animals
- chat

Up to very recent the food control system was conducted by the following bodies

- Ministry of Health, Ministry of Agriculture and Rural Development, Quality and Standard Authority of Ethiopia are responsible for food safety and standard setting
- Ministry of Health is responsible for food safety and Ministry of Agriculture and Rural Development is responsible for animal and plant health.
- Quality and Standard Authority of Ethiopia serves as a secretariat for standard setting can either adapt, or adopt Codex standard or other international standards.

- With the *current restructuring* the Food control at the federal level is controlled by the Ethiopian Food and Drug Control Agency, which is under the process of endorsement by the house of representative.

- the Ethiopian health and nutrition research institute was mandated by the federal ministry of health to strengthen the national laboratory system
- EHNRI was established by an act of parliament (proclamation no 26/1996)of the federal democratic republic of Ethiopia

- EHNRI, is the technical arm of the Ministry of Health and the responsible body for building the capacity of the national laboratory system,EHNRI

here are public health laboratories at the federal level nd regional states

The two largest federal and referal level Laboratories are:

The standard laboratory conducts microbiological nd chemical tests as per Ethiopian Standard.

The microbiology lab. Is accredited

- **The Ethiopian Health and Nutrition Research Laboratories which are also involved in public health services**
- **The microbiology lab is under the process of accreditation**

- The public health laboratory system is currently in development
- A master plan is developed for the public health laboratory system

.....THANK YOU.....

UNIDO International Training Program

Food Laboratory Management and Practice

NATIONAL FOOD SAFETY SITUATION IN KENYA

AGEMBO CLARKSON
KENYA BUREAU OF STANDARDS

1

INTODUCTION

Area 582,646 sq. Kilometres
Population approx. 40 million
GDP growth rates 4.3 (2004)
Agriculture accounts for about 24% of Kenya's GDP
75% of population depends on agriculture
66% of manufacturing sector is agro based
It has one of the most developed horticultural sector in Africa

2

Agricultural Production - 2003

Category	Commodity	Amount produced
Horticulture	Fruits and Vegetables	3.2 Million MT
Cereals	Maize, Rice, Sorghum, wheat, Millet,	3.0 Million MT
Milk	Fresh milk	3.1 Billion litres
Meat	Red meat, Pork, Poultry	0.4 Million MT
Sea food	Fresh water fish	139,811 MT
	Marine fish and other marine products	8,000 MT

3

Consumption agricultural produce

- Cereals – Kenya imports key grains (Maize, wheat and Rice) to supplement local production
- Fish – Domestic market consumes 70%
- Milk – 70% consumed at market level, 30% at family level
- Fruits and vegetables – 4% exported the rest consumed in local market
- Meat and meat products - >90% consumed locally Nairobi and Mombasa cities consuming the largest

4

Food processing

- Food and beverage industry comprises more than 2000 businesses
- Agro processing is the largest manufacturing sub-sector accounting for 13% of total manufacturing output
- Fish - >20 processing companies mainly for export
- Milk and Dairy processing is estimated at 2.5 million litres per day
- Cereal grains – there are over 20 major grain processors

5

Exports

- Grains – No exports were recorded in 2003 and 2004
- Sea food – 30% is exported. Nile perch accounts for 84% of total fish export, Tuna 13%.
- Dairy products – over 200 MT of milk and cream, 44 MT of butter and ghee exported in 2003
- Fresh fruits and vegetables – over 70,000 MT exported in 2003

6

National food safety system

- Food safety is managed by various government agencies under different ministries and laws
- The aim is to promote public health, consumer protection against health hazards and to enhance economic growth

7

Government agencies

- Department of public health
- Pharmacy and poisons board
- Department of Veterinary services
- Department of fisheries
- Pest control and products board
- Kenya Plant Health Inspectorate services
- Kenya Agricultural Research Institute
- Horticultural Crops Development Authority
- Kenya Bureau of standards
- National Council for Science and Technology

8

Impact on the Economy

Disease outbreaks in Kenya have affected;

- Productivity
- Income generation
- Expenditure on health

Failure to meet EU (Importing Countries) safety requirements has attracted threats of banning the horticultural produce (2002) and a ban on fish export (1998 -1999)

9

Impact on Health

Food – borne diseases are a problem in Kenya. In 2004 the following incidences were reported;

- Gastrenteriritis – 722,275
- Typhoid – 643,151
- Dysentry – 600,660
- Afflatoxin poisoning – 323
- Brucellosis – 198
- Cholera - 56

10

Social Impact

- Family members/caregivers spend more time looking after the sick
- Income/savings is depleted leading to increased poverty

11

Overview of Food safety system

Human resource capacity

- Capacity is inadequate in terms of knowledge in FSMS e.g. HACCP and Risk analysis among food inspectors and food safety managers in small and medium scale businesses.

12

Overview of Food safety system

Standards and Technical Regulations

- Food standards and regulations are available but their implementation and enforcement is not coordinated

13

Overview of Food safety system

Food –borne disease surveillance

- Is carried out by ministry of Health, however, statistics are not well analysed and documented

14

Overview of Food safety system

Laboratory support services for food hazards

The existing laboratory support services in regulatory agencies include;

- Radiation
- Pathogens
- Mycotoxins
- Heavy metals
- Pesticides and veterinary residues

15

KEBS' Testing Laboratories

- The Kenya Bureau of Standards Laboratories commenced operations in 1981 and have since expanded to 14 laboratories comprising of 10 Laboratories in Nairobi, 2 laboratories in Mombasa and 2 Laboratories in Kisumu.
- The Laboratories are managed in accordance to international standard ISO/IEC 17025 - *General requirements for competence of testing and calibration Laboratories.*

16

KEBS' Testing Laboratories

KEBS Laboratory customers include:

- Manufacturers
- Exporters/importers
- NGOs
- Government departments
- Researchers and Research institutions
- Individuals
- Internal customers (QAD,I/E,SDD)

17

KEBS Testing Organization

Testing Services has the following laboratories:-

Biochemical Labs

- Organic Chemistry - including chromatography and petroleum testing laboratories
- Inorganic Chemistry
- Food and Agriculture – including mycotoxin testing
- Microbiology

Materials Labs

- Electrical
- Mechanical- including NDT
- Civil
- Textile
- Polymers

18

Categories of sample

- Quality control samples
- Import Inspection samples
- Complaint samples
- Private samples
- Standard development samples

19

Food And Agriculture Laboratories

- Cereal and cereal products
- Animal feeds
- Dairy products
- Meat and meat products
- Beverages
- Fats and oils
- Fruits, vegetables and their products
- Sugar confectionery
- Alcoholic drinks
- Carbonated drinks
- Fertilizers

20

Microbiology Laboratory

- Foods
- Feeds
- Drinking water
- Cosmetics
- Disinfectants
- Textiles

21

Laboratory Accreditation

In the year 2000, KEBS applied for laboratory accreditation to ISO/IEC17025 by the United Kingdom accreditation (UKAS) for various tests in the chemistry and microbiology areas. Formal accreditation was granted in 2001.

22

Proficiency Testing

The laboratories participate in proficiency testing for various products. The proficiency testing is provided by;

- FAPAS
- LGC
- La farge
- EAC/SADC-MET

23

Proficiency Testing

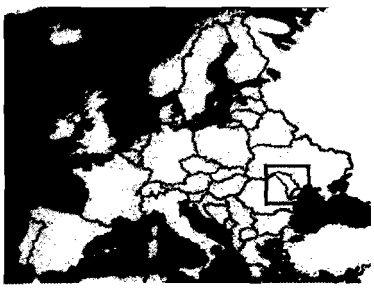
The laboratories have also initiated and participated in a regional EAC PT scheme. This is through the East African Community SQMT subcommittee on testing where the KEBS food laboratory is a provider solid matrix sample (wheat flour).

24

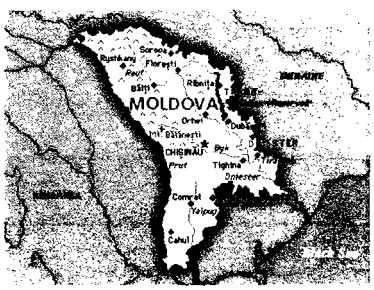
Vision for the Future

The acquisition of the GC-MS and LC-MS equipment in conjunction with other existing equipment will enable KEBS laboratories to carry out analysis for gases, performance enhancing drugs, micronutrients, and characterization of materials.

25



REPUBLIC OF MOLDOVA



Republic of Moldova is a landlocked country in Eastern Europe, located between Romania to the west and Ukraine to the north, east and south.

Capital City: Chisinau
Other Cities: Tiraspol, Balti, Tighina, Rîbnita
Area: 33,843 sq. Km
Population: 3.4 million, excluding the estimated Transnistrian population of 580 000.

Moldova's proximity to the Black Sea gives it a mild and sunny climate. The fertile soil supports wheat, corn, barley, tobacco, sugar beet, and soybeans. Beef and dairy cattle are raised, and beekeeping is widespread. Moldova's best-known product comes from its extensive and well-developed vineyards concentrated in the central and southern regions. In addition to world-class wine, Moldova produces liqueurs and sparkling wine. It is also known for its sunflower seeds, walnuts, apples, and other fruits.

The agriculture is the most important economic sector in the Republic of Moldova (RM).Primary agriculture contributes to about 20 % of GDP. When agro-processing is included, agriculture accounts for more than one third of the national economy. Agriculture is also the largest economic sector in terms of employment (40% of population). Moreover, food and agricultural products have accounted for about two third of RM's total export in the past five years.

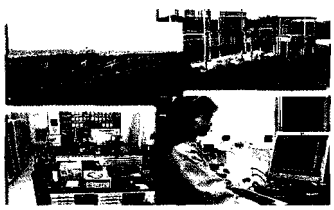
Trading Partners

Exports $1.43 billion f.o.b. (2007 est.)

Main partners Russia 35.8%, Italy 13.9%, Romania 10%, Germany 7.3%, Ukraine 6.6%, Belarus 6%, U.S. 4.6% (2008)

Imports $1.83 billion f.o.b. (2007 est.)

Main Partners Ukraine 24.6%, Russia 12.2%, Romania 9.3%, Germany 8.5%, Italy 7.4% (2008)



Effective national food control system is essential to protect human health and consumer's interest. It is also critical to enabling country to assure the safety and quality of food entering into international trade and to ensure that imported foods conform to national requirements. The new global environment for food trade places considerable obligations on both importing and exporting countries to strengthen their food control system and to implement and enforce risk based strategies.

The food safety system in the RM is under change now in such a way as to comply with EU's aqlus comunautaire in food safety area.

Food Control Management

1. **Ministry of Agriculture and Processed Industry**
   Responsible for the food safety inspection and control activities for food of animal origin
2. **Ministry of Health**
   Responsible for the food safety inspection and control activities for food of non-animal origin
3. **Ministry of Economy and Trade**
   Responsible for consumer's right protection

Laboratory service

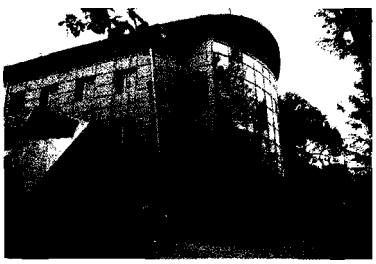
Laboratory control is carried out in authorized and accredited institution:

-**Republican Center for Veterinary Diagnostic** (implemented ISO 17025 for chemical, microbiological, parasitological and radionuclide analyses in food of animal origin)

-3 regional veterinary laboratories

-**The National Center for Scientific and Applied Preventive Medicine** (implemented ISO 17025 for chemical, microbiological, parasitological and radionuclide analyses in food of non-animal origin and potable water)

-36 Centers for Preventive Medicine



Republican Center for Veterinary Diagnostic

Was founded in August 1945
RCfVD consists of two departments
- Animal Health
- Food Safety Control

Animal Health

Department of Parasitology

Department of Virology

Department of Radiology

Department of Bacteriology

Department of Pathology

Department of quality control of veterinary medicines

Food Safety Control

Department of Chemistry

Department of Microbiology

Food Safety Control
Laboratory tests performed

Physico-chemicals analyses:

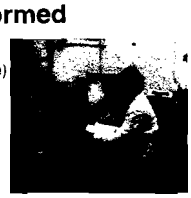
- moisture, chlorides, protein, fat, nitrates;
- phosphate, solubility, starch;
- dry matter, free fatty acids;
- inhibitory substances, acidity, purity;
- density, pasteurization

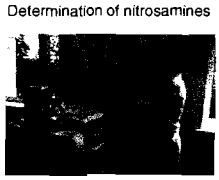
Food Safety Control
Laboratory tests performed

Chemical:

Determination of heavy metals (Pb,Cd,Hg,As,Cu,Zn, Fe)

- Determination of residues of antibiotics, nitrofuranes, sulphonamides
- Determination of pesticides
- Determination of aflatoxins
- Determination of hormonal drugs
- Determination of nitrosamines

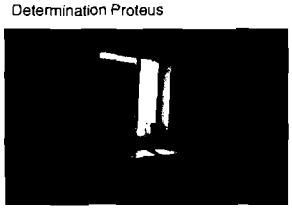




Food Safety Control
Laboratory tests performed

Microbiological:

Determination of total Number of microbes
Determination of coliforms
Determination of Salmonella
Determination of Sulfite-reducing clostridia
Determination Staphilococcus aureus
Determination B.cereus
Determination Proteus



The objective of the Food Safety Strategy implementation

-Harmonization of the national food legislation with EU legislation;

-Harmonization of laboratory testing methods;

-Implementation of an efficient system for monitoring programs for contaminants and residues , food additives;

-Introducing of rapid alert system;

-Implementation of GHP and HACCP in food industry enterprises;

-Improving of efficiency of food inspection system;

THE END

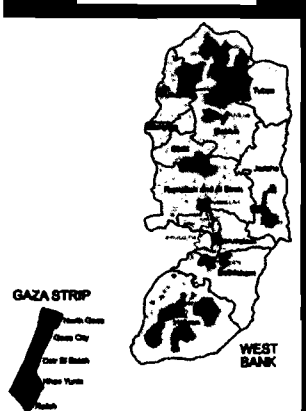
THANK YOU!

A-Najah National University

Food control, Food laboratories, Quality control systems in Palestine

Mohammad Ass'd Mohammad (Abdel Qader) Mousa
Lab. Superintendet CBDA Center
An-Najah National University –Nablus- Palestine

Palestine Authority



Capital of Palestine :Jerusalem
Population:4,148,000
Language: Arabic
Established May,4 1994
Currency :Jordan Dinar,
Israeli new Sheqel
Time zone UTC+2
Agriculture :Olive Tree
more then 12 million olive tree

Introduction

The Palestinian food industry has been one of the important and most rabidly developing sectors in Palestine.

Food industry development and growth have been obvious in the last decade with total market sales of approximately $350 million per year.

Introduction

The food processing industry played main role in the Palestinian economy; it contributed with more than Twenty Three percent of the production value, Thirteen percent of the added value total .
The number of organization working in the food and drinks processing industries in west bank and Gaza reached more than one thousand five hundred organization which divide into Ten Sectors

The Food industries Sectors in Palestine

The Food industries Sectors in Palestine :
Meat Processing and preserving, ***Fruits and Vegetables*** Processing and Preserving , Vegetarian and Animal ***Fats and Oils***, ***Diary Production*** , ***Serials*** Production, ***Animal Feed*** Production, Production of ***Bakeries Products*** , Production of ***Chocolate and Sugar Sweets Products*** , Production of ***Noodle*** , Production of Other Food Products

Measurements and Specification Law in Palestine

This law established a new body, the Palestinian specifications and measurements organization, (Palestine Standard institute) ,its goal is to keep the health ,security, and safety of the citizen ,save the industry stability, support the commercial exchange through improving the quality of the local products to export it to other countries

Quality System in Palestine

The main quality systems in Palestine at the processing are the ISO, and HACCP, and the Palestinian quality certificate those quality systems are very important and it contributes in helping the Palestine Food companies in achieving its competitive advantage. Moreover, it is used as an effective tool of marketing at the external markets. At the current time, only a little percent that doesn't exceed 10 % applies these System in the Palestinian Food industries.

The Palestine Standard Institute supervision or Palestinian quality certificate

This certificate is important for the marketing at the local market as it could a quality and standard indicators, it could also be good for the marketing at the Arab countries markets because there is a recognition of each standard ,but it is not appropriate for marketing at the Israeli or foreign markets.

The Palestine Standard Institute

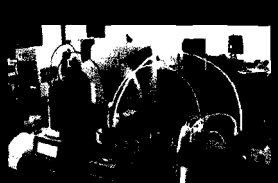
The Palestine Standard Institute is only entity in Palestine, which sets food standards , and give Certified that the Food Laborites in the public and private sector is works according the requirements of ISO/IEC 17025

Type of Food Laborites in Palestine

In Palestine there are many Food Laborites Divide between the public Sector(for example the Central Public Health Laboratory(Ministry of Health)) and Private Sector (for example The Chemical , Biological and Drug Analysis center(CBDA) (in An-Najah National University)) ,(The Scientific Center for Food, Drug and Toxins Analysis (Private Labor))

A-Najah National University

Chemical, Biological & Drug Analysis Center (CBDA)



CBDA Center was established 1999 in Nablus City in West bank to assist in the field of food analysis and shelf life.

CBDA Center provides its services to the private and public sectors.

The center is accredited by the Palestinian Standard Institution With a certificate No. 16. and working according to the requirements of ISO/IEC 17025

Staff of CBDA Center

The policy of the center is to employ highly professionals in the field of food, pharmaceuticals, cosmetics and detergents analysis.

Chemical ,Biological & Drug Analysis Center Units

Food and Animal Feed Analysis Unit	Drugs of Abused Analysis unit	Drugs and Cosmetics Analysis unit	Detergents Analysis Unit	Polymer Analysis Unit (New)	Air pollution Analysis Unit (New)	Water Analysis Unit	Training and Research Unit

Important instruments **available at the CBDA center**

GC/MS,FTIR,UV/Vis Spectrophotometer ,Elemental Analyzer, Vapor Pressure Osmometer,Karl Fisher ,Refractometer, Polarimeter,Mosture balance, Flame photometer,Atomic Absorptin Spectrophotometer,Kjeldahl Analyzer,Viscometer,Aflatoxine flourometer,Luminescence Spectrophotometer ,pH meters,Conductometers,Incubators, Laminar flow,Autoclave,Pharmaceuticals analysis instruments (Pharama test),Bomb Calorimeter.

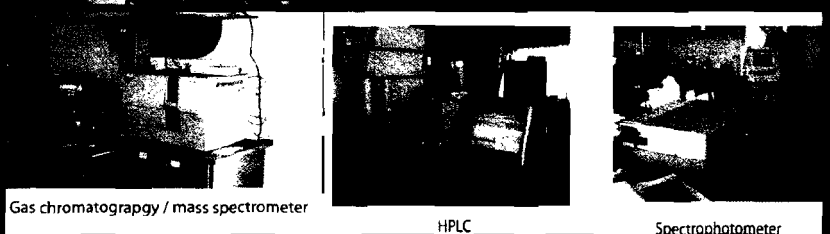
Analysis available at the CBDA center
Chemical and Physical

The Food Chemical and Physical analysis in the CBDA Center are more than Sixty Analysis for all Type of Food .
For example Protein, Aflatoxine , Vitamins, Pesticide , Fat , Total Carbohydrate (Fiber, Sugar, Invertid Sugar, Starch).....etc

Analysis available at the CBDA center
Microbiological Analysis

Sterility test, Bacillus, Clostridium, Escherichia coli, Steptococcus Fecalis , Listeria,Total Coliform Count, , Salmonella and Shigella, Parasites and Worms, Total fecal coiform, Aerobic Plate Count, Anaerobic plate count, Total Yeast & Molds, Staphylococcus aurous

Image of same Modern Instilment
In the CBDA Center



Gas chromatograpgy / mass spectrometer

HPLC

Spectrophotometer

For more information please visit CBDA Center web. Page

email:

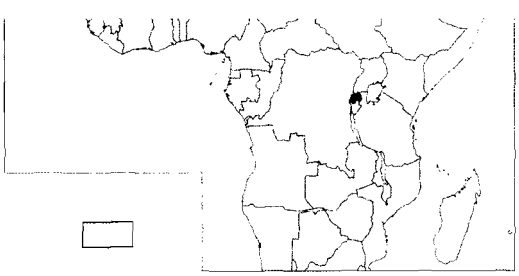
zawatehm@yahoo.com

Thank You

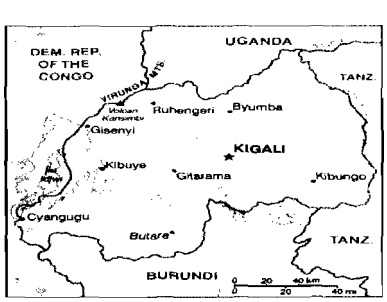
The role of Quality Infrastructure in National economy-Rwanda's case

Philip Nzaire- TUBITAK-MRC (Turkey)

Position of Rwanda on the African Continent



Map of Rwanda



National structures

- National flag 
- Court of Arms 
- Motto: "Unity, Work, Patriotism"

Local government provinces

- Rwanda is divided into five provinces (*intara*) and subdivided into thirty districts (*akarere*). The provinces are:
- Northern Province
- Eastern Province
- Southern Province
- Western Province
- Kigali Province
- Prior to 1 January 2006, Rwanda was composed of twelve provinces (known as prefectures up to 2001), but these were abolished in full and redrawn as part of a program of decentralization and reorganization.

General information

Capital	Kigali
Official languages	Kinyarwanda, French, English
Government	Republic
Independence	From Belgium
Date	July 1, 1962

Total area	26,338 km² **10,169 sq mi**
Population-2009 estimate	9,998,000
2002 census	8,128,553
Density	379.6/km² 983.2/sq mi
GDP (PPP)	2008 estimate
Total	$10.004 billion
Per capita	$1,043
Currency	Rwandan Franc
Rate (Rfw:USD)	573.01(selling) 566.17(buying)

Major pillars of Rwanda

- vision 2020 umurenge, EDPRS,
- good governance,
- social justice,
- economic development,
- social welfare,

Economic growth in different Rwandan sectors:

- **Agriculture:**
- The Rwandan economy thrives on the agricultural outputs of the country, employing almost 91% of the national population. The 1.1 million hectares (2.8 million acres) of arable land area in Rwanda cultivate food crops for domestic consumption and cash crops for commercial purposes.

Rwanda information

- In fact, coffee and tea as main cash crops, account for 80% of all export revenues.
- Though crop production was severely hampered following famine, the country soon recovered itself from its catastrophes and increased the annual agricultural production substantially.
- Today, this sector contributes 41.6% to the Rwandan GDP.

Agriculture and economy

- Agriculture accounts for approximately 42% of GDP and employs about 90% of the population.
- Beans, sorghum, potatoes and cassava are the main food staples.
- Coffee, tea, bananas and potatoes are the main domestic cash crops

Industry sectors

- Total number of industries visited by RBS=140
- Food industries= 78.5%
- Non-food industries = 21.5%
- Industry products at a level of certification = 22%
- Products certified = 2
- Products applied to be certified = 7

Industry:

- The manufacturing industries of Rwanda accounted for 11.3% of the Rwandan GDP in 1998. There was a 75% fall in the Rwandan industrial production in 1994, following political and social turmoils in the country.
- Soon, the industrial activities of Rwanda recovered and in 2000, the GDP contribution from this sector was 20%. In 2001, the growth rate of industrial output was 7%.
- Average GDP contribution from Rwanda's industrial sector is 21.9%

The major industries in Rwanda are:

- Production of wine, metal goods, soft drinks, sugar, beer, tea, flour, cigars and coffee
- Textile industry
- Soap industry
- Automobile repairing
- Cement manufacturing units
- Plants producing medicines and life-saving drugs
- Exploitation of natural gas
- Processing of foodstuffs

Industries cont'd..

- Manufacturing of artisan goods such as pottery, wicker baskets,
- Bricks
- Footwear
- Floor Tiles
- Roofing material
- Pesticides
- Production of matches
- Pyrethrum refinery
- Paint factories

Export/Import figures

Merchandise trade (% of GDP)	14.5	19.1	20.8
Exports of goods and services (% of GDP)	8.3	8.3	9.6
Imports of goods and services (% of GDP)	24.6	27.5	27.4

Foreign trade:

- The Rwandan export market is dominated by coffee and tea, accounting for 80% of all export incomes. In fact, during 1996 and 1999, the export activities escalated by 424%. The principal export goods are:
- Coffee - 56%
- Tea - 27%
- Gold - 17%
- Animal skins and hides - 0.9%
- Kenya is the main export partner of Rwanda.

From 1996 to 1999, the Rwandan imports increased by a small margin of 25%. In 1999, the country imported commodities like:

- Food products
- Cement
- Machinery and other equipments
- Steel
- Petroleum products
- Building and construction materials

The main suppliers of Rwandan import commodities are:

- Belgium
- Kenya
- Japan
- China
- UAE,
- Uganda

Tourism

- Tourism is one of the fastest growing sectors and is now the country's leading foreign exchange earner; generating US$214 million in 2008, up by 54% on the previous year. Despite the genocide, the country is increasingly perceived internationally as a safe destination, and one million people are estimated to have visited the country in 2008, up from 826,374 in 2007.

Tourism cont'd

The country's most popular tourist activity is the tracking of:

- mountain gorillas, in the Volcanoes National Park.
- Nyungwe Forest, home to chimpanzees,
- Ruwenzori colobus and other primates,
- The resorts of Lake Kivu, and Akagera, a small savanna reserve in the east of the country.
- Each year in June, the country celebrates Kwita Izina - The Baby Mountain Gorilla Naming Ceremony.
- People come from all over the country and the world to participate in this unique event.

-

- Europe has traditionally been the main destination for Rwandan exports, reflecting the dominance of traditional commodity exports, coffee and tea.
- Coffee growers represent about 400,000 people.
- Tea production and processing is primarily managed by state-owned factories employing about 53,000 workers.

-

- Rwanda's Diagnostic Trade Integrated System (DTIS), validated in 2005, acknowledges that – in the short-term – reinforcement of these sectors through *inter alia* increasing productivity and raising quality is key to poverty reduction.
- In addition, standards for product safety and quality and opportunities for increasing horticultural exports from Rwanda are sections that figure prominently in the DTIS

Food safety system in Rwanda

- No legally established single Body incharge of food safety
- There exists many organs in the food safety like:
  Ministry of Agriculture for policy on agriculture production and dissemination of animal breeds and seeds to the population through institutions like RARDA, RADA, RODA,
  Ministry of Health on public utilities sanitation and general policy on hygiene – carying out inspections in hotels for hygiene
  Local council authorities

Food safety cont'd

Rwanda Bureau of Standards

- Rwanda Bureau of Standards (RBS) is a public institution established by the law N° 43/2006 of 05/10/2006 determining the responsibilities, organization and functioning of the Rwanda Bureau of Standards (RBS) to undertake all activities pertaining to the development of Standards, Quality assurance, Testing and Metrology in the country.

RBS cont'd

Vision

The vision of RBS is to be a trusted party in providing internationally recognized and customer suited standardization services.

Mission

To provide standards based solutions for Consumer Protection and Trade promotion for socio-economic growth in a safe and stable environment.

Food safety cont'

- RBS is the only body with powers to define and document national standards.
- Public services and public or private firms must present their standards to RBS for adoption at national level. The Bureau is governed by the Board of Directors composed of major stakeholders from government, industry and academic institutions, as well as consumer associations.

What is quality infrastructure?

-**Quality infrastructure** relates to all fields of Metrology, Standards and Testing, of Quality Management and Conformity Assessment, including Certification and Accreditation.

-Applications of Quality Infrastructure are seen in everyday use from domestic appliances to sophisticate areas like construction materials.

Quality infrastructure is a Prerequisite

- Quality infrastructure must ensure:
  elaboration and implementation of technical regulations for products and passage from compulsory standards to voluntary ones;
  application of code of practice, Good Agricultural Practices, Good Manufacturing Practices regarding standardization;
  elimination of all measures that pose [illegible] barriers – not only customs taxes and quantitative restrictions, but all the measures with equivalent (protectionist) effects

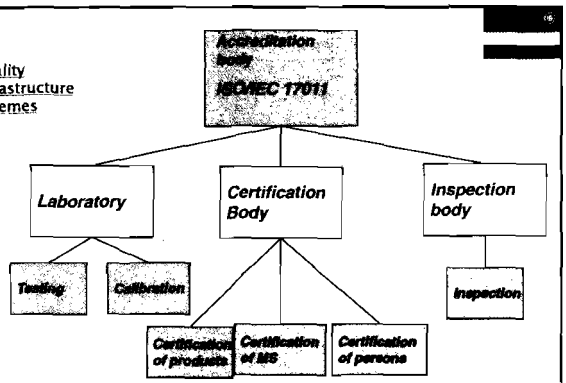
Quality infrastructure cont'd

- In today's business environment, a functioning Quality Infrastructure is a prerequisite for access to regional and global markets and a key determinant of competitive advantage. Globalization of world trade and the requirements of the Agreements on Technical Barriers to Trade (TBT) and Sanitary and Phytosanitary Measures (SPS) of the World Trade Organization (WTO) for the reduction of technical barriers to trade urgently call for measures to consolidate quality infrastructure

Quality infrastructure cont'd

A proper quality infrastructure will not only protect society and nature from damaging services and products, but will also improve competitiveness and complement the knowledge and skills given to the country's workforce.

Quality Infrastructure Schemes



Facilities at RBS

- Standards development
- Imports inspection at border posts
- Industrial inspections
- Products and management certification services
- Market surveillance
- Biochemical testing
- Calibration services: **mass, temp, pressure, volume, dimension, electricity (force,),**
- Verification of petrol pumps, weighing scales
- Agriculture and livestock products certification
- TBT enquiry point

Impacts due to quality infrastructure reforms

- Rwandan processing companies and service providers will receive ISO 9001 Certification locally through the accredited bodies of Rwanda- saving significant amounts of time and money that previously had to be spent on foreign certification bodies.
- Rwandan factories throughout the country can now access mobile calibration services and therefore do not need to stop production to send measuring equipment for calibration.

Impacts cont'd

- Customers in markets are now assured that they are getting proper measurements after trained inspectors have been introduced.
- Industries following the newly adapted/adopted Rwanda standards can now be sure that they are meeting the requirements of potential export markets.

Laboratory accreditation

- Accreditation Procedure by which an authoritative body gives formal recognition that a body (laboratory) or person (signatory) is competent to carry out specific tasks (scope)

Challenges to accreditation

- Pressure from the market
  - customers and stakeholders
- Costs of accreditation
  - high
- Maintenance of the accreditation system
  - personnel changes
  - procurement process

Current Testing Laboratories: Position

- Completed documentations required (Three tiers)
- Currently carying out internal audits
- After internal audit, will do corrective actions in three to four weeks (where appropriate)
- Will invite external Pre-audit Assessors from EAC by Feb 2010 (funding by UNIDO)
- Will apply for accreditation by April/May 2010
- Have catered for increasing the scope of testing to cover Mycotoxons in foods, Heavy metals, Pesticide residues and vitamins in foods.

Current scope of testing

- Chemical parameters:
  Heavy metals in water (copper, iron, lead), potassium, sodium, chloride, sulfate, nitrate, nitrite, pH, methanol, ethanol, fat, protein, fibre content, total ash, acid insoluble ash, volatile acids, brix, viscosity, moisture content, solid non-fat, freezing point depression, total solid, total alkali, peroxide value, acid value, iodine value, soap content, sodium chloride, sulphur dioxide, sugar polarization, insoluble impurities, conductivity, total dissolved solids.

Scope

- Microbiological parameters:
  Total viable count, total coliforms, faecal coliforms, E.coli, Salmonella, Shigella, enterobacteriaceae, Streptococcus faecalis, Staphylococcus aureus, Yeasts and moulds, sulfite reducing organisms and sulfite reducing spores.

Major projects

- Expanding the testing laboratories
- Hiring consultancy for training TLU staff on chromatography (pesticides and others)-funding by EU: tender was launched, no bidder qualified, to be re-launched
- Hiring consultancy for training TLU staff on spectroscopy-World Bank funding: Tender was launched, evaluation completed, contract to be signed

Projects cont'd

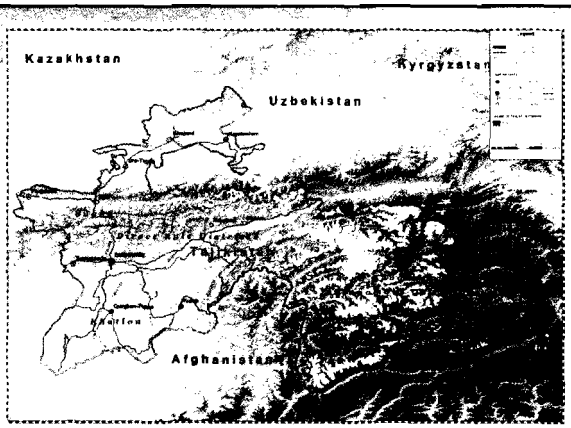
- Procurement of equipment for testing Heavy metals-World Bank funding: Equipment has arrived, awaiting commissioning by suppliers
- Procuring other necessary equipment-UNIDO funding: Pending UNIDO's decision
- Training staff: UNIDO and EU will fund some targeted trainings like Equipment operation and maintenance, Laboratory Assessors course, method validation, measurement uncertainties

Thank you

THE COUNTRY REPORT ABOUT THE NATIONAL FOOD LABORATORY MANAGEMENT IN TAJIKISTAN

Ahmadov I.
Soil Sciences Institute

Dushanbe - 2009



GENERAL INFORMATION

- **Tajikistan is located in a southeast of Central Asia;**
- **There are borders with the Republic of Uzbekistan and the Kyrgyz Republic in west and north of the country;**
- In east and in south with PR of China (430 km) and IR of Afghanistan (1030 km);
- Tajikistan is a typical mountainous country;
- The Pamirs-Alai mountain ecosystem – pick 7495 m a.s.l.;
- 60 % of the republic territory are located at heights 2500 m a.s.l;
- 93 % of Tajikistan territory occupied by mountains.

GENERAL INFORMATION

- The area of Tajikistan is 143.1 thousand sq. km;
- The population is 7.2 mln;
- 73% of the population lives in rural areas;
- More than 40% of the population are in the age below 14;
- Administrative divisions: Districts of Republican Government Subordination; Khatlon oblast, Sogd oblast; Gorno – Badakhshan Autonomous oblast and Dushanbe City;
- Tajikistan is agrarian country

Gross agriculture production (thousand t.)

	2001	2004	2007
Cereal	494,2	891,6	931,2
Potatoes	308,2	527,2	662,1
Vegetable	398,5	681,5	835,1
Melon plantation	96,9	150,4	254,2
Fruits	143,8	144,4	157,2
Grapes	109,7	93,2	116,9

Livestock production (thousand t.)

	2001	2004	2007
Meat	30,0	48,4	59,4
Milk	382,6	490,2	583,6
Egg (million)	41,9	77,7	111,2

oblast

Food security

- Tajikistan is in zone of a catastrophic food own production;
- In 2007, production of meat and meat products in relation to the recommended standards were lower then the 7,7 times;
- In 2007, imports of food products totaled $ 195,3 mln, while food exports amounted to $ 37,2 mln;

Food security

- The amount of food imports in the whole imports has exceeded the proportion of food exports in the whole exports of more than 6 times.
- Food imports in 2007 increased in comparison with 2006 at 58.8%, including the import of ready food products - at 40.9%, fats and oils of animal and plant origin. - at 49.2.

Food security

- The cost of food of tile consumer basket, in ac-tual consumption prices at the beginning of 2007 to a family member was 63.55 somoni, with the standards of nutrition at 132. 72 somani.

Food control, food laboratories, quality control systems

- Control to be implemented by the state on the base of existing legislative;
- The main laboratory is under the «Tajikstandart»;
- Also the control to be implemented by the number of laboratories of different institutions located over the country (for example – at markets, customs, etc.);
- There is define all components including harmful substances.

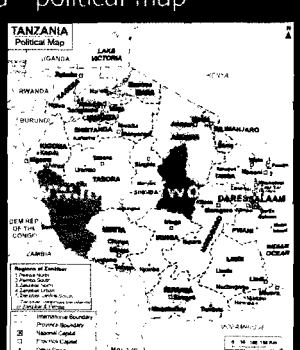
THANK YOU VERY MUCH FOR YOUR ATTENTION

TANZANIA

FOOD QUALITY CONTROL

Agnes Njau Mneney
Principal Quality Assurance Officer
Tanzania Bureau Of Standards

Tanzania - political map



INTRODUCTION

- The economy of Tanzania is on agriculture based (including animal production and fisheries) which accounts for more than 60% of the GDP. More than 80% of the population is rural based and depend entirely on agriculture for food and cash earnings

Intro cont.

- It is subsistence traditional small holder-based and rain fed activity.
- Tanzania exports are primarily agricultural commodities with Fisheries products, coffee, cashew nuts, tobacco and cotton constituting the largest sectors. Mining sector is the second in export.
- Major trading partners of Tanzania include:

i. EEC and Asia that account for total Tanzania export of 38.4% and 32.3% respectively.
ii. SADC member countries account for 11.7% and 10.9% of the country's imports and exports respectively.
iii. EAC member countries accounted for 5.5% of total exports from Tanzania while imports from these countries accounted for 6.9% by end 1998 (WTO Trade Policy, 2000).

Introd. Cont.

- The country is still under micro- and macro-economic adjustments in line with globalization and market liberalization forces in the world.
- Under the adjustments, food safety has been recognized as a prerequisite for national food security and for both regional and international trade in food.
- in view of this recognition food functions in the country is in the process of re-organization to enhance food safety and quality.

2. FOOD SAFETY AND QUALITY CONTROL FUNCTIONS

- The Ministries empowered by Laws the responsibilities for food quality and safety functions include:
- Ministry of Health,
- Ministry of Agriculture and Food Security,
- Ministry of Livestock development and fisheries, and
- Ministry of Industries and Trade, These laws include

Different Laws in Food quality and safety

- **Tanzania Food, Drugs and Cosmetics Act, 2003.** An Executive Agency under the Ministry of Health and regulatory body responsible for preparations of regulations for the control of quality and safety of food, drugs (including herbal drugs), cosmetics, and medical devices.
- **The Plant Protection Act** - regulate the importation and exportation of plant products to and from the country with the view of controlling diseases and pests. The Plant Protection Section in the Ministry of Agriculture and Food Security in collaboration with the TFDA do issue permits for the export/importation of food products of plant origin. Such food products are inspected and certified for safety and quality at the points of entry/exit.

Laws cont.

- **The Fisheries Act** - empowers regulation and assurance of safety and quality of all fishery products produced and processed in the country. The Fisheries division in the very ministry controls and monitors the safety and quality of fishery products exported from, or imported into the country.
- **Radiation Control Act** - Under this law, a National Radiation Control Commission has been established and is charged with the function of monitoring all food products produced and/or processed in, and food products exported from or imported into the country for the presence of radioactive material.

Laws cont.

- **Standards Act** - empowers for the promulgation of national standards including standards for food products. Standards are referenced to by the sector ministries when preparing requisite legislation as stipulated under the relevant laws.
- TBS does also carry out product third party certification based on available compulsory national standards. Similarly, imported products (including foods) are monitored by TBS under a Batch Third Party Certification Scheme
- **Tropical Pesticides Research Institute (TPRI) Act** – empowers the responsibilities for registration and approval of pesticides for use in the country in line with toxicity rating.

Available rules & regulations

- Storage, distribution manufacturing, preparation & sale of Food and foodstuffs
- Food additives, pesticides residues
- Sanitary at food processing and food service levels
- Food labelling
- Date marking
- Sampling procedures
- Importation and exportation of food materials

Rules & Regulatios

- In-process **quality control**;
- on an individual or shared basis, to ensure the safety and **quality** of their products
- Licensing & registration of **food** premises;
- Closure of unhygienic **food** premises;
- Health **control** of **food** handlers;
- Advertising of **food**
- Use of safe **food** packaging material;
- Ante-mortem and/or post-mortem examination of **food** animals.

Available regulation cont.

- Provision and adequacy of sanitation measures on board aircraft, trains,
- ship, and in-service terminals;
- Irradiation of **food**;
- **Quality** certification by an appropriate authority;
- Quarantine measures;
- Waranty measures;
- Penalities.

Testing Laboratories

- Available at regulatory bodies including:

i. TFDA
ii. TBS
iii. TPRI
iv. NFQC
v. GCLA

challenges

- The functions are fragmented into different ministries without coordination
- There is no available national Food Policy
- Outdated laws and regulations
- Inadequate enforcement mechanisms
- Inadequate capacity in products conformity assessment – human resources and testing facilities

Current status

- Accreditation of testing laboratories as per ISO/IEC 17025 is on progress
- Review of Food laws and regulations is on progress
- System registrations as per relevant standards is on progress

MWISHO

THANKS FOR LISTENING

UNISWORK VIII- Food Laboratory Management and Practice Program

- Nov. 16-20, 2009
- TUBITAK, Marmara Research Centre, Food Institute.

Kudret AVCI
ANKARA PCL

GENERAL VIEW OF FOOD INDUSTRY

Ratio to total production industry: average 20 %

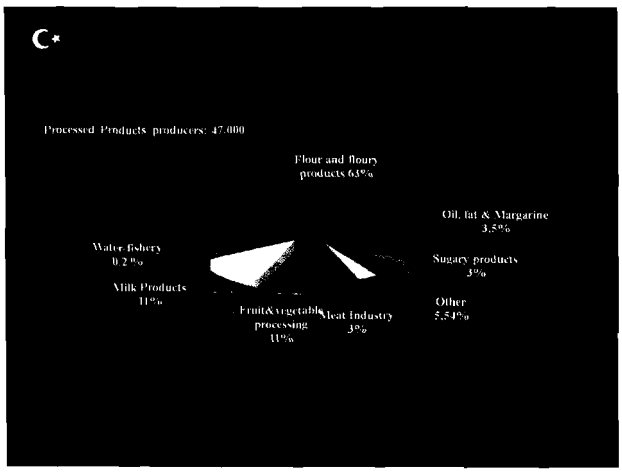
Employment: 471.000 people

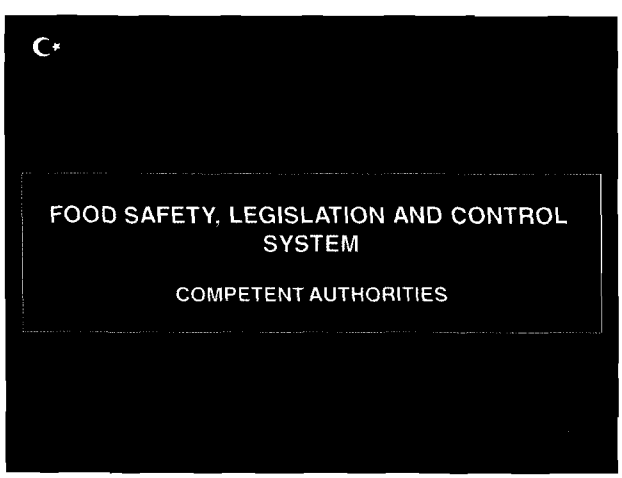
Managerial employment: 28.000 people

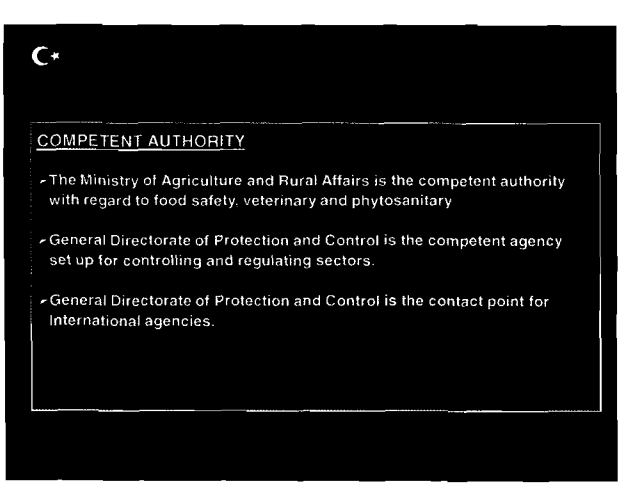
Number of food producer:476.000establishments

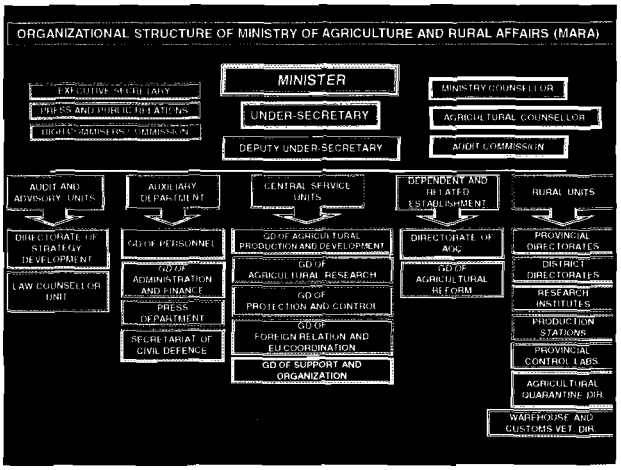
Large capacity producers: 2.000 establishments

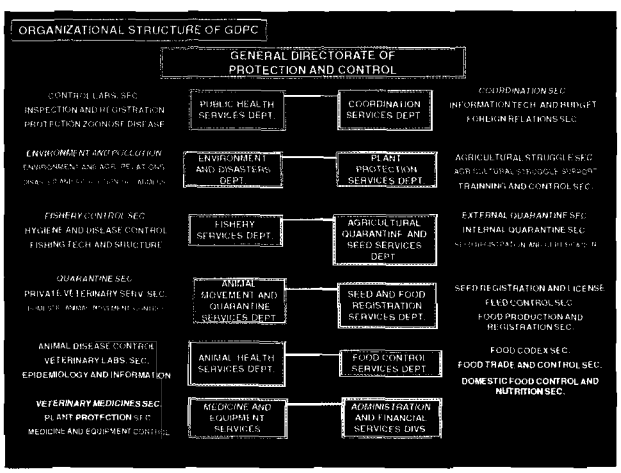
- Active Capacity : 68-74.6 %
- Total production: 418.000.000 tons
- Number of sales and consumption facilities: 400.000


Processed Products producers: 47.000
Flour and floury products 63%
Oil, fat & Margarine 3,5%
Sugary products 3%
Other 5,54%
Meat Industry 3%
Fruit&Vegetable processing 11%
Milk Products 11%
Water fishery 0.2 %


FOOD SAFETY, LEGISLATION AND CONTROL SYSTEM
COMPETENT AUTHORITIES


COMPETENT AUTHORITY
The Ministry of Agriculture and Rural Affairs is the competent authority with regard to food safety, veterinary and phytosanitary
General Directorate of Protection and Control is the competent agency set up for controlling and regulating sectors.
General Directorate of Protection and Control is the contact point for International agencies.





- Legal Framework (Primary Legislation):
  - *Decree Law no. 441 on Organization and Duties of* **MARA** (O.G. 09.07.1991, No. 20955)
  - The Food Law no.5179 (O.G. 05.06.2004, No.25483)
  - Animal Health and Inspection Law no. 3285 (O.G. 08.05.1986, No.19109)
  - *Animal Breeding Law no. 4631 (O.G. 28.02.2001, No. 24338)*
  - Feed Law no.1734 (O.G. 07.07.1973, No. 14557)
  - Fisheries Law no. 1380 (O.G. 22.03.1971, No.13799)
  - Law of 308 on Registration Control and Certification of Seed for Sowing (O.G. 29.08.1963, No.11493)
  - Law on Protection of Breeder's Rights for New Plant Varieties no. 5042 (O.G. 08.01.2004, No. 25347)
  - Agricultural Struggle and Plant Quarantine Law no. 6968 (O.G. 08.05.1986, No. 9615)

C*

OTHER MINISTRIES INVOLVED AND THEIR COMPETENCIES

- Ministry of Interior (via Municipalities and Province Management)

Issuing working permission and registration of certain food establishments acc. to the Law no. 5216, Law no. 5393 and Law no. 5197

- Ministry of Health (General Directorate of Primary Health Services)

The principles concerning water, special foods intended for medical purposes are specified by the Ministry of Health.

The Ministry of Health has the right of intervention in emergencies concerning public health acc. to Law no.5179

C*

OTHER MINISTRIES INVOLVED AND THEIR COMPETENCIES

- Organized Industrial Areas

Within the organized industrial areas, food establishments working licenses issued by these organizations acc. to Law no. 4562

- Ministry of Environment and Forestry

Animal protection Law no. 5199 is giving responsibility and competence to the Ministry of Environment and Forestry.

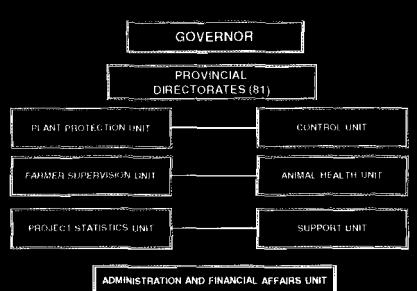
C*

- Legislation related to the Other Ministries:
  - The Food Law no.5179 (O.G. 05.06.2004, No.25483)
  - Metropolitan Municipality Law no. 5216 (O.G. 23.07.2004, No. 25531)
  - Municipality Law no. 5393 (O.G. 13.07.2005, No. 25874)
  - Provincial Management Law no. 5197 (O.G. 04.03.2005, No. 25745)
  - Organized Industrial Areas Law No. 4562 (O.G. 15.04.2000, No.24025)
  - Animal Protection Law no. 5199 (O.G. 01.06.2004, No. 25509)
  - Public Hygiene Law no.1593 (O.G. 06.05.1930, No. 1489)
  - Consumer Protection and Competition Law no. 4077 (O.G. 08.03.1995, No. 22221)
  - The Law Relating To The Preparation And Implementation Of The Technical Legislation On The Products no. 4703 (O.G. 11.06.2001, No. 24459)
  - Customs Law no. 4458 (O.G. 04.11.1999, No. 23866)
  - Environment Law no.2872 (O.G. 11.08.1983, No.18132)

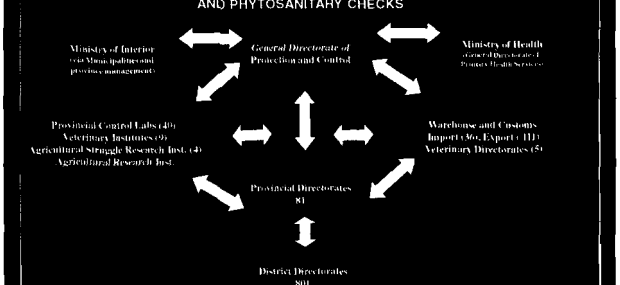
As regards the system of controls:

- Official duties on Food Safety, Veterinary and Phytosanitary area *carried out by* GDPC through:
  - 81 Province Agricultural Directorates
  - 803 District Directorates
  - National Food Reference Laboratory
  - 39 Provincial Control Laboratories
  - 8 Veterinary Control and Research Institutes
  - 1 Food Control and Central Research Institute
  - 4 Research Institute on Agricultural Struggle
- **The Ministry's Provincial Agricultural Directorates in 81 Provinces are the units responsible for food control/inspection, veterinary and phytosanitary checks.**

ORGANIZATIONAL STRUCTURE OF PROVINCIAL DIRECTORATES



COMMUNICATION OF GDPC RELATED WITH FOOD CONTROL, VETERINARY AND PHYTOSANITARY CHECKS



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MAIN TASKS AND RESPONSIBILITIES OF GDPC

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Main Tasks and Responsibilities:

- Adoption EU legislation on food safety, veterinary and phytosanitary
- Protection of animal health, control and eradication of diseases
- Approval and control of clinics, policlinics and animal hospitals
- Set up and implement animal registration system
- Sustain and protect the resources on fisheries
- Animal movement controls and quarantine measures
- Protection of plant health, phytosanitary, quarantine measures,

C*

Main Tasks and Responsibilities:

- Development environmental friendly and alternative agricultural struggle techniques
- Encouraging the use of early warning system on agricultural struggle
- Licensing machine and equipments for agricultural struggle,
- Registration and production permission of Plant Protection Products and Veterinary Drugs,
- Registration, approval and control food establishments,
- Risk based controls and inspections,
- Export and import controls,
- Establishing laboratories, authorization private control laboratories

Delegation of specific tasks:

- Delegation of certain tasks is possible on veterinary sector:
  - Private veterinarians can do vaccinate to the pets and follow up their health controls.
  - Private veterinarians can participate to the vaccination programmes prepared by the Ministry
  - Private veterinarians can work at the slaughterhouses

- Ministry can delegate certain tasks to the appointed organizations on quality, risk analysis, certification and other subjects deemed appropriate by the Ministry. (Article 24 Food law no.5179)

Delegation of specific tasks:

- Private control laboratories are appointed by the Ministry for the analysis of samples taken during export and import controls of foodstuffs acc. to the Law no. 5179.

- Control and Certification bodies are authorized for organic farming acc. to the Law no. 5262 on organic farming.

- Authorization is given to private organizations for marking wooden packaging materials.

SUPERVISION OF INSPECTION SERVICES

C*

- The Audit Commission (Chairmanship of Inspection Committee) and the GDPC is responsible for the supervision.
- Inspections of GDPC are carried out by monitoring from the written reports and with follow-up of the work being performed during on-site general inspections.
- Inspections of Audit Commission are carried out by monitored with routine inspections and special inspections in case of complaints.

C*

RESOURCES

C*

Control Expenses of MARA are financed by MARA Budget.

MARA Budget covers:

- All costs of inspections
- Personnel salary
- Expenses of MARA local units, provincial control and research institute laboratories

Fee is not taken to cover the costs occasioned by inspections of slaughterhouses, cutting plants, fish auction centres, wholesale market, milk establishments and storage facilities; controls on products; licensing and import and export controls.

C*

CONTROL SYSTEM

C*

- The control and the inspection of the food business establishments are carried out by "food inspectors" and "food inspector assistants" employed by MARA in 81 Provincial Directorates.
- *Total number of staff involved in control and inspections is 4807.*
- Food inspectors should have at least bachelors' degree in relevant field (veterinarian, agricultural engineer, food engineer, chemical engineer, chemist, etc.) and "food inspector assistants" must have at least two year degree.
- Staff to be employed as inspectors are subject to training and qualifying exam. They are also trained on regular intervals.

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- As a result of the controls and inspections, sanctions are imposed in *accordance with the Law No: 5179 for the establishments not complying* with legislation.
- Sanctions imposed are fines, taking to the court in cases of threat to human health, cessation of the production and cancellation of the approval, withdrawal of the products from the market and destruction of the products.

LABORATORIES

- Veterinary Control and Research Institutes

– Animal Health Services Department of GDPC is responsible to appoint and control Veterinary laboratories.

Laboratories;

8 Veterinary Control and Research Institutes (regional labs.for animal diseases)
Ankara National FMD Institute

–Reference Laboratories;

Ankara National FMD Institute
Etlik Central Veterinary Control and Research Institute
Pendik Veterinary Control and Research Institute
Bornova Veterinary Control and Research Institute
Konya Veterinary Control and Research Institute

- Provincial Control Laboratories

Public Health Services Department of GDPC is responsible to coordinate Provincial Control Laboratory Directorates and also appoint and control private laboratories

Laboratories for the official controls of food and feed;

39 Provincial Control Laboratories
Bursa Food Control and Central Research Institute
51 Private Control Laboratories only for food analysis (appointed by the Ministry)

Reference Laboratories;

National Food Reference Laboratory (Ankara-NEW)
Ankara Provincial Control Laboratory
Izmir Provincial Control Laboratory
Bursa Food Control and Central Research Institute

C*

- Plant Health Laboratories

Plant Health Laboratories;

4 Agricultural Struggle Research Institutes
7 Agricultural Quarantine Laboratories
5 Seed Certification Central Directorate
1 Seed Registration and Certification Central Directorate of Ankara

Agricultural Struggle Research Institutes also dealing with quarantine controls and monitoring programmes

- Reference Laboratories:

Ankara Seed Registration and Certification Central Directorate for the plant health analysis.

C*

- Accreditation of Laboratories

Accredited Laboratories acc. to EN ISO/IEC 17025 on 'General requirements for the competence of testing and calibration laboratories'

- Animal Health and food analysis:

Etlik Central Veterinary Control and Research Institute for 21 test methods

- Food and feed analysis:

Istanbul Provincial Control Laboratory
Izmir Provincial Control Laboratory
Ankara Provincial Control Laboratory
Mersin Provincial Control Laboratory
Samsun Provincial Control Laboratory
Bursa Food Control and Central Research Institute

- *Private Laboratories on food analysis;*

Aydın Exchange of Commerce Private Food Control Laboratory
Environmental Industrial Analysis Private Food Control Laboratory

C*

2009 THE YEAR OF FOOD

- 2009 – announced as the 'food year' by the MARA.
- To inform public about food safety and healthy life.
- On 14th February, 'hello 174 food line' call center got in charge. Receives calls of complaints, wishes, questions on food.

2009 THE YEAR OF FOOD

THANK YOU FOR YOUR ATTENTION

FOOD CONTROL DUTIES AND FOOD LABORATORIES

- Food control duties in Turkey are carried out by the Ministry of Agriculture and Rural Affairs,General Directorate of Protection and Control
- the aims of this directorate are ;

- To carry out food foreign trade control and inspections at each phase of the production in food production sites.
- To ensure qualified and reliable food supply.
- To perform studies in order to ensure efficient nutrition of the public.
- To prepare and publish Product Codex
- To make registrations of food packing production facilities and to permit their manufactured products.
- To prepare food industry inventories.

- As a result .The top priority of the ministry policy about food safety is the "farm-to-fork" approach by giving priority to consumer demands and the right for high quality and safe food. To achieve this, an efficient and effective food control system along the food chain should be established and implemented.

- For this purpose, food analyses are perfomed by 39 provincial control laboratories , one central institute of food control and research which are belong to the ministry and by some private laboratories which are authorized by ministry

- Most of the laboratories have been accretitated in accordance to the TS EN ISO / IEC 17025:2000 , the number of the accretitated laboratories in Turkey are increasing year by year

CENTRAL INSTITUTE of FOOD CONTROL and RESEARCH, BURSA

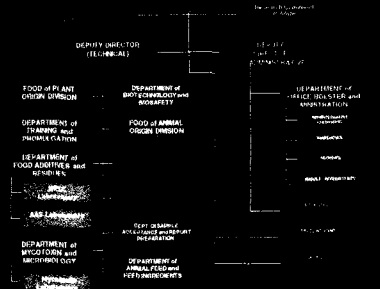
HISTORY

Our institute started its activities on 25.02.1961 in Bursa, under the name of "RESEARCH INSTITUTE of CANNED FOODS". The main objective of the institute was to build and develop the industry of canned foods in Marmara District, the region which has the greatest growth rate of fruit and vegetables in Turkey.

Name was finally changed to "CENTRAL INSTITUTE of FOOD CONTROL and RESEARCH" on the 14.11.2000 as a result of the new responsibility of food control activity; in addition to food research activities

BURSA CENTRAL INSTITUTE of FOOD CONTROL and RESEARCH

DIRECTOR



CURRENT STAFF

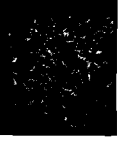
TOTAL : 137

The educational attainment of the researchers

	Bachelor	Master	Ph.D	Ass. Prof.	Total
					38
					15
					12
					3
					1
Total	17	31	14	1	72

Fields of Activity

- 1. Running required analyses collaterally with the regulations, in samples of the food, animal feed, water and industrial waste water, which were taken by Control Departments of Agricultural Provincial Directorates.



Fields of Activity

- performing physical, chemical, microbiological, food additives and residues analyses at the imported and exported foods.
- performing required analyses in the samples such as food, animal food and waste water coming from private sector with the purpose of auto - control.
- Performing some analyses in seafood products and in their hunting areas; required by international regulations.

Fields of Activity

- Conducting joint researches in the samples of food, feed, water and fisheries in the collaboration with, ministries, universities and private sector .

Fields of Activity

- *Arranging applied training for researchers, producers, and investors.
- *Providing training period for trainee's coming from domestic and foreign faculties and other schools.
- *Delivering research results to the related people and organisations as books and journals, etc.
- *Organising congresses, symposia, etc. at national and international levels.

Fields of Activity

- *Determination of the natural ingredients of food and animal feed, and their suitability of for the industry.
- *Natural ingredients of food and animal feed, and their processing technologies.
- *Enrichment of food and animal feed for nutrients and foodstuffs in order to have functional properties.

Fields of Activity

- Development of new products and determination of their processing technologies.
- *Finding new technologies of processing and preserving food and animal feed.
- *Minimising the loss of food and animal feed during ingathering, transportation, processing, storing, packing and consuming.
- *Finding most convenient analysis methods in quality control of food and animal feed and their raw material.

Fields of Activity

- *Determination of quality criteria of food and animal feed and collecting data for preparing Turkish Food Codex.
- Increasing the storage life of food and animal feed
- *Choosing applicable materials for packing of food and animal feed, quality control of tin packaging, and determination of some chemical materials migrate to the food from its packaging.
- *Preventing problems which have been faced in the technology of processing and preserving food and animal feed

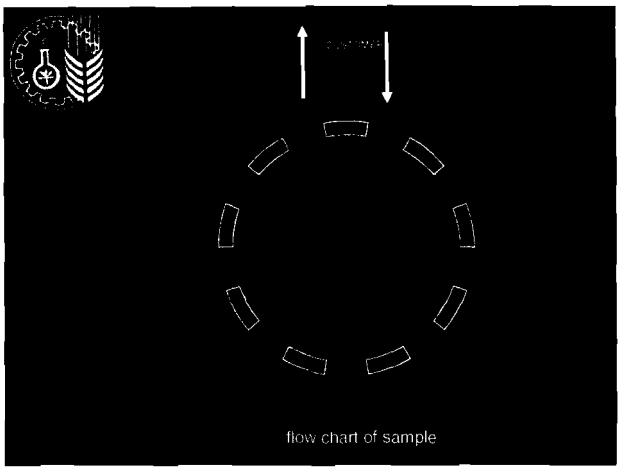
Fields of Activity

- *Determination of additives, residues and contaminants in food and animal feed
- *Finding new raw materials for animal feed industry.
- *Utilisation by-products from food industry for animal feed production.
- *Current status and capacity of food industry and animal feed industry and their possible capacities of development.

DEPARTMENT Of SAMPLE ACCEPTANCE and REPORT PREPARATION

Fields of Activity

-Acceptance of Samples
-Price Quotes
-Preparation and delivering the reports



DEPARTMENT of BIOTECHNOLOGY and BIOSAFETY

Biotechnology And Biosafety concept is a quite new issue for Turkish public. Genetically Enginereed Food has an increased its market share significiently in recent years. Therefore there is a public concern over the potential risks posed to human health and the environment by genetically modified (GMO) food. Department Of Biotechnology And Biosafety has been continuing its activities since 2004

DEPARTMENT of BIOTECHNOLOGY and BIOSAFETY

Fields of Activity

Izolation of DNA

DEPARTMENT of BIOTECHNOLOGY and BIOSAFETY

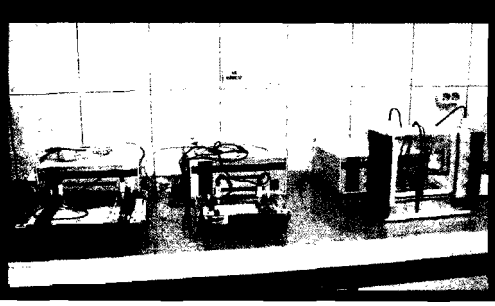
Fields of Activity

-Identification of GMO's in processed and non processed food.

DEPARTMENT of BIOTECHNOLOGY and BIOSAFETY

Fields of Activity

- Implementations of new methods for GMO's
- Training



DEPARTMENT of BIOTECHNOLOGY and BIOSAFETY

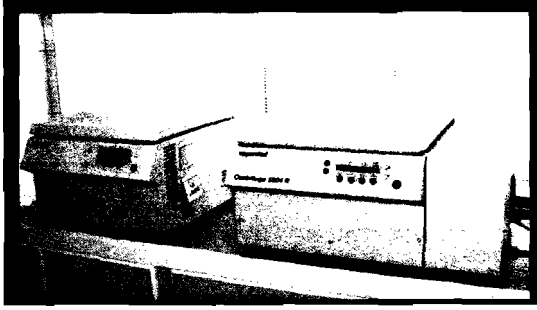
Fields of Activity

- Safety Issue of Genetically Modified Organizms:

DEPARTMENT of BIOTECHNOLOGY and BIOSAFETY

Fields of Activity

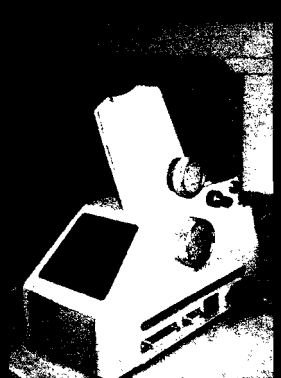
- Method validation of GMO analyses in different food matrixs



FOOD of PLANT ORIGIN DIVISION

Fields of Activity

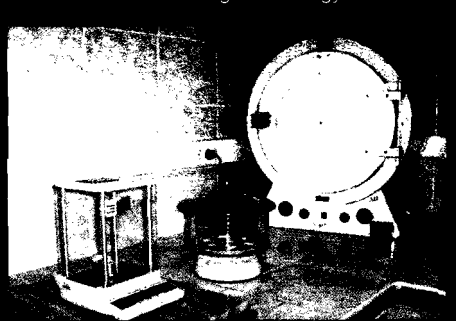
- Processing Technology of Unsaturated Vegetable Oil



FOOD of PLANT ORIGIN DIVISION

Fields of Activity

- Processing Technology of Olive Oil



FOOD of PLANT ORIGIN DIVISION

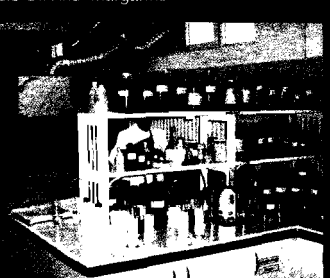
Fields of Activity

- Processing Technology of Margarine

FOOD of PLANT ORIGIN DIVISION

Fields of Activity

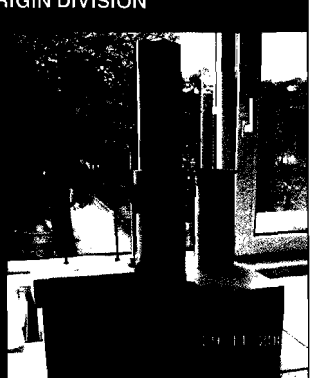
- Running Chemical, Pyhsical And Physicochemical Analyses i Vegetable Oil And Margarine



FOOD of PLANT ORIGIN DIVISION

Fields of Activity

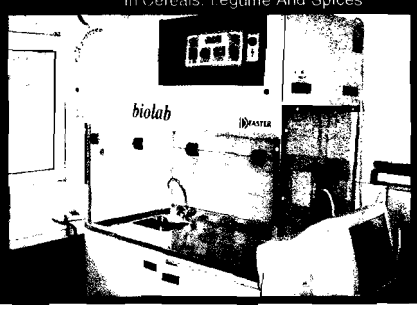
- Macaroni,Flour, Bakery and Bread
- Leguminosar
- Instant soup,
- Spices, dressings, Tea, caffee,Cocoa



FOOD of PLANT ORIGIN DIVISION

Fields of Activity

- Running Chemical, Pyhsical And Physicochemical Analyses In Cereals, Legume And Spices



FOOD of PLANT ORIGIN DIVISION

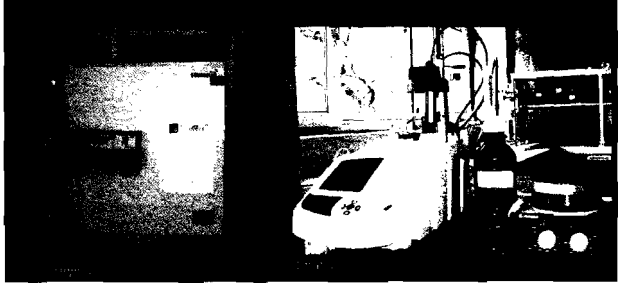
Fields of Activity

- - Canned food, Juice , Concantrated juice, dried vegetables ve frozen foods fermented foods
- - Sugar and candies products (White refined **sugar** , satiny candy , Turkish Delight etc...), traditional candies (pekmez, pestil, cezerye, helva etc.).

FOOD of PLANT ORIGIN DIVISION

Fields of Activity

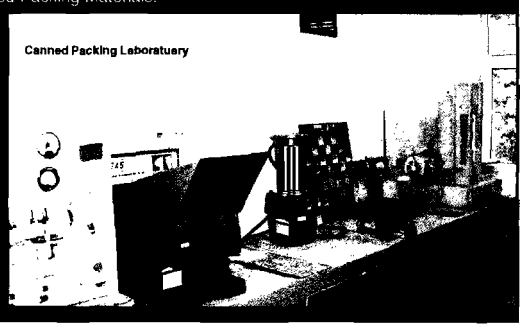
- honey, Jam ve marmalade,
- Soft Drinks, non-alcohol beverages and effervescent **powder beverages**
- Deserts made with sugar syrup (kadayif, sutlac, keskul, hosmerim, gullac etc...)



FOOD of PLANT ORIGIN DIVISION

Fields of Activity

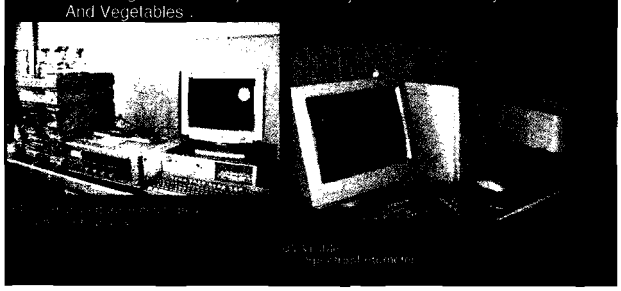
- Food Packing Materials.



FOOD of PLANT ORIGIN DIVISION

Fields of Activity

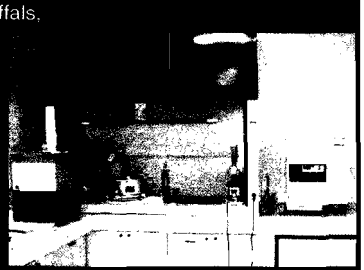
- Conducting researchs for Improving Processing Technology Of Fruits And Vegetables
- Running Chemical, Pyhsical And Physicochemical Analyses In Fruits And Vegetables .



FOOD of ANIMAL ORIGIN DIVISION

Fields of Activity

- Processing Technology Of Meat And Fishery Products
- -Processing technology of Soudjuk, sousage , salami and bacon
- Animal fats,giblets, offals,
- Eggs



FOOD of ANIMAL ORIGIN DIVISION

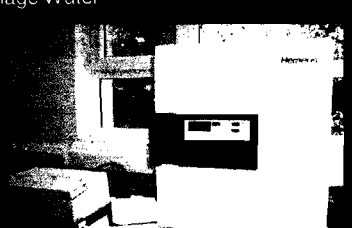
Fields of Activity

- - Residues of Veterinary medicines
- - Marine Biotoxin (ASP,DSP,PSP,vb.)
- - Processing Technology Of Fish And Fishery Products

FOOD of ANIMAL ORIGIN DIVISION

Fields of Activity

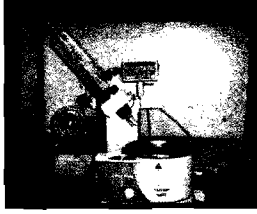
- - Tab water. Drinking water
- - Waters from fishery farms
- - Waste water
- Drainage Water



FOOD of ANIMAL ORIGIN DIVISION

Fields of Activity

- - Running Chemical, Pyhsical And Physicochemical Analyses In Meat and Fishery Products



FOOD of ANIMAL ORIGIN DIVISION

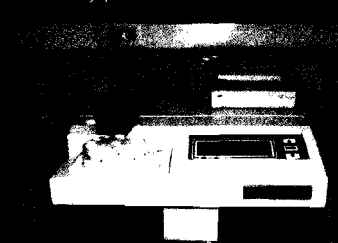
Fields of Activity –

Reproduction of Research animals

FOOD of ANIMAL ORIGIN DIVISION

Fields of Activity

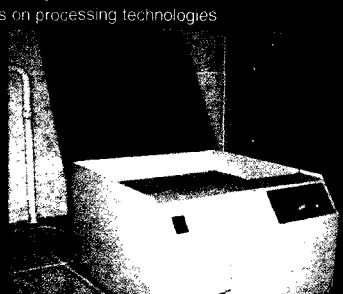
- Milk (raw,pasteurized,sterilized), powdered milk, yogurt, cheese, butter and other diary products



FOOD of ANIMAL ORIGIN DIVISION

Fields of Activity

- Improving the new products
- Conducting researches on processing technologies



FOOD of ANIMAL ORIGIN DIVISION

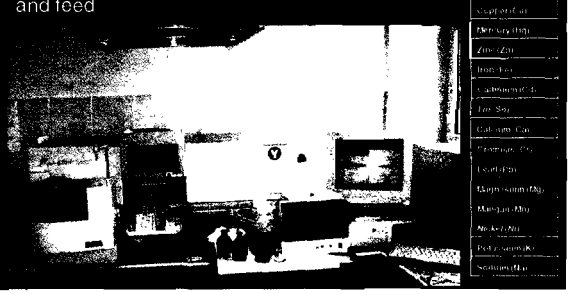
Fields of Activity

- Running Chemical, Pyhsical And Physicochemical Analyses In Milk and Diary Products

DEPARTMENT of FOOD ADDITIVES and RESIDUES

Fields of Activity

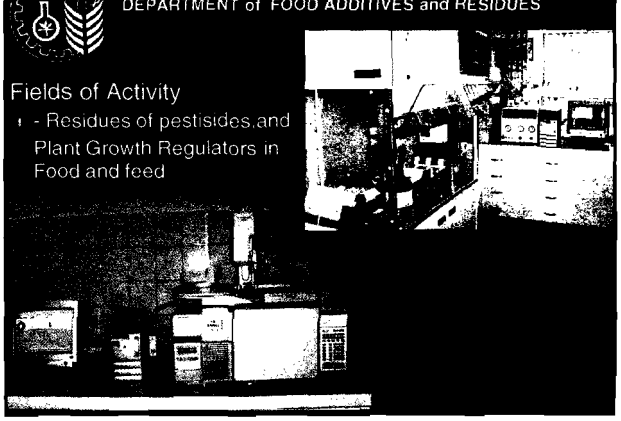
- Fesidues of minerals and metal residues in food and feed



DEPARTMENT of FOOD ADDITIVES and RESIDUES

Fields of Activity

- Residues of pestisides and Plant Growth Regulators in Food and feed

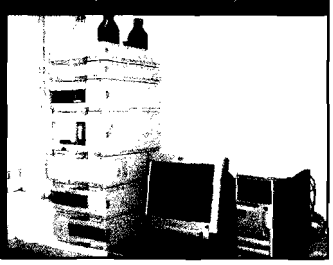


DEPARTMENT of FOOD ADDITIVES and RESIDUES

Fields of Activity

conducting researches in the topics below

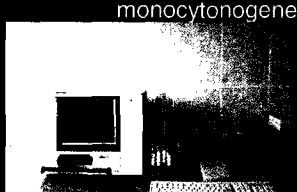
- Nitrozamin , nitrate and nitrite
- Food Additives (Preservatives, Sweeteners, Food Dyes),

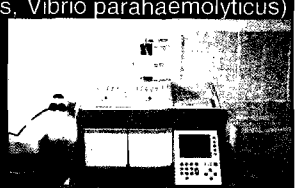


DEPARTMENT of MICROBIOLOGY and MICOTOXIN

Fields of Activity

- - Izolation and identification of some pathogen microorganisms (Salmonella spp., Shigella spp., Staphylococcus aureus, Clostridium perfringens, Listeria monocytonogenes, Vibrio parahaemolyticus)



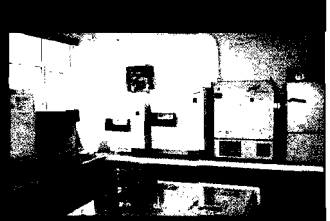


Mini Api - identification of microorganisms

DEPARTMENT of MICROBIOLOGY and MICOTOXIN

Fields of Activity

- **Izolation and identification of enteric indicator organisms**
- **(E. coli and Coliform Bacterias)**



Incubation Chamber

Microscope

DEPARTMENT of MICROBIOLOGY and MICOTOXIN

Fields of Activity

- - Izolation of mildew, mould and yeast from foods
- (Mesophilic aerobic-anaerobic bacteria
- Thermophilic aerobic-anaerobic bacteria
- Psicrophilic aerobic-anaerobic bacteria
- Acide- tolerans B.couqulans
- Spored bacteria

DEPARTMENT of MICROBIOLOGY and MYCOTOXIN

Fields of Activity

- -Mycotoxins in food and feed.

DEPARTMENT of ANIMAL FEED and FEED INGREDIENTS

Fields of Activity

- Feed Ingradients of animal or plant origin

DEPARTMENT of ANIMAL FEED and FEED INGREDIENTS

Fields of Activity

- By-products of agricultural industry

DEPARTMENT of ANIMAL FEED and FEED INGREDIENTS

Fields of Activity

- Routine Chemical, Physical And Physicochemical Analyses In Feed and Feed Ingredients

DEPARTMENT of TRAINING and PROMULGATION

Fields of Activity

- Delivering research results to the related persons and organisations as books and journals, etc.
- *Organising congress, symposia, etc. at national and international levels.

DEPARTMENT of TRAINING and PROMULGATION

- Fields of Activity –
- *Arranging applied training for researchers, producers, and investors.
- *Providing training period for trainee's coming from domestic and abroader faculties and other schools.

DEPARTMENT of OFFICE BOLSTER and MINISTRATION

Fields of Activity

- Cleaning the institute, catering etc.
- Reception, archives etc.

Provinces Sending Their Samples

- In the area of food control: Provincial Agricultural Directorates of Bursa, Kocaeli, Sakarya and Yalova send samples they have taken . At the samples of food, animal feed, drinking water and industrial waste water; required analyses are carried out by accordance with current regulations. Official reports of analysis are sent to the Directorates mentioned above.
- Our institute is the reference laboratory, appointed by EU and therefore , in the area of fishery products , Provincial Agricultural Directorates of İstanbul, Bartın, Kastamonu, Çanakkale,Zonguldak and Kırklareli send their samples to our institute

CERTIFICATE

We are an accreditated laboratuary in accordance to TS EN ISO / IEC 17025:2000; since 30th October 2004

TÜRKAK

TS EN ISO/IEC 17025

16004-01

We received the certificate of TS EN ISO 9001:2000 on 24th Oct. 2002

TÜV CERT

DIN EN ISO 9001

CA: 04100 30410

Accreditation Number: AB-0030-T

- Our Institute has been acredited by TURKAK in those food materials:
- Honey, Vegetable Products, Wine and Champagne, Wheat Flour, Vegetable Oils, Sugary Products, Foodstuffs, Cheese, Yoghurt and Ice Cream, Milk Powder, Whey Powder, Milk, Ayran, Cream, Condensed Milk, Raw Milk, Butter, Dried Fruits, Feed and Feed Raw Materials, Feed Additives , Vegetable Origin Feed. Drinking Water and Surface Water

ACCREDITATED METHODS

- Aflatoxin B1, B2, G1, G2 and *Total Aflatoxin Test*
- Benzoic Acid and Sorbic Acid Test
- Total Sulphur Dioxide(SO2) Test
- Free Fatty Acid Test
- Color Test
- Moisture Test
- Ash Test
- *Domoic acid - Amnesic* shellfish
- poisoning (ASP) toxin-Test
- Glucose and Fructose Test
- *Diastase Number Test*
- Saccharose Test
- Total Acidity
- pH Test
- Salt Test
- Crude Protein Test
- Total Sugar, Invert Sugar, Saccharose test
- Sulphur Dioxide Test
- Dried Gluten in Dried Matter Test, Iodine Number Test
- Peroxide Number Test

- Refractive Index Test
- HMF Test
- Lead Test
- Caffein Test
- *Sweetening agent - Saccharin Test*
- Acesulfam K Test
- Aspartame Test
- Total Mesophilic Aerob Bacteria
- Yeast and Mould Test
- Staphylococci Test
- Iron, Copper, Zinc, Tin, Lead
- Cadmium, Arsenic, Mercury, Aluminum
- E.coli Test, Total Coliform, Fecal coliform, Salmonella spp. Test, Listeria monocytogenes Test
- Suspended Solid Content
- Nitrogen(ammonium) Test
- Dry Matter Test
- Freezing Point Test
- Crude Protein Test
- Acidity
- Fat Test, Salt Test
- Aflatoxin A Test
- Crude Oil Test
- Crude Cellulose Test
- Crude Ash Test
- Crude Protein Test
- Dry Matter and Humidity Test
- Starch Test
- Total Phosphorous Test
- Crude Oil Test
- Brix Test
- Nitrate Test
- Patulin Test
- Nitrogen(ammonium) Test

- Nitrogen(nitrite) Test
- Oil ve Grease Test
- Conductivty Test
- Chloride Test
- Sulfate Test
- Totaf Hardness Test
- Nitrate Test
- Chemical Oxygen Demand Test
- Orthophosphate Test
- Determination of Potassium, Magnesium, Calcium, Sodium, Iron, Copper, Tin, Zinc, Aluminum, Lead, Cadmium, Arsenic, Mercury, Antimony, Selenium, Chromium, Nickel, Manganese, Cobalt by ICP-MS
- Determination of PAH Test (Benzo(a) pyrene, Benzo(b) fluoranthene, Benzo(g,h,p) perylene, Benzo(k) fluoranthene, Indeno(1,2,3-cd) pyrene by HPLC

- Determination of The Selected Organophosphorus Pesticides:Acephate, Azinphos-ethyl,
- Bromophos-ethyl, Bromophos-methyl,
- Carbosulfos, Chlorfenvinphos,
- Chlorpyrifos-ethyl,
- Chlorpyrifos-methyl, Coumaphos
- Diazinon,S-Methyl-, Diazinon,
- Dichlorvos, Dicrotophos, Dimefox,
- Disulfoton, Disulfoton, Ditalimfos,
- Ethion, Ethoprophos, Etrimfos,
- Fenamiphos, Fenchlorphos,
- Fenitrothion, Fenthion, Fonofos,
- Formothion, Fosthiazate,
- Heptenophos,
- Isazafos, Isofenphos, Malaoxon
- Malathion, Mecarbam, Mephosfolan,
- Methamidophos, Methidathion,
- Mevinphos, Monocrotophos,
- Omethoate, Paraoxon-ethyl,
- Paraoxon-methyl, Parathion-ethyl,
- Parathion-methyl, Phenthoate,
- Phorate,
- Phosalone, Phosmet, Phosphamidon,
- Pirimiphos-ethyl, Pirimiphos-methyl,
- Prothiophos, Pyrazophos,
- Pyridaphenthion, Quinalphos,
- Sulfotep, Sulprofos, Tebufos,
- Terbufos- sulfon, Tetrachlorvinphos,
- Thiometon, Tolclofos-methyl,

- Aldrin, Alpha-endosulfan, Alachlor
- Alpha HCH, Beta-endosulfan,
- Beta HCH, Bifenthrin,
- Benzoximate, Captan,
- Chlorothalonil, Chlorthal-dimethyl,
- Cyfluthrin, Cypermethrin, Delta HCH,
- Deltamethrin, Dichlofluanid, Dieldrin,
- Endosulfan sulfat, Endrin,
- Esfenvalerate, Etaconazol,
- Etofenprox, Fenvalerate, Folpet,
- Gamma HCH (Lindane), Heptachlor,
- Heptachlor endo epoxide (trans-iso-A),
- Heptachlor-exo-epoxide (cis-iso-B),
- Hexachlorobenzene, Hexaconazole
- Imazalil, Iprodione,
- Lambda-cyhalothrin, o,p-DDD,
- (2,4-DDD), o,p-DDE, (2,4-DDE),
- o,p-DDT (2,4-DDT), p,p-DDD
- (4,4-DDD), p,p-DDE (4,4-DDE),
- p,p-DDT (4,4-DDT), Oxadixyl,
- Penconazole, Permethrin,
- Procymidone, Propyzamide,
- Quintozene, Tetraconazole,
- Tetradifon,
- Trifloxystrobin, Trifluralin,
- Vinclozolin,

Thanks for your listening

UNIDO International Training Program on Food Laboratory Management and Practice

Gebze – Kocaeli, TURKEY
16-20 November 2009

BY ANNETTE NABBENGO

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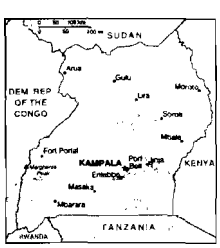
website: www.unbs.go.ug

UGANDA, THE PEARL OF AFRICA

- Uganda is a landlocked, developing country in central eastern Africa.
- Infrastructure is adequate in Kampala, the capital, but is limited in other areas.



Uganda - Geography

- Area: 241,040 sq. km. (93,070 sq. mi.); about the size of Oregon.
- Cities: *Capital*--Kampala (2002 pop. 1.2 million). *Other cities*--Jinja, Gulu, Mbale, Mbarara.
- Terrain: 18% inland water and swamp; 12% national parks, forest, and game reserves; 70% forest, woodland, grassland. Uganda has the source of river Nile at Jinja
- Climate: In the northeast, semi-arid--rainfall less than 50 cm. (20 in.); in southwest, rainfall 130 cm. (50 in.) or more. Two dry seasons: Dec.-Feb. and June-July.

Uganda - People

- Population (2007): 30.9 million.
- Annual growth rate (2004 est.): 4.0%.
- Ethnic groups: African 99%, European, Asian, Arab 1%.
- Religions (2007): Christian 85%, Muslim 12%, other 2%.
- Languages: English (official); Luganda and Swahili widely used; other Bantu and Nilotic languages.
- Education: *Attendance* (2000; primary school enrollment, public and private) - 89%. *Literacy* (2003) - 70%.
- Health: *Infant mortality rate*--86/1,000. *Life expectancy* -45.3 yrs.

Uganda - Economy

- GDP (nominal, 2007/2008): $12.3 billion.
- Inflation rate (annual headline or CPI, 2007/2008): 7%.
- Natural resources: Copper, cobalt, limestone, phosphate, oil.
- Agriculture: *Cash crops* - coffee, tea, cotton, tobacco, sugar cane, cut flowers, vanilla. *Food crops* - bananas, corn, cassava, potatoes, millet, pulses. *Livestock and fisheries* - beef, goat meat, milk, Nile perch, tilapia.
- Industry: Processing of agricultural products (cotton ginning, coffee curing), cement production, light consumer goods, textiles.
- Trade: *Exports* (2007/2008 est.) - $1.75 billion: coffee, fish and fish products, tea, electricity, horticultural products, vanilla, cut flowers, remittances from abroad.
- *Major markets* - EU, Kenya, South Africa, U.K., U.S.
- *Imports* (2007/2008 est.) - $3.4 billion: capital equipment, vehicles, petroleum, medical supplies, chemical, cereals.
- *Major suppliers* - OPEC countries, Kenya, EU, India, South Africa, China, U.S.

Uganda – Economy cont'

- Uganda's economy has great potential. Endowed with significant natural resources, including ample fertile land, regular rainfall, and mineral deposits.
- Agricultural products supply nearly all of Uganda's foreign exchange earnings, with coffee (of which Uganda is Africa's second leading producer) accounting for about 15% and fish 12% of the country's exports in 2007/2008.
- Exports of non-traditional products, including apparel, hides, skins, vanilla, vegetables, fruits, cut flowers, and fish are growing, while traditional exports such as cotton, tea, and tobacco continue to be mainstays

Food products

- Animal origin;
  - Meat,
  - Dairy and dairy products,
  - Fish and fishery products,
  - Poultry
- Plant origin;
  - Cash crops (coffee, tea)
  - food crops (cassava, potatoes, bananas etc)
  - Horticultural products

Authorities responsible for quality and safety of foods

Authorities in Uganda that are responsible for monitoring the quality of food products and consumer health;

- Ministry of health
- Ministry of Agriculture, Animal industry and Fisheries
- Uganda National Bureau of Standards
- Dairy Development Authority
- Local Government (Health inspectors)
- Coffee Development Authority

Quality Control in food industries

- Many organizations today are striving to meet and even exceed their customer requirements.
- A number of Laboratories are now moving for international accreditation to ISO 17025
- Many companies have attained ISO 9001 certification.
- However these are mainly medium-large scale industries
- Small scale industries are often left out because of the high cost of getting certification.

Quality Control cont'

- These systems are based on Quality management principles and standards. In order to gain confidence in the market, companies have gone ahead to seek independent confirmation or certification that their system are capable of delivering the desired results.

Quality Control cont'

- In a multi-disciplinary situation like UNBS, where the different units have to implement various Guidelines and Standards to demonstrate competence and adherence to the international best practices, ISO 9001 provides a good spring board on which to launch other forms of recognitions/certifications based on those sector standards and guidelines. This is so since in all situations, the management system requirements are almost the same. Implementation of ISO 9000 solves one part of the equation. What then remains is to implement the technical requirements of the relevant guideline/standards to achieve the desired recognition or certification or accreditation.

Quality Control

What is involved in Quality Control?

- **Inspection**
- **Sampling**
- **Testing**
- **Process control (*adjusting conditions*)**

Note: Quality control is the duty of the manufacturer

Quality systems

Examples of Quality System Models applied in industries:

- ISO 9000 - Quality Management System
  - *Applicable to all types of organizations*
  - *Describes the over-all Quality Management System but not the technical specifications for products*
- GMP - Good Manufacturing Practices
  - *Applicable to Food & Pharmaceutical industries, etc*
  - *Covers fundamental principles, procedures and means needed to design suitable environment for products of acceptable quality*

Quality systems

- GHP - Good Hygiene Practices
  - *Applicable to Food & related industries*
  - *Describes basic hygienic measures which should be met by industries/ establishments, and form the prerequisites to other approaches e.g. HACCP*
- **HACCP - Hazard Analysis Critical Control Point**
  - *Applicable to Food industries*
  - *Examines what can potentially go wrong at each stage in an operation, along with possible causes, and the likely effect before deploying effective control mechanisms*

Quality systems

- **ISO 14000 - Environment Management System**
  - *Applicable to all types of organizations whose operations have an impact or potential impact on the environment*
  - *Describes requirements of doing activities while catering for/preserving the environment*
- **GLP- Good Laboratory Practices**
  - *Based on ISO 17025 Standard and is applicable to testing and calibration laboratories*
  - *Describes the general requirements for competence of testing and calibration laboratories*
- **OH&S - Occupational Health and Safety System**
  - *Concerns health and safety at work places*

Quality systems

Requirements of a quality system:

1. **Management Responsibility**
2. **Quality system structure**
3. **Process control**
4. **Records and document control**
5. **Resource Management**
6. **System Review and improvement**

An example of a monitored flow of a product (fish) in Uganda

Freshness checks on arrival at the factory, Fish cleaning, filleting and skinning processes, Fillet trimming and grading

Fillet packaging, chilled fish packaging

Problems in food industries

- Poor Hygiene
- Adulteration of food by some unscrupulous traders
- Obsolete laws and regulations
- Poor law enforcement mechanisms
- Lack of awareness on the part of some industries practitioners
- Very few laboratories and the few are concentrated in the capital
- Relatively high testing charges due to the economic situation in the Country
- Lack of appreciation of standards by some manufacturers
- Lack of enough trained personnel in the manufacturing industry
- End-of-line inspection/acceptance sampling
- Dumping
- Uninformed consumers and traders
- Lack of infrastructure

Interventions

- Hygiene
  - Inspection
  - Sensitization
  - Interpretation in to local languages
- Obsolete laws
  - New laws on food safety being made
- Enforcement
  - Government agencies are pooling resources together to meet the demand and improve the situation
- Awareness
  - Sensitization/training by the government agencies

Food Laboratories in Uganda

- There are different types, some are in-house labs at factories, institutional labs like in Agric ministry (genetic lab) and then food testing labs for export and import purposes.
- Food testing labs; 5
  - Chemiphar testing lab (accredited by BELTEST)
  - UNBS Micro Lab (accredited by Sanas)
  - Government Chemists
  - SGS
  - Uganda Fisheries Lab (accredited by Sanas)

Quality Assurance in Microbiological Analysis (UNBS)

- Sample acquisition, receipt and storage
- Preparation of initial suspension and decimal dilutions (calculating measurement of uncertainty)
- Culture media: preparation, sterilisation and storage
- Culture media: performance testing, evaluation ad control
- Methods for use in performance testing are validated
- General guidance for the enumeration and identification of micro-organisms, bacteria, fungi and yeasts.
- Proficiency tests are carried out

Annette Nabbengo, 16 - 20 November 2009,
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Equipment performance and preparation of QA manual (UNBS)

- Maintenance
- Calibration
- Keeping an equipment inventory.
- ISO 17025 Quality Manual
- Conducting an internal audit

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TÜBİTAK
MAM

INTERNATIONAL WORKSHOP AND STUDY TOUR ON: FOOD LABORATORIES MANAGEMENT AND PRACTICE

UNISWORK VIII

TUBITAK

MARMARA RESEARCH CENTRE

FOOD INSTITUTE

LECTURE NOTES

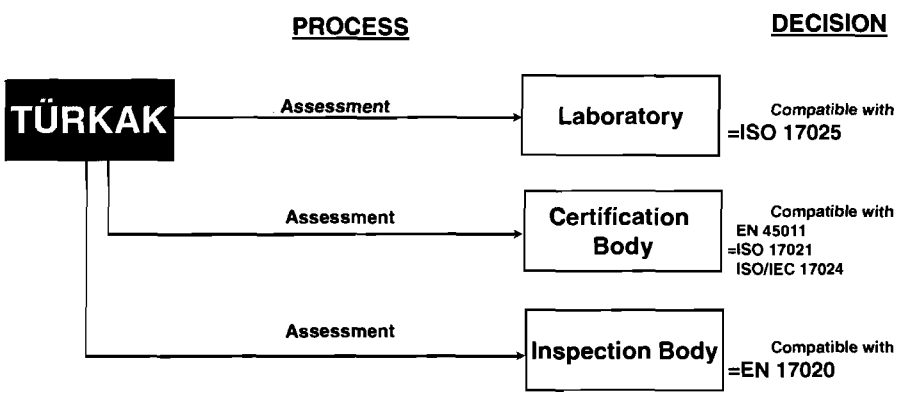
Gebze- Kocaeli, TURKEY

16-20 November 2009

Introduction to Laboratory Quality Systems and Laboratory Management

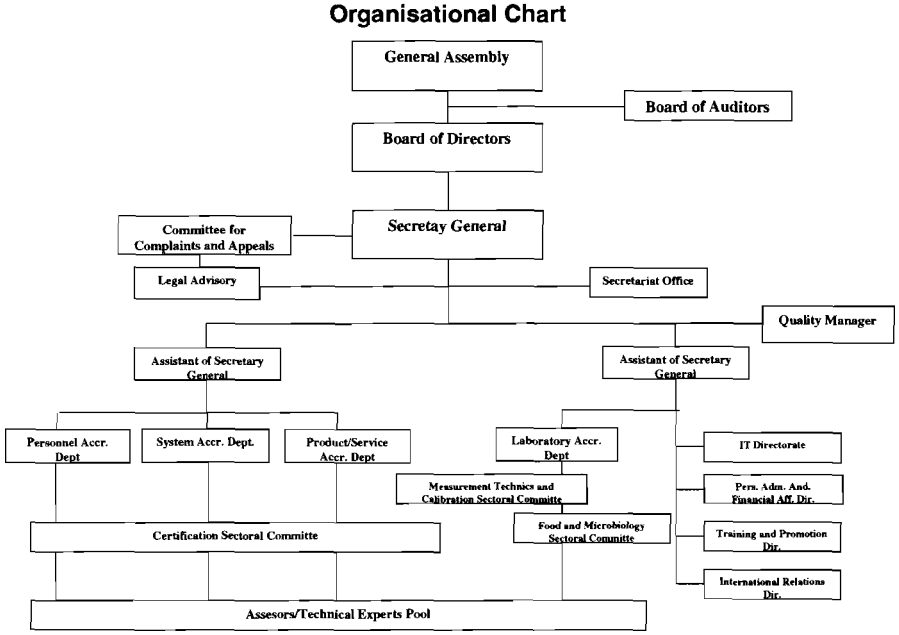
Pınar Yıldızlar AKSU
Turkish Accreditation Agency
Laboratory Accreditation Department
Case Manager, Lead Assessor

What is Accreditation Service?



Accreditation : evaluation according to nationally and internationally recognized criteria, approval of competence and assessment at regular intervals of laboratories, inspection and certification bodies

Organisational Chart



Status of TURKAK Accreditation

As at 15 March 2005

Application Department	Accredited
Laboratory Accrd. Dept.	
▪Calibration Laboratories	61
▪Testing Laboratories	239
Personnel Accred. Dept.	9
System Accred. Dept.	38
Product/Service Accrd. Dept.	
▪ Inspection Bodies	44
▪ Product Cert. Bodies	15
TOTAL	406

ISO/IEC 17025

- 1 Scope
- 2 Normative references
- 3 Terms and definitions
- **4 Management requirements**
- **5 Technical requirements**

ISO/IEC 17025

- **4 Management requirements**
  - 4.1 Organisation and management
  - 4.2 Quality system
  - 4.3 Document control
  - 4.4 Request, tender and contract review
  - 4.5 Sub-contracting of test and calibration
  - 4.6 Purchasing services and supplies
  - 4.7 Service to the client

ISO/IEC 17025

3(5)

- **4 Management requirements**
  - 4.8 Complaints
  - 4.9 Control of non-conforming T/C work
  - 4.10 Improvement
  - 4.11 Corrective action
  - 4.12 Preventive action
  - 4.13 Records
  - 4.14 Internal audits
  - 4.15 Management reviews

# ISO/IEC 17025
4(5)

- **5 Technical requirements**
  - 5.1 General
  - 5.2 Personnel
  - 5.3 Accommodation and env conditions
  - 5.4 T/C methods incl sampling
  - 5.5 Equipment

# ISO/IEC 17025
5(5)

- **5 Technical requirements**
  - 5.6 Measurement traceability
  - 5.7 Sampling
  - 5.8 Handling and transportation of items
  - 5.9 Assuring the quality of T/C results
  - 5.10 Reporting the results

ISO/IEC 17025,
4 Management requirements

Legal identity

The laboratory or the organisation of which it is part shall be an entity that can be held legally responsible

- Examples: Private company, public enterprise, public or local authority
- If the laboratory is part of a legal entity, the accreditation is granted to the legal entity in question

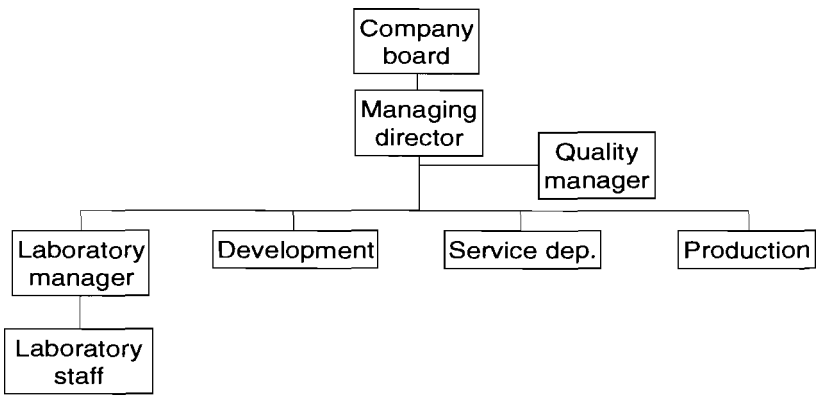
Impartiality, independence and integrity

- **Identify potential conflicts of interest**
- Separated from production
- Directly under the central quality department or top management
- Remuneration: No payment per piece of work produced
- If laboratory concerned with e.g. Design: clear separation of different responsibilities
- Bankruptcy

Management and organisation

- competent to perform tests concerned
  - personnel aware of area of responsibility
  - supervision
- technical manager and deputy technical manager,
- appointed quality manager
- document showing responsibilities, available and kept up-to-date
- Top management shall ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

Organisation chart



Quality manual and related documentation, 1/2

- Quality policy statement
- organisation and management structure
- relations between management etc. and quality system
- procedures for documentation
- job descriptions
- approved signatories
- procedures for traceability
- scope
- procedures for reviews of all new work
- reference to procedure

Quality manual and related documentation, 2/2

- proc. for handling of objects
- ref. to equipment and standards used
- ref. to proc. for calibr., verif. & maintenance of equipment
- ref. to verification practice
- proc.for feedback and corrective action
- arrangements for departures
- proc. for complaints
- proc. for confidentiality & proprietary rights
- proc. for audit and review

Quality System

- Top management shall provide evidence of commitment to the development and implementation of the management system and to continually improving its effectiveness.
- Top management shall communicate to the organization the importance of meeting customer requirements as well as statutory and regulatory requirements.
- Top management shall ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

Document control

- internally generated or from external sources
- a master list identifying the revision status of the documents
- appropriate documents available
- documents periodically reviewed
- uniquely identified
- obsolete documents suitable marked

Procedures leading to a contract shall ensure that:

- The requirements including the methods to be used are adequately defined,documented and understood
- the laboratory has the capability and resources to meet the requirements
- the appropriate test and/or calibration method is selected and capable of meeting the clients requirements

Subcontracting

- test and calibration
  - normally not
  - if subcontracting - same requirements
  - inform client in writing
  - retain details
  - register of all subcontractors
  - accredited laboratories ok.
  - not accredited - approval by accreditation body

Outside support services and supplies

- Use only those with adequate quality to sustain confidence in results.
- Procedures to ensure that purchased equipment etc. comply with specified requirements.
- Record of all "approved" suppliers

Co-operation with the client

- Afford clients co-operation
- Ensure confidentiality to other clients

Complaints...

- Documented policy and procedures
- Record all complaints
- Doubt concerning the quality of the results - audit promptly
- determine the root cause(s) of the problem
- ensure that corrective actions taken have been effective

Improvement

- The laboratory shall continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

Procedure for how documents are to be:

- **Registered**(registration identification,edition number)
- **Updated**(revision, recall or approval)

List of persons involved in these activities, showing their responsibilities and authority.

Training

Records

- **Key word: Traceability**
- maintain a suitable record system
- complying with any existing regulations
- "all" original observations, calculations etc.
- for an appropriate period
- sufficient information for permitting repetition of test or calibration
- identification of involved personnel
- safely stored, held secure and in confidence

Quality audits

- Regular reviews in order to ensure that the quality system is fully implemented in practice.
- Inspection to ensure that the quality manual and its related documents are being complied with at all levels of the work.
- Audit procedures as described in the quality manual.
- The person responsible for quality is responsible for ensuring that the audits are carried out in accordance with the plan.

Quality system reviews

- Previous revisions
- Visits of accreditation bodies
- Results from internal quality audits
- Complaints
- Resources - personnel, equipment
- Training
- Improvement
- Plan for introduction of desired changes
- Future planning
- Documentation
- Archiving

Parameters influencing on the result (5.1.1)

- Human factors
- accommodation (premises) and environment
- test / calibration method
- Equipment
- measurement traceability
- sampling
- the handling of test / calibration items

Personnel

- Education
- Training
- No formal requirements
- Annual assessment
- Technical knowledge
- Experience

Personnel
Education/Training - verification

- No formal requirements on education
- However, some accreditations are based upon certification personnel
- The competence of the laboratory personnel is verified by the technical assessors (annual assessment)
- The effectiveness of the training actions taken shall be evaluated.
- Sample - test procedure
- It is then the management´s responsibility to verify all other personnel

Personnel -

example, criteria for qualifications Technical manager of the laboratory

- At least M.Sc. In the technical field
- Five years experience in analytical testing
- Be familiar with all analytical methods of the laboratory
- Be the laboratory´s expert on the following instruments:GC, LC
- Have management experience

Personnel

Assessment of the capability to maintain competence 1/2

Procedures for

- introducing new personnel to the administrative and technical procedures of the laboratory
- training personnel to special jobs
- training personnel to meet present and future demands
- qualifying personnel to work independently

Personnel

Assessment of the capability to maintain competence 2/2

Procedures for

- participation in interlaboratory comparisons
- incorporating feedback from interlaboratory comparisons
- internal quality audits to detect non-compliances due to insufficient training, etc.

Personnel records

- Qualifications
- Training
- Experience

of the technical personnel.

Personnel - In the quality system

- Criteria of competence (qualifications) for different appointments within the laboratory (especially for "key persons")
- Criteria for how to verify that the required competence is attained
- Guarantee competent handling of methods/instruments that are not in regular use

Accommodation and environment

1/2

- Eliminate all environmental factors influencing the results
- Evaluate the environmental conditions in the laboratory
- Protect from abnormal conditions
- Certain activities might require:
  - Screened rooms
  - Stabilised mains voltage
  - Special earthing
  - Protection against vibration and noise
  - Protection against dust and humidity

Accommodation and environment
2/2

- Remaining influence parameters must be considered when calculating the total uncertainty
- Continuous control and monitoring of the environment
- Special requirements for site testing
- Controlled access to all test areas
- Adequate measures shall be taken to ensure good housekeeping

Accommodation and environment
Site testing

- Checks/calibrations of equipment not belonging to the laboratory
- Monitoring of environment is especially important
- Check for transport damages
- Is the test method applicable for site testing?

Accommodation and environment
Access to test areas

- Confidentiality and security
- Protect equipment and test samples, e.g. From contamination
- Locked doors

Methods that can be accredited

All standard and in house methods that ensure a well defined result can beincluded in an accreditation. The uncertainty of measurement must be determined, and the method must be thoroughly documented, and if possible be verified and confirmed against some other method.

ISO/IEC 17025,
5.5 Equipment

The laboratory shall be furnished with all items of equipment required for correct performance of the tests and measurements.

* Properly maintained
* Labelled or marked
* Recorded

Equipment

- Documentation regarding equipment containing at least the following:
  - Name of the item of equipment
  - Manufacturer's name, type identification and serial number
  - Date received and date placed in service
  - Current location
  - Condition when received (new, used, reconditioned etc.)
  - Details of maintenance carried out
  - History of any damage, malfunction, modification and repair.

Equipment, 3/3

- Calibration.
- Controls between calibrations.
- Procedures for taking defective equipment out of service. Labelling.
- Procedures for determining the effect of defective equipment on previous tests.
- Written instructions on the use of equipment including manuals from the manufacturer.
- Calibration labelling.

Common questions about calibration

WHAT should be calibrated

HOW IT should be calibrated

HOW OFTEN IT should be calibrated

Calibration system

- Complete documentation (method, equipment, interval, results) for all calibrations made by the laboratory on its own equipment
- System for marking instruments with regard to calibration status.
- Calibration interval for equipment.
- Traceability to national and international reference standards.
- All reference instruments calibrated at national standards laboratory or accredited laboratory.
- Reference instruments checked before and after calibration.
- Reference instruments shall be used only for calibrations.

Calibration system

- At least following shall be recorded:
  - Description of the equipment with unique identification code
  - Place of calibration
  - Calibration method
  - Reference instrument used
  - Influence parameters
  - Results and corrections
  - Measurement uncertainty
  - The person who has done the calibration
  - The date of calibration

Handling of calibration and test items

- A system for identifying the samples or items to be tested or calibrated
- Documents/marking
- Must be possible to identify sample or item with results of measurements made
- Anonymously handling, e.g. to other clients
- Bonded storage where necessary

Handling of calibration and test items

- Prevent damage, e.g. contamination, corrosion or the application of stresses
- Any relevant instructions shall be observed
- Clear rules for the receipt, retention and disposal of samples or items

Handling of calibration and test items

- Upon receipt, the condition of the calibration or test item, including any abnormalities or departures from standard condition as prescribed in the relevant calibration or test method, shall be recorded
- Where there is any doubt as to the item's suitability for calibration or test, the laboratory shall consult the client for further instruction before proceeding

Test comparisons

To investigate the laboratories´ability to perform tests within their scope of accreditation.

Quality control data shall be analysed and, where they are found to be outside pre-defined criteria,planned action shall be taken to correct the problem and to prevent incorrect results from being reported.

Certificates and reports
Contents

- Headings shall be standardised as far as possible
- Corrections or additions after issue: "Amendment/Addendum to test report serial number ... (or as otherwise identified)"
- Shall not include any advice or recommendation arising from the results
- Any extrapolation of results from statistically selected test objects to the properties of a lot, batch or production quantity shall be contained in a separate document.

Certificates and reports
Contents

- Results from not accredited test method may be included in the test report. However, they shall cover only the minority of the report and shall be indicated.
- Results from subcontractors may be included. However, the shall cover only the minority of the report and shall be indicated.

Certificates and reports
Contents

- The certificate or report shall include characterisation and condition of the calibration or test item.
- If clients require transmission of results by facsimile, staff shall follow documented procedures.

Guidance Documents In Laboratory Accreditation

- EA Guidelines
  - **EA-4/02 Expressions of the Uncertainty of Measurements in Calibration**
  - **EA-4/07 Traceability of Measuring and Test Equipment *to National Standards***
  - **EA-4/09 Accreditation for Sensory Testing Laboratories**
  - **EA-4/10Accreditation for Laboratories Performing Microbiological Testing**
  - **EA-4/14 Selection and Use of Reference Materials**
  - **EA-4/15 Accreditation for Bodies Performing non-Destructive Testing**
  - **EA-4/16 EA Guidelines on the Expression of Uncertainty in Quantitative testing**

Guidance Documents In Laboratory Accreditation

- ILAC Guidelines
  - **ILAC G7:1996 Accreditation Requirements and Operating Criteria for Horseracing Laboratories**
  - **ILAC G8:1996 Guidelines on Assessment and Reporting of Compliance with Specification**
  - **ILAC G9:2005 Guidelines for the Selection and Use of Reference Materials**
  - **ILAC G12:2000 Guidelines for the Requirements for the Competence of Reference Materials Producers**
  - **ILAC G13:2000 Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes**
  - **ILAC G17:2002 Introducing the Concept of Uncertainty of Measurement in Testing in Association with the Application of the Standard ISO/IEC 17025**
  - **ILAC G18:2002 The Scope of Accreditation and Consideration of Methods and Criteria for the Assessment of the Scope in Testing**
  - **ILAC G19:2002 Guidelines for Forensic Science Laboratories**
  - **ILAC G20:2002 Guidelines on Grading of Non-conformities**
  - **ILAC G22:2004 Use of Proficiency Testing as a Tool for Accreditation in Testing**

INTERNATIONAL STANDARD

ISO/IEC 17025

Second edition
2005-05-15

General requirements for the competence of testing and calibration laboratories

Exigences générales concernant la compétence des laboratoires d'étalonnages et d'essais

Reference number
ISO/IEC 17025:2005(E)

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Contents

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Foreword

ISO (the International Organization for Standardization) and IEC (the International Electrotechnical Commission) form the specialized system for worldwide standardization. National bodies that are members of ISO or IEC participate in the development of International Standards through technical committees established by the respective organization to deal with particular fields of technical activity. ISO and IEC technical committees collaborate in fields of mutual interest. Other international organizations, governmental and non-governmental, in liaison with ISO and IEC, also take part in the work. In the field of conformity assessment, the ISO Committee on conformity assessment (CASCO) is responsible for the development of International Standards and Guides.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

Draft International Standards are circulated to the national bodies for voting. Publication as an International Standard requires approval by at least 75 % of the national bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/IEC 17025 was prepared by the *ISO Committee on conformity assessment* (CASCO).

It was circulated for voting to the national bodies of both ISO and IEC, and was approved by both organizations.

This second edition cancels and replaces the first edition (ISO/IEC 17025:1999), which has been technically revised.

Introduction

The first edition (1999) of this International Standard was produced as the result of extensive experience in the implementation of ISO/IEC Guide 25 and EN 45001, both of which it replaced. It contained all of the requirements that testing and calibration laboratories have to meet if they wish to demonstrate that they operate a management system, are technically competent, and are able to generate technically valid results.

The first edition referred to ISO 9001:1994 and ISO 9002:1994. These standards have been superseded by ISO 9001:2000, which made an alignment of ISO/IEC 17025 necessary. In this second edition, clauses have been amended or added only when considered necessary in the light of ISO 9001:2000.

Accreditation bodies that recognize the competence of testing and calibration laboratories should use this International Standard as the basis for their accreditation. Clause 4 specifies the requirements for sound management. Clause 5 specifies the requirements for technical competence for the type of tests and/or calibrations the laboratory undertakes.

Growth in the use of management systems generally has increased the need to ensure that laboratories which form part of larger organizations or offer other services can operate to a quality management system that is seen as compliant with ISO 9001 as well as with this International Standard. Care has been taken, therefore, to incorporate all those requirements of ISO 9001 that are relevant to the scope of testing and calibration services that are covered by the laboratory's management system.

Testing and calibration laboratories that comply with this International Standard will therefore also operate in accordance with ISO 9001.

Conformity of the quality management system within which the laboratory operates to the requirements of ISO 9001 does not of itself demonstrate the competence of the laboratory to produce technically valid data and results. Nor does demonstrated conformity to this International Standard imply conformity of the quality management system within which the laboratory operates to all the requirements of ISO 9001.

The acceptance of testing and calibration results between countries should be facilitated if laboratories comply with this International Standard and if they obtain accreditation from bodies which have entered into mutual recognition agreements with equivalent bodies in other countries using this International Standard.

The use of this International Standard will facilitate cooperation between laboratories and other bodies, and assist in the exchange of information and experience, and in the harmonization of standards and procedures.

General requirements for the competence of testing and calibration laboratories

1 Scope

1.1 This International Standard specifies the general requirements for the competence to carry out tests and/or calibrations, including sampling. It covers testing and calibration performed using standard methods, non-standard methods, and laboratory-developed methods.

1.2 This International Standard is applicable to all organizations performing tests and/or calibrations. These include, for example, first-, second- and third-party laboratories, and laboratories where testing and/or calibration forms part of inspection and product certification.

This International Standard is applicable to all laboratories regardless of the number of personnel or the extent of the scope of testing and/or calibration activities. When a laboratory does not undertake one or more of the activities covered by this International Standard, such as sampling and the design/development of new methods, the requirements of those clauses do not apply.

1.3 The notes given provide clarification of the text, examples and guidance. They do not contain requirements and do not form an integral part of this International Standard.

1.4 This International Standard is for use by laboratories in developing their management system for quality, administrative and technical operations. Laboratory customers, regulatory authorities and accreditation bodies may also use it in confirming or recognizing the competence of laboratories. This International Standard is not intended to be used as the basis for certification of laboratories.

NOTE 1 The term 'management system' in this International Standard means the quality, administrative and technical systems that govern the operations of a laboratory.

NOTE 2 Certification of a management system is sometimes also called registration.

1.5 Compliance with regulatory and safety requirements on the operation of laboratories is not covered by this International Standard.

1.6 If testing and calibration laboratories comply with the requirements of this International Standard, they will operate a quality management system for their testing and calibration activities that also meets the principles of ISO 9001. Annex A provides nominal cross-references between this International Standard and ISO 9001. This International Standard covers technical competence requirements that are not covered by ISO 9001.

NOTE 1 It might be necessary to explain or interpret certain requirements in this International Standard to ensure that the requirements are applied in a consistent manner. Guidance for establishing applications for specific fields, especially for accreditation bodies (see ISO/IEC 17011) is given in Annex B.

NOTE 2 If a laboratory wishes accreditation for part or all of its testing and calibration activities, it should select an accreditation body that operates in accordance with ISO/IEC 17011.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/IEC 17000, *Conformity assessment — Vocabulary and general principles*

VIM, *International vocabulary of basic and general terms in metrology*, issued by BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML

NOTE Further related standards, guides, etc. on subjects included in this International Standard are given in the Bibliography.

3 Terms and definitions

For the purposes of this document, the relevant terms and definitions given in ISO/IEC 17000 and VIM apply.

NOTE General definitions related to quality are given in ISO 9000, whereas ISO/IEC 17000 gives definitions specifically related to certification and laboratory accreditation. Where different definitions are given in ISO 9000, the definitions in ISO/IEC 17000 and VIM are preferred.

4 Management requirements

4.1 Organization

4.1.1 The laboratory or the organization of which it is part shall be an entity that can be held legally responsible.

4.1.2 It is the responsibility of the laboratory to carry out its testing and calibration activities in such a way as to meet the requirements of this International Standard and to satisfy the needs of the customer, the regulatory authorities or organizations providing recognition.

4.1.3 The management system shall cover work carried out in the laboratory's permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

4.1.4 If the laboratory is part of an organization performing activities other than testing and/or calibration, the responsibilities of key personnel in the organization that have an involvement or influence on the testing and/or calibration activities of the laboratory shall be defined in order to identify potential conflicts of interest.

NOTE 1 Where a laboratory is part of a larger organization, the organizational arrangements should be such that departments having conflicting interests, such as production, commercial marketing or financing do not adversely influence the laboratory's compliance with the requirements of this International Standard.

NOTE 2 If the laboratory wishes to be recognized as a third-party laboratory, it should be able to demonstrate that it is impartial and that it and its personnel are free from any undue commercial, financial and other pressures which might influence their technical judgement. The third-party testing or calibration laboratory should not engage in any activities that may endanger the trust in its independence of judgement and integrity in relation to its testing or calibration activities.

4.1.5 The laboratory shall

a) have managerial and technical personnel who, irrespective of other responsibilities, have the authority and resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system, and to identify the occurrence of departures from the management system or from the procedures for performing tests and/or calibrations, and to initiate actions to prevent or minimize such departures (see also 5.2);

b) have arrangements to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;

c) have policies and procedures to ensure the protection of its customers' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results;

d) have policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgement or operational integrity;

e) define the organization and management structure of the laboratory, its place in any parent organization, and the relationships between quality management, technical operations and support services;

f) specify the responsibility, authority and interrelationships of all personnel who manage, perform or verify work affecting the quality of the tests and/or calibrations;

g) provide adequate supervision of testing and calibration staff, including trainees, by persons familiar with methods and procedures, purpose of each test and/or calibration, and with the assessment of the test or calibration results;

h) have technical management which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of laboratory operations;

i) appoint a member of staff as quality manager (however named) who, irrespective of other duties and responsibilities, shall have defined responsibility and authority for ensuring that the management system related to quality is implemented and followed at all times; the quality manager shall have direct access to the highest level of management at which decisions are made on laboratory policy or resources;

j) appoint deputies for key managerial personnel (see Note);

k) ensure that its personnel are aware of the relevance and importance of their activities and how they contribute to the achievement of the objectives of the management system.

NOTE Individuals may have more than one function and it may be impractical to appoint deputies for every function.

4.1.6 Top management shall ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

4.2 Management system

4.2.1 The laboratory shall establish, implement and maintain a management system appropriate to the scope of its activities. The laboratory shall document its policies, systems, programmes, procedures and instructions to the extent necessary to assure the quality of the test and/or calibration results. The system's documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

4.2.2 The laboratory's management system policies related to quality, including a quality policy statement, shall be defined in a quality manual (however named). The overall objectives shall be established, and shall be reviewed during management review. The quality policy statement shall be issued under the authority of top management. It shall include at least the following:

a) the laboratory management's commitment to good professional practice and to the quality of its testing and calibration in servicing its customers;

b) the management's statement of the laboratory's standard of service;

c) the purpose of the management system related to quality;

d) a requirement that all personnel concerned with testing and calibration activities within the laboratory familiarize themselves with the quality documentation and implement the policies and procedures in their work; and

e) the laboratory management's commitment to comply with this International Standard and to continually improve the effectiveness of the management system.

NOTE The quality policy statement should be concise and may include the requirement that tests and/or calibrations shall always be carried out in accordance with stated methods and customers' requirements. When the test and/or calibration laboratory is part of a larger organization, some quality policy elements may be in other documents.

4.2.3 Top management shall provide evidence of commitment to the development and implementation of the management system and to continually improving its effectiveness.

4.2.4 Top management shall communicate to the organization the importance of meeting customer requirements as well as statutory and regulatory requirements.

4.2.5 The quality manual shall include or make reference to the supporting procedures including technical procedures. It shall outline the structure of the documentation used in the management system.

4.2.6 The roles and responsibilities of technical management and the quality manager, including their responsibility for ensuring compliance with this International Standard, shall be defined in the quality manual.

4.2.7 Top management shall ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

4.3 Document control

4.3.1 General

The laboratory shall establish and maintain procedures to control all documents that form part of its management system (internally generated or from external sources), such as regulations, standards, other normative documents, test and/or calibration methods, as well as drawings, software, specifications, instructions and manuals.

NOTE 1 In this context "document" could be policy statements, procedures, specifications, calibration tables, charts, text books, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hard copy or electronic, and they may be digital, analog, photographic or written.

NOTE 2 The control of data related to testing and calibration is covered in 5.4.7. The control of records is covered in 4.13.

4.3.2 Document approval and issue

4.3.2.1 All documents issued to personnel in the laboratory as part of the management system shall be reviewed and approved for use by authorized personnel prior to issue. A master list or an equivalent document control procedure identifying the current revision status and distribution of documents in the management system shall be established and shall be readily available to preclude the use of invalid and/or obsolete documents.

4.3.2.2 The procedure(s) adopted shall ensure that:

a) authorized editions of appropriate documents are available at all locations where operations essential to the effective functioning of the laboratory are performed;

b) documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements;

c) invalid or obsolete documents are promptly removed from all points of issue or use, or otherwise assured against unintended use;

d) obsolete documents retained for either legal or knowledge preservation purposes are suitably marked.

4.3.2.3 Management system documents generated by the laboratory shall be uniquely identified. Such identification shall include the date of issue and/or revision identification, page numbering, the total number of pages or a mark to signify the end of the document, and the issuing authority(ies).

4.3.3 Document changes

4.3.3.1 Changes to documents shall be reviewed and approved by the same function that performed the original review unless specifically designated otherwise. The designated personnel shall have access to pertinent background information upon which to base their review and approval.

4.3.3.2 Where practicable, the altered or new text shall be identified in the document or the appropriate attachments.

4.3.3.3 If the laboratory's document control system allows for the amendment of documents by hand pending the re-issue of the documents, the procedures and authorities for such amendments shall be defined. Amendments shall be clearly marked, initialled and dated. A revised document shall be formally re-issued as soon as practicable.

4.3.3.4 Procedures shall be established to describe how changes in documents maintained in computerized systems are made and controlled.

4.4 Review of requests, tenders and contracts

4.4.1 The laboratory shall establish and maintain procedures for the review of requests, tenders and contracts. The policies and procedures for these reviews leading to a contract for testing and/or calibration shall ensure that:

a) the requirements, including the methods to be used, are adequately defined, documented and understood (see 5.4.2);

b) the laboratory has the capability and resources to meet the requirements;

c) the appropriate test and/or calibration method is selected and is capable of meeting the customers' requirements (see 5.4.2).

Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the customer.

NOTE 1 The request, tender and contract review should be conducted in a practical and efficient manner, and the effect of financial, legal and time schedule aspects should be taken into account. For internal customers, reviews of requests, tenders and contracts can be performed in a simplified way.

NOTE 2 The review of capability should establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests and/or calibrations in question. The review may also encompass results of earlier participation in interlaboratory comparisons or proficiency testing and/or the running of trial test or calibration programmes using samples or items of known value in order to determine uncertainties of measurement, limits of detection, confidence limits, etc.

NOTE 3 A contract may be any written or oral agreement to provide a customer with testing and/or calibration services.

4.4.2 Records of reviews, including any significant changes, shall be maintained. Records shall also be maintained of pertinent discussions with a customer relating to the customer's requirements or the results of the work during the period of execution of the contract.

NOTE For review of routine and other simple tasks, the date and the identification (e.g. the initials) of the person in the laboratory responsible for carrying out the contracted work are considered adequate. For repetitive routine tasks, the review need be made only at the initial enquiry stage or on granting of the contract for on-going routine work performed under a general agreement with the customer, provided that the customer's requirements remain unchanged. For new, complex or advanced testing and/or calibration tasks, a more comprehensive record should be maintained.

4.4.3 The review shall also cover any work that is subcontracted by the laboratory.

4.4.4 The customer shall be informed of any deviation from the contract.

4.4.5 If a contract needs to be amended after work has commenced, the same contract review process shall be repeated and any amendments shall be communicated to all affected personnel.

4.5 Subcontracting of tests and calibrations

4.5.1 When a laboratory subcontracts work, whether because of unforeseen reasons (e.g. workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g. through permanent subcontracting, agency or franchising arrangements), this work shall be placed with a competent subcontractor. A competent subcontractor is one that, for example, complies with this International Standard for the work in question.

4.5.2 The laboratory shall advise the customer of the arrangement in writing and, when appropriate, gain the approval of the customer, preferably in writing.

4.5.3 The laboratory is responsible to the customer for the subcontractor's work, except in the case where the customer or a regulatory authority specifies which subcontractor is to be used.

4.5.4 The laboratory shall maintain a register of all subcontractors that it uses for tests and/or calibrations and a record of the evidence of compliance with this International Standard for the work in question.

4.6 Purchasing services and supplies

4.6.1 The laboratory shall have a policy and procedure(s) for the selection and purchasing of services and supplies it uses that affect the quality of the tests and/or calibrations. Procedures shall exist for the purchase, reception and storage of reagents and laboratory consumable materials relevant for the tests and calibrations.

4.6.2 The laboratory shall ensure that purchased supplies and reagents and consumable materials that affect the quality of tests and/or calibrations are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements defined in the methods for the tests and/or calibrations concerned. These services and supplies used shall comply with specified requirements. Records of actions taken to check compliance shall be maintained.

4.6.3 Purchasing documents for items affecting the quality of laboratory output shall contain data describing the services and supplies ordered. These purchasing documents shall be reviewed and approved for technical content prior to release.

NOTE The description may include type, class, grade, precise identification, specifications, drawings, inspection instructions, other technical data including approval of test results, the quality required and the management system standard under which they were made.

4.6.4 The laboratory shall evaluate suppliers of critical consumables, supplies and services which affect the quality of testing and calibration, and shall maintain records of these evaluations and list those approved.

4.7 Service to the customer

4.7.1 The laboratory shall be willing to cooperate with customers or their representatives in clarifying the customer's request and in monitoring the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other customers.

NOTE 1 Such cooperation may include:

a) providing the customer or the customer's representative reasonable access to relevant areas of the laboratory for the witnessing of tests and/or calibrations performed for the customer;

b) preparation, packaging, and dispatch of test and/or calibration items needed by the customer for verification purposes.

NOTE 2 Customers value the maintenance of good communication, advice and guidance in technical matters, and opinions and interpretations based on results. Communication with the customer, especially in large assignments, should be maintained throughout the work. The laboratory should inform the customer of any delays or major deviations in the performance of the tests and/or calibrations.

4.7.2 The laboratory shall seek feedback, both positive and negative, from its customers. The feedback shall be used and analysed to improve the management system, testing and calibration activities and customer service.

NOTE Examples of the types of feedback include customer satisfaction surveys and review of test or calibration reports with customers.

4.8 Complaints

The laboratory shall have a policy and procedure for the resolution of complaints received from customers or other parties. Records shall be maintained of all complaints and of the investigations and corrective actions taken by the laboratory (see also 4.11).

4.9 Control of nonconforming testing and/or calibration work

4.9.1 The laboratory shall have a policy and procedures that shall be implemented when any aspect of its testing and/or calibration work, or the results of this work, do not conform to its own procedures or the agreed requirements of the customer. The policy and procedures shall ensure that:

a) the responsibilities and authorities for the management of nonconforming work are designated and actions (including halting of work and withholding of test reports and calibration certificates, as necessary) are defined and taken when nonconforming work is identified;

b) an evaluation of the significance of the nonconforming work is made;

c) correction is taken immediately, together with any decision about the acceptability of the nonconforming work;

d) where necessary, the customer is notified and work is recalled;

e) the responsibility for authorizing the resumption of work is defined.

NOTE Identification of nonconforming work or problems with the management system or with testing and/or calibration activities can occur at various places within the management system and technical operations. Examples are customer complaints, quality control, instrument calibration, checking of consumable materials, staff observations or supervision, test report and calibration certificate checking, management reviews and internal or external audits.

4.9.2 Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the corrective action procedures given in 4.11 shall be promptly followed.

4.10 Improvement

The laboratory shall continually improve the effectiveness of its management system through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive actions and management review.

4.11 Corrective action

4.11.1 General

The laboratory shall establish a policy and a procedure and shall designate appropriate authorities for implementing corrective action when nonconforming work or departures from the policies and procedures in the management system or technical operations have been identified.

NOTE A problem with the management system or with the technical operations of the laboratory may be identified through a variety of activities, such as control of nonconforming work, internal or external audits, management reviews, feedback from customers and from staff observations.

4.11.2 Cause analysis

The procedure for corrective action shall start with an investigation to determine the root cause(s) of the problem.

NOTE Cause analysis is the key and sometimes the most difficult part in the corrective action procedure. Often the root cause is not obvious and thus a careful analysis of all potential causes of the problem is required. Potential causes could include customer requirements, the samples, sample specifications, methods and procedures, staff skills and training, consumables, or equipment and its calibration.

4.11.3 Selection and implementation of corrective actions

Where corrective action is needed, the laboratory shall identify potential corrective actions. It shall select and implement the action(s) most likely to eliminate the problem and to prevent recurrence.

Corrective actions shall be to a degree appropriate to the magnitude and the risk of the problem.

The laboratory shall document and implement any required changes resulting from corrective action investigations.

4.11.4 Monitoring of corrective actions

The laboratory shall monitor the results to ensure that the corrective actions taken have been effective.

4.11.5 Additional audits

Where the identification of nonconformities or departures casts doubts on the laboratory's compliance with its own policies and procedures, or on its compliance with this International Standard, the laboratory shall ensure that the appropriate areas of activity are audited in accordance with 4.14 as soon as possible.

NOTE Such additional audits often follow the implementation of the corrective actions to confirm their effectiveness. An additional audit should be necessary only when a serious issue or risk to the business is identified.

4.12 Preventive action

4.12.1 Needed improvements and potential sources of nonconformities, either technical or concerning the management system, shall be identified. When improvement opportunities are identified or if preventive action is required, action plans shall be developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformities and to take advantage of the opportunities for improvement.

4.12.2 Procedures for preventive actions shall include the initiation of such actions and the application of controls to ensure that they are effective.

NOTE 1 Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

NOTE 2 Apart from the review of the operational procedures, the preventive action might involve analysis of data, including trend and risk analyses and proficiency-testing results.

4.13 Control of records

4.13.1 General

4.13.1.1 The laboratory shall establish and maintain procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. Quality records shall include reports from internal audits and management reviews as well as records of corrective and preventive actions.

4.13.1.2 All records shall be legible and shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Retention times of records shall be established.

NOTE Records may be in any media, such as hard copy or electronic media.

4.13.1.3 All records shall be held secure and in confidence.

4.13.1.4 The laboratory shall have procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records.

4.13.2 Technical records

4.13.2.1 The laboratory shall retain records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report or calibration certificate issued, for a defined period. The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original. The records shall include the identity of personnel responsible for the sampling, performance of each test and/or calibration and checking of results.

NOTE 1 In certain fields it may be impossible or impractical to retain records of all original observations.

NOTE 2 Technical records are accumulations of data (see 5.4.7) and information which result from carrying out tests and/or calibrations and which indicate whether specified quality or process parameters are achieved. They may include forms, contracts, work sheets, work books, check sheets, work notes, control graphs, external and internal test reports and calibration certificates, customers' notes, papers and feedback.

4.13.2.2 Observations, data and calculations shall be recorded at the time they are made and shall be identifiable to the specific task.

4.13.2.3 When mistakes occur in records, each mistake shall be crossed out, not erased, made illegible or deleted, and the correct value entered alongside. All such alterations to records shall be signed or initialled by the person making the correction. In the case of records stored electronically, equivalent measures shall be taken to avoid loss or change of original data.

4.14 Internal audits

4.14.1 The laboratory shall periodically, and in accordance with a predetermined schedule and procedure, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the management system and this International Standard. The internal audit programme shall address all elements of the management system, including the testing and/or calibration activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

NOTE The cycle for internal auditing should normally be completed in one year.

4.14.2 When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test or calibration results, the laboratory shall take timely corrective action, and shall notify customers in writing if investigations show that the laboratory results may have been affected.

4.14.3 The area of activity audited, the audit findings and corrective actions that arise from them shall be recorded.

4.14.4 Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

4.15 Management reviews

4.15.1 In accordance with a predetermined schedule and procedure, the laboratory's top management shall periodically conduct a review of the laboratory's management system and testing and/or calibration activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review shall take account of:

— the suitability of policies and procedures;

— reports from managerial and supervisory personnel;

— the outcome of recent internal audits;

— corrective and preventive actions;

— assessments by external bodies;

— the results of interlaboratory comparisons or proficiency tests;

— changes in the volume and type of the work;

— customer feedback;

— complaints;

— recommendations for improvement;

— other relevant factors, such as quality control activities, resources and staff training.

NOTE 1 A typical period for conducting a management review is once every 12 months.

NOTE 2 Results should feed into the laboratory planning system and should include the goals, objectives and action plans for the coming year.

NOTE 3 A management review includes consideration of related subjects at regular management meetings.

4.15.2 Findings from management reviews and the actions that arise from them shall be recorded. The management shall ensure that those actions are carried out within an appropriate and agreed timescale.

5 Technical requirements

5.1 General

5.1.1 Many factors determine the correctness and reliability of the tests and/or calibrations performed by a laboratory. These factors include contributions from:

— human factors (5.2);

— accommodation and environmental conditions (5.3);

— test and calibration methods and method validation (5.4);

— equipment (5.5);

— measurement traceability (5.6);

— sampling (5.7);

— the handling of test and calibration items (5.8).

5.1.2 The extent to which the factors contribute to the total uncertainty of measurement differs considerably between (types of) tests and between (types of) calibrations. The laboratory shall take account of these factors in developing test and calibration methods and procedures, in the training and qualification of personnel, and in the selection and calibration of the equipment it uses.

5.2 Personnel

5.2.1 The laboratory management shall ensure the competence of all who operate specific equipment, perform tests and/or calibrations, evaluate results, and sign test reports and calibration certificates. When using staff who are undergoing training, appropriate supervision shall be provided. Personnel performing specific tasks shall be qualified on the basis of appropriate education, training, experience and/or demonstrated skills, as required.

NOTE 1 In some technical areas (e.g. non-destructive testing) it may be required that the personnel performing certain tasks hold personnel certification. The laboratory is responsible for fulfilling specified personnel certification requirements. The requirements for personnel certification might be regulatory, included in the standards for the specific technical field, or required by the customer.

NOTE 2 The personnel responsible for the opinions and interpretation included in test reports should, in addition to the appropriate qualifications, training, experience and satisfactory knowledge of the testing carried out, also have:

— relevant knowledge of the technology used for the manufacturing of the items, materials, products, etc. tested, or the way they are used or intended to be used, and of the defects or degradations which may occur during or in service;

— knowledge of the general requirements expressed in the legislation and standards; and

— an understanding of the significance of deviations found with regard to the normal use of the items, materials, products, etc. concerned.

5.2.2 The management of the laboratory shall formulate the goals with respect to the education, training and skills of the laboratory personnel. The laboratory shall have a policy and procedures for identifying training needs and providing training of personnel. The training programme shall be relevant to the present and anticipated tasks of the laboratory. The effectiveness of the training actions taken shall be evaluated.

5.2.3 The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's management system.

5.2.4 The laboratory shall maintain current job descriptions for managerial, technical and key support personnel involved in tests and/or calibrations.

NOTE Job descriptions can be defined in many ways. As a minimum, the following should be defined:

— the responsibilities with respect to performing tests and/or calibrations;

— the responsibilities with respect to the planning of tests and/or calibrations and evaluation of results;

— the responsibilities for reporting opinions and interpretations;

— the responsibilities with respect to method modification and development and validation of new methods;

— expertise and experience required;

— qualifications and training programmes;

— managerial duties.

5.2.5 The management shall authorize specific personnel to perform particular types of sampling, test and/or calibration, to issue test reports and calibration certificates, to give opinions and interpretations and to operate particular types of equipment. The laboratory shall maintain records of the relevant authorization(s), competence, educational and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

5.3 Accommodation and environmental conditions

5.3.1 Laboratory facilities for testing and/or calibration, including but not limited to energy sources, lighting and environmental conditions, shall be such as to facilitate correct performance of the tests and/or calibrations.

The laboratory shall ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. Particular care shall be taken when sampling and tests and/or calibrations are undertaken at sites other than a permanent laboratory facility. The technical requirements for accommodation and environmental conditions that can affect the results of tests and calibrations shall be documented.

5.3.2 The laboratory shall monitor, control and record environmental conditions as required by the relevant specifications, methods and procedures or where they influence the quality of the results. Due attention shall be paid, for example, to biological sterility, dust, electromagnetic disturbances, radiation, humidity, electrical supply, temperature, and sound and vibration levels, as appropriate to the technical activities concerned. Tests and calibrations shall be stopped when the environmental conditions jeopardize the results of the tests and/or calibrations.

5.3.3 There shall be effective separation between neighbouring areas in which there are incompatible activities. Measures shall be taken to prevent cross-contamination.

5.3.4 Access to and use of areas affecting the quality of the tests and/or calibrations shall be controlled. The laboratory shall determine the extent of control based on its particular circumstances.

5.3.5 Measures shall be taken to ensure good housekeeping in the laboratory. Special procedures shall be prepared where necessary.

5.4 Test and calibration methods and method validation

5.4.1 General

The laboratory shall use appropriate methods and procedures for all tests and/or calibrations within its scope. These include sampling, handling, transport, storage and preparation of items to be tested and/or calibrated, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of test and/or calibration data.

The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of items for testing and/or calibration, or both, where the absence of such instructions could jeopardize the results of tests and/or calibrations. All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be kept up to date and shall be made readily available to personnel (see 4.3). Deviation from test and calibration methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the customer.

NOTE International, regional or national standards or other recognized specifications that contain sufficient and concise information on how to perform the tests and/or calibrations do not need to be supplemented or rewritten as internal procedures if these standards are written in a way that they can be used as published by the operating staff in a laboratory. It may be necessary to provide additional documentation for optional steps in the method or additional details.

5.4.2 Selection of methods

The laboratory shall use test and/or calibration methods, including methods for sampling, which meet the needs of the customer and which are appropriate for the tests and/or calibrations it undertakes. Methods published in international, regional or national standards shall preferably be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

When the customer does not specify the method to be used, the laboratory shall select appropriate methods that have been published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment. Laboratory-developed methods or methods adopted by the laboratory may also be used if they are appropriate for the intended use and if they are validated. The customer shall be informed as to the method chosen. The laboratory shall confirm that it can properly operate standard methods before introducing the tests or calibrations. If the standard method changes, the confirmation shall be repeated.

The laboratory shall inform the customer when the method proposed by the customer is considered to be inappropriate or out of date.

5.4.3 Laboratory-developed methods

The introduction of test and calibration methods developed by the laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources.

Plans shall be updated as development proceeds and effective communication amongst all personnel involved shall be ensured.

5.4.4 Non-standard methods

When it is necessary to use methods not covered by standard methods, these shall be subject to agreement with the customer and shall include a clear specification of the customer's requirements and the purpose of the test and/or calibration. The method developed shall have been validated appropriately before use.

NOTE For new test and/or calibration methods, procedures should be developed prior to the tests and/or calibrations being performed and should contain at least the following information:

a) appropriate identification;

b) scope;

c) description of the type of item to be tested or calibrated;

d) parameters or quantities and ranges to be determined;

e) apparatus and equipment, including technical performance requirements;

f) reference standards and reference materials required;

g) environmental conditions required and any stabilization period needed;

h) description of the procedure, including

- affixing of identification marks, handling, transporting, storing and preparation of items,
- checks to be made before the work is started,
- checks that the equipment is working properly and, where required, calibration and adjustment of the equipment before each use,
- the method of recording the observations and results,
- any safety measures to be observed;

i) criteria and/or requirements for approval/rejection;

j) data to be recorded and method of analysis and presentation;

k) the uncertainty or the procedure for estimating uncertainty.

5.4.5 Validation of methods

5.4.5.1 Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

5.4.5.2 The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

NOTE 1 Validation may include procedures for sampling, handling and transportation.

NOTE 2 The techniques used for the determination of the performance of a method should be one of, or a combination of, the following:

— calibration using reference standards or reference materials;

— comparison of results achieved with other methods;

— interlaboratory comparisons;

— systematic assessment of the factors influencing the result;

— assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.

NOTE 3 When some changes are made in the validated non-standard methods, the influence of such changes should be documented and, if appropriate, a new validation should be carried out.

5.4.5.3 The range and accuracy of the values obtainable from validated methods (e.g. the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the customers' needs.

NOTE 1 Validation includes specification of the requirements, determination of the characteristics of the methods, a check that the requirements can be fulfilled by using the method, and a statement on the validity.

NOTE 2 As method-development proceeds, regular review should be carried out to verify that the needs of the customer are still being fulfilled. Any change in requirements requiring modifications to the development plan should be approved and authorized.

NOTE 3 Validation is always a balance between costs, risks and technical possibilities. There are many cases in which the range and uncertainty of the values (e.g. accuracy, detection limit, selectivity, linearity, repeatability, reproducibility, robustness and cross-sensitivity) can only be given in a simplified way due to lack of information.

5.4.6 Estimation of uncertainty of measurement

5.4.6.1 A calibration laboratory, or a testing laboratory performing its own calibrations, shall have and shall apply a procedure to estimate the uncertainty of measurement for all calibrations and types of calibrations.

5.4.6.2 Testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

NOTE 1 The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

— the requirements of the test method;

— the requirements of the customer;

— the existence of narrow limits on which decisions on conformity to a specification are based.

NOTE 2 In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 5.10).

5.4.6.3 When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

NOTE 1 Sources contributing to the uncertainty include, but are not necessarily limited to, the reference standards and reference materials used, methods and equipment used, environmental conditions, properties and condition of the item being tested or calibrated, and the operator.

NOTE 2 The predicted long-term behaviour of the tested and/or calibrated item is not normally taken into account when estimating the measurement uncertainty.

NOTE 3 For further information, see ISO 5725 and the Guide to the Expression of Uncertainty in Measurement (see Bibliography).

5.4.7 Control of data

5.4.7.1 Calculations and data transfers shall be subject to appropriate checks in a systematic manner.

5.4.7.2 When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of test or calibration data, the laboratory shall ensure that:

a) computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;

b) procedures are established and implemented for protecting the data; such procedures shall include, but not be limited to, integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;

c) computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test and calibration data.

NOTE Commercial off-the-shelf software (e.g. wordprocessing, database and statistical programmes) in general use within their designed application range may be considered to be sufficiently validated. However, laboratory software configuration/modifications should be validated as in 5.4.7.2 a).

5.5 Equipment

5.5.1 The laboratory shall be furnished with all items of sampling, measurement and test equipment required for the correct performance of the tests and/or calibrations (including sampling, preparation of test and/or calibration items, processing and analysis of test and/or calibration data). In those cases where the laboratory needs to use equipment outside its permanent control, it shall ensure that the requirements of this International Standard are met.

5.5.2 Equipment and its software used for testing, calibration and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the tests and/or calibrations concerned. Calibration programmes shall be established for key quantities or values of the instruments where these properties have a significant effect on the results. Before being placed into service, equipment (including that used for sampling) shall be calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications. It shall be checked and/or calibrated before use (see 5.6).

5.5.3 Equipment shall be operated by authorized personnel. Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) shall be readily available for use by the appropriate laboratory personnel.

5.5.4 Each item of equipment and its software used for testing and calibration and significant to the result shall, when practicable, be uniquely identified.

5.5.5 Records shall be maintained of each item of equipment and its software significant to the tests and/or calibrations performed. The records shall include at least the following:

a) the identity of the item of equipment and its software;

b) the manufacturer's name, type identification, and serial number or other unique identification;

c) checks that equipment complies with the specification (see 5.5.2);

d) the current location, where appropriate;

e) the manufacturer's instructions, if available, or reference to their location;

f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration;

g) the maintenance plan, where appropriate, and maintenance carried out to date;

h) any damage, malfunction, modification or repair to the equipment.

5.5.6 The laboratory shall have procedures for safe handling, transport, storage, use and planned maintenance of measuring equipment to ensure proper functioning and in order to prevent contamination or deterioration.

NOTE Additional procedures may be necessary when measuring equipment is used outside the permanent laboratory for tests, calibrations or sampling.

5.5.7 Equipment that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labelled or marked as being out of service until it has been repaired and shown by calibration or test to perform correctly. The laboratory shall examine the effect of the defect or departure from specified limits on previous tests and/or calibrations and shall institute the "Control of nonconforming work" procedure (see 4.9).

5.5.8 Whenever practicable, all equipment under the control of the laboratory and requiring calibration shall be labelled, coded or otherwise identified to indicate the status of calibration, including the date when last calibrated and the date or expiration criteria when recalibration is due.

5.5.9 When, for whatever reason, equipment goes outside the direct control of the laboratory, the laboratory shall ensure that the function and calibration status of the equipment are checked and shown to be satisfactory before the equipment is returned to service.

5.5.10 When intermediate checks are needed to maintain confidence in the calibration status of the equipment, these checks shall be carried out according to a defined procedure.

5.5.11 Where calibrations give rise to a set of correction factors, the laboratory shall have procedures to ensure that copies (e.g. in computer software) are correctly updated.

5.5.12 Test and calibration equipment, including both hardware and software, shall be safeguarded from adjustments which would invalidate the test and/or calibration results.

5.6 Measurement traceability

5.6.1 General

All equipment used for tests and/or calibrations, including equipment for subsidiary measurements (e.g. for environmental conditions) having a significant effect on the accuracy or validity of the result of the test, calibration or sampling shall be calibrated before being put into service. The laboratory shall have an established programme and procedure for the calibration of its equipment.

NOTE Such a programme should include a system for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform tests and calibrations.

5.6.2 Specific requirements

5.6.2.1 Calibration

5.6.2.1.1 For calibration laboratories, the programme for calibration of equipment shall be designed and operated so as to ensure that calibrations and measurements made by the laboratory are traceable to the International System of Units (SI) (*Système international d'unités*).

A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to SI units may be achieved by reference to national measurement standards. National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute. When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification (see also 5.10.4.2).

NOTE 1 Calibration laboratories fulfilling the requirements of this International Standard are considered to be competent. A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to this International Standard, for the calibration concerned, is sufficient evidence of traceability of the calibration data reported.

NOTE 2 Traceability to SI units of measurement may be achieved by reference to an appropriate primary standard (see VIM:1993, 6.4) or by reference to a natural constant, the value of which in terms of the relevant SI unit is known and recommended by the General Conference of Weights and Measures (CGPM) and the International Committee for Weights and Measures (CIPM).

NOTE 3 Calibration laboratories that maintain their own primary standard or representation of SI units based on fundamental physical constants can claim traceability to the SI system only after these standards have been compared, directly or indirectly, with other similar standards of a national metrology institute.

NOTE 4 The term "identified metrological specification" means that it must be clear from the calibration certificate which specification the measurements have been compared with, by including the specification or by giving an unambiguous reference to the specification.

NOTE 5 When the terms "international standard" or "national standard" are used in connection with traceability, it is assumed that these standards fulfil the properties of primary standards for the realization of SI units.

NOTE 6 Traceability to national measurement standards does not necessarily require the use of the national metrology institute of the country in which the laboratory is located.

NOTE 7 If a calibration laboratory wishes or needs to obtain traceability from a national metrology institute other than in its own country, this laboratory should select a national metrology institute that actively participates in the activities of BIPM either directly or through regional groups.

NOTE 8 The unbroken chain of calibrations or comparisons may be achieved in several steps carried out by different laboratories that can demonstrate traceability.

5.6.2.1.2 There are certain calibrations that currently cannot be strictly made in SI units. In these cases calibration shall provide confidence in measurements by establishing traceability to appropriate measurement standards such as:

— the use of certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material;

— the use of specified methods and/or consensus standards that are clearly described and agreed by all parties concerned.

Participation in a suitable programme of interlaboratory comparisons is required where possible.

5.6.2.2 Testing

5.6.2.2.1 For testing laboratories, the requirements given in 5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

NOTE The extent to which the requirements in 5.6.2.1 should be followed depends on the relative contribution of the calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements should be strictly followed.

5.6.2.2.2 Where traceability of measurements to SI units is not possible and/or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required as for calibration laboratories (see 5.6.2.1.2).

5.6.3 Reference standards and reference materials

5.6.3.1 Reference standards

The laboratory shall have a programme and procedure for the calibration of its reference standards. Reference standards shall be calibrated by a body that can provide traceability as described in 5.6.2.1. Such reference standards of measurement held by the laboratory shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated. Reference standards shall be calibrated before and after any adjustment.

5.6.3.2 Reference materials

Reference materials shall, where possible, be traceable to SI units of measurement, or to certified reference materials. Internal reference materials shall be checked as far as is technically and economically practicable.

5.6.3.3 Intermediate checks

Checks needed to maintain confidence in the calibration status of reference, primary, transfer or working standards and reference materials shall be carried out according to defined procedures and schedules.

5.6.3.4 Transport and storage

The laboratory shall have procedures for safe handling, transport, storage and use of reference standards and reference materials in order to prevent contamination or deterioration and in order to protect their integrity.

NOTE Additional procedures may be necessary when reference standards and reference materials are used outside the permanent laboratory for tests, calibrations or sampling.

5.7 Sampling

5.7.1 The laboratory shall have a sampling plan and procedures for sampling when it carries out sampling of substances, materials or products for subsequent testing or calibration. The sampling plan as well as the sampling procedure shall be available at the location where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods. The sampling process shall address the factors to be controlled to ensure the validity of the test and calibration results.

NOTE 1 Sampling is a defined procedure whereby a part of a substance, material or product is taken to provide for testing or calibration of a representative sample of the whole. Sampling may also be required by the appropriate specification for which the substance, material or product is to be tested or calibrated. In certain cases (e.g. forensic analysis), the sample may not be representative but is determined by availability.

NOTE 2 Sampling procedures should describe the selection, sampling plan, withdrawal and preparation of a sample or samples from a substance, material or product to yield the required information.

5.7.2 Where the customer requires deviations, additions or exclusions from the documented sampling procedure, these shall be recorded in detail with the appropriate sampling data and shall be included in all documents containing test and/or calibration results, and shall be communicated to the appropriate personnel.

5.7.3 The laboratory shall have procedures for recording relevant data and operations relating to sampling that forms part of the testing or calibration that is undertaken. These records shall include the sampling procedure used, the identification of the sampler, environmental conditions (if relevant) and diagrams or other equivalent means to identify the sampling location as necessary and, if appropriate, the statistics the sampling procedures are based upon.

5.8 Handling of test and calibration items

5.8.1 The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention and/or disposal of test and/or calibration items, including all provisions necessary to protect the integrity of the test or calibration item, and to protect the interests of the laboratory and the customer.

5.8.2 The laboratory shall have a system for identifying test and/or calibration items. The identification shall be retained throughout the life of the item in the laboratory. The system shall be designed and operated so as to ensure that items cannot be confused physically or when referred to in records or other documents. The system shall, if appropriate, accommodate a sub-division of groups of items and the transfer of items within and from the laboratory.

5.8.3 Upon receipt of the test or calibration item, abnormalities or departures from normal or specified conditions, as described in the test or calibration method, shall be recorded. When there is doubt as to the suitability of an item for test or calibration, or when an item does not conform to the description provided, or the test or calibration required is not specified in sufficient detail, the laboratory shall consult the customer for further instructions before proceeding and shall record the discussion.

5.8.4 The laboratory shall have procedures and appropriate facilities for avoiding deterioration, loss or damage to the test or calibration item during storage, handling and preparation. Handling instructions provided with the item shall be followed. When items have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded. Where a test or calibration item or a portion of an item is to be held secure, the laboratory shall have arrangements for storage and security that protect the condition and integrity of the secured items or portions concerned.

NOTE 1 Where test items are to be returned into service after testing, special care is required to ensure that they are not damaged or injured during the handling, testing or storing/waiting processes.

NOTE 2 A sampling procedure and information on storage and transport of samples, including information on sampling factors influencing the test or calibration result, should be provided to those responsible for taking and transporting the samples.

NOTE 3 Reasons for keeping a test or calibration item secure can be for reasons of record, safety or value, or to enable complementary tests and/or calibrations to be performed later.

5.9 Assuring the quality of test and calibration results

5.9.1 The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or internal quality control using secondary reference materials;

b) participation in interlaboratory comparison or proficiency-testing programmes;

c) replicate tests or calibrations using the same or different methods;

d) retesting or recalibration of retained items;

e) correlation of results for different characteristics of an item.

NOTE The selected methods should be appropriate for the type and volume of the work undertaken.

5.9.2 Quality control data shall be analysed and, where they are found to be outside pre-defined criteria, planned action shall be taken to correct the problem and to prevent incorrect results from being reported.

5.10 Reporting the results

5.10.1 General

The results of each test, calibration, or series of tests or calibrations carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test or calibration methods.

The results shall be reported, usually in a test report or a calibration certificate (see Note 1), and shall include all the information requested by the customer and necessary for the interpretation of the test or calibration results and all information required by the method used. This information is normally that required by 5.10.2, and 5.10.3 or 5.10.4.

In the case of tests or calibrations performed for internal customers, or in the case of a written agreement with the customer, the results may be reported in a simplified way. Any information listed in 5.10.2 to 5.10.4 which is not reported to the customer shall be readily available in the laboratory which carried out the tests and/or calibrations.

NOTE 1 Test reports and calibration certificates are sometimes called test certificates and calibration reports, respectively.

NOTE 2 The test reports or calibration certificates may be issued as hard copy or by electronic data transfer provided that the requirements of this International Standard are met.

5.10.2 Test reports and calibration certificates

Each test report or calibration certificate shall include at least the following information, unless the laboratory has valid reasons for not doing so:

a) a title (e.g. "Test Report" or "Calibration Certificate");

b) the name and address of the laboratory, and the location where the tests and/or calibrations were carried out, if different from the address of the laboratory;

c) unique identification of the test report or calibration certificate (such as the serial number), and on each page an identification in order to ensure that the page is recognized as a part of the test report or calibration certificate, and a clear identification of the end of the test report or calibration certificate;

d) the name and address of the customer;

e) identification of the method used;

f) a description of, the condition of, and unambiguous identification of the item(s) tested or calibrated;

g) the date of receipt of the test or calibration item(s) where this is critical to the validity and application of the results, and the date(s) of performance of the test or calibration;

h) reference to the sampling plan and procedures used by the laboratory or other bodies where these are relevant to the validity or application of the results;

i) the test or calibration results with, where appropriate, the units of measurement;

j) the name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the test report or calibration certificate;

k) where relevant, a statement to the effect that the results relate only to the items tested or calibrated.

NOTE 1 Hard copies of test reports and calibration certificates should also include the page number and total number of pages.

NOTE 2 It is recommended that laboratories include a statement specifying that the test report or calibration certificate shall not be reproduced except in full, without written approval of the laboratory.

5.10.3 Test reports

5.10.3.1 In addition to the requirements listed in 5.10.2, test reports shall, where necessary for the interpretation of the test results, include the following:

a) deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;

b) where relevant, a statement of compliance/non-compliance with requirements and/or specifications;

c) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

d) where appropriate and needed, opinions and interpretations (see 5.10.5);

e) additional information which may be required by specific methods, customers or groups of customers.

5.10.3.2 In addition to the requirements listed in 5.10.2 and 5.10.3.1, test reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

a) the date of sampling;

b) unambiguous identification of the substance, material or product sampled (including the name of the manufacturer, the model or type of designation and serial numbers as appropriate);

c) the location of sampling, including any diagrams, sketches or photographs;

d) a reference to the sampling plan and procedures used;

e) details of any environmental conditions during sampling that may affect the interpretation of the test results;

f) any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

5.10.4 Calibration certificates

5.10.4.1 In addition to the requirements listed in 5.10.2, calibration certificates shall include the following, where necessary for the interpretation of calibration results:

a) the conditions (e.g. environmental) under which the calibrations were made that have an influence on the measurement results;

b) the uncertainty of measurement and/or a statement of compliance with an identified metrological specification or clauses thereof;

c) evidence that the measurements are traceable (see Note 2 in 5.6.2.1.1).

5.10.4.2 The calibration certificate shall relate only to quantities and the results of functional tests. If a statement of compliance with a specification is made, this shall identify which clauses of the specification are met or not met.

When a statement of compliance with a specification is made omitting the measurement results and associated uncertainties, the laboratory shall record those results and maintain them for possible future reference.

When statements of compliance are made, the uncertainty of measurement shall be taken into account.

5.10.4.3 When an instrument for calibration has been adjusted or repaired, the calibration results before and after adjustment or repair, if available, shall be reported.

5.10.4.4 A calibration certificate (or calibration label) shall not contain any recommendation on the calibration interval except where this has been agreed with the customer. This requirement may be superseded by legal regulations.

5.10.5 Opinions and interpretations

When opinions and interpretations are included, the laboratory shall document the basis upon which the opinions and interpretations have been made. Opinions and interpretations shall be clearly marked as such in a test report.

NOTE 1 Opinions and interpretations should not be confused with inspections and product certifications as intended in ISO/IEC 17020 and ISO/IEC Guide 65.

NOTE 2 Opinions and interpretations included in a test report may comprise, but not be limited to, the following:

— an opinion on the statement of compliance/noncompliance of the results with requirements;

— fulfilment of contractual requirements;

— recommendations on how to use the results;

— guidance to be used for improvements.

NOTE 3 In many cases it might be appropriate to communicate the opinions and interpretations by direct dialogue with the customer. Such dialogue should be written down.

5.10.6 Testing and calibration results obtained from subcontractors

When the test report contains results of tests performed by subcontractors, these results shall be clearly identified. The subcontractor shall report the results in writing or electronically.

When a calibration has been subcontracted, the laboratory performing the work shall issue the calibration certificate to the contracting laboratory.

5.10.7 Electronic transmission of results

In the case of transmission of test or calibration results by telephone, telex, facsimile or other electronic or electromagnetic means, the requirements of this International Standard shall be met (see also 5.4.7).

5.10.8 Format of reports and certificates

The format shall be designed to accommodate each type of test or calibration carried out and to minimize the possibility of misunderstanding or misuse.

NOTE 1 Attention should be given to the lay-out of the test report or calibration certificate, especially with regard to the presentation of the test or calibration data and ease of assimilation by the reader.

NOTE 2 The headings should be standardized as far as possible.

5.10.9 Amendments to test reports and calibration certificates

Material amendments to a test report or calibration certificate after issue shall be made only in the form of a further document, or data transfer, which includes the statement:

"Supplement to Test Report [or Calibration Certificate], serial number... [or as otherwise identified]",

or an equivalent form of wording.

Such amendments shall meet all the requirements of this International Standard.

When it is necessary to issue a complete new test report or calibration certificate, this shall be uniquely identified and shall contain a reference to the original that it replaces.

Annex A
(informative)

Nominal cross-references to ISO 9001:2000

Table A.1 — Nominal cross-references to ISO 9001:2000

ISO 9001:2000	ISO/IEC 17025
Clause 1	Clause 1
Clause 2	Clause 2
Clause 3	Clause 3
4.1	4.1, 4.1.1, 4.1.2, 4.1.3, 4.1.4, 4.1.5, 4.2, 4.2.1, 4.2.2, 4.2.3, 4.2.4
4.2 1	4.2.2, 4.2.3, 4.3.1
4.2.2	4.2.2, 4.2.3, 4.2.4
4.2.3	4.3
4.2.4	4.3.1, 4.12
5.1	4.2.2, 4.2.3
5.1 a)	4.1.2, 4.1.6
5.1 b)	4.2.2
5.1 c)	4.2.2
5.1 d)	4.15
5.1 e)	4.1.5
5.2	4.4.1
5.3	4.2.2
5.3 a)	4.2.2
5.3 b)	4.2.3
5.3 c)	4.2.2
5.3 d)	4.2.2
5.3 e)	4.2.2
5.4.1	4.2.2 c)
5.4.2	4.2.1
5.4.2 a)	4.2.1
5.4.2 b)	4.2.1
5.5.1	4.1.5 a), f), h)
5.5.2	4.1.5 i)
5.5.2 a)	4.1.5 i)
5.5.2 b)	4.11.1
5.5.2 c)	4.2.4
5.5.3	4.1.6
5.6.1	4.15
5.6.2	4.15
5.6.3	4.15

ISO 9001:2000	ISO/IEC 17025
6.1 a)	4.10
6.1 b)	4.4.1, 4.7, 5.4.2, 5.4.3, 5.4.4, 5.10.1
6.2.1	5.2.1
6.2.2 a)	5.2.2, 5.5.3
6.2.2 b)	5.2.1, 5.2.2
6.2.2 c)	5.2.2
6.2.2 d)	4.1.5 k)
6.2.2 e)	5.2.5
6.3.1 a)	4.1.3, 4.12.1.2, 4.12.1.3, 5.3
6.3.1 b)	4.12.1.4, 5.4.7.2, 5.5, 5.6
6.3.1 c)	4.6, 5.5.6, 5.6.3.4, 5.8, 5.10
6.4	5.3.1, 5.3.2, 5.3.3, 5.3.4, 5.3.5
7.1	5.1
7.1 a)	4.2.2
7.1 b)	4.1.5 a), 4.2.1, 4.2.3
7.1 c)	5.4, 5.9
7.1 d)	4.1, 5.4, 5.9
7.2.1	4.4.1, 4.4.2, 4.4.3, 4.4.4, 4.4.5, 5.4, 5.9, 5.10
7.2.2	4.4.1, 4.4.2, 4.4.3, 4.4.4, 4.4.5, 5.4, 5.9, 5.10
7.2.3	4.4.2, 4.4.4, 4.5, 4.7, 4.8
7.3	5, 5.4, 5.9
7.4.1	4.6.1, 4.6.2, 4.6.4
7.4.2	4.6.3
7.4.3	4.6.2
7.5.1	5.1, 5.2, 5.4, 5.5, 5.6, 5.7, 5.8, 5.9
7.5.2	5.2.5, 5.4.2, 5.4.5
7.5.3	5.8.2
7.5.4	4.1.5 c), 5.8
7.5.5	4.6.1, 4.12, 5.8, 5.10
7.6	5.4, 5.5
8.1	4.10, 5.4, 5.9
8.2.1	4.10
8.2.2	4.11.5, 4.14
8.2.3	4.11.5, 4.14, 5.9
8.2.4	4.5, 4.6, 4.9, 5.5.2, 5.5.9, 5.8, 5.8.3, 5.8.4, 5.9
8.3	4.9
8.4	4.10, 5.9
8.5.1	4.10, 4.12
8.5.2	4.11, 4.12
8.5.3	4.9, 4.11, 4.12

ISO/IEC 17025 covers several technical competence requirements that are not covered by ISO 9001:2000.

Annex B
(informative)

Guidelines for establishing applications for specific fields

B.1 The requirements specified in this International Standard are stated in general terms and, while they are applicable to all test and calibration laboratories, explanations might be needed. Such explanations on applications are herein referred to as applications. Applications should not include additional general requirements not included in this International Standard.

B.2 Applications can be thought of as an elaboration of the generally stated criteria (requirements) of this International Standard for specified fields of test and calibration, test technologies, products, materials or specific tests or calibrations. Accordingly, applications should be established by persons having appropriate technical knowledge and experience, and should address items that are essential or most important for the proper conduct of a test or calibration.

B.3 Depending on the application at hand, it may be necessary to establish applications for the technical requirements of this International Standard. Establishing applications may be accomplished by simply providing detail or adding extra information to the already generally stated requirements in each of the clauses (e.g. specific limitations to the temperature and humidity in the laboratory).

In some cases the applications will be quite limited, applying only to a given test or calibration method or to a group of calibration or test methods. In other cases the applications may be quite broad, applying to the testing or calibration of various products or items or to entire fields of testing or calibration.

B.4 If the applications apply to a group of test or calibration methods in an entire technical field, common wording should be used for all of the methods.

Alternatively, it may be necessary to develop a separate document of applications to supplement this International Standard for specific types or groups of tests or calibrations, products, materials or technical fields of tests or calibrations. Such a document should provide only the necessary supplementary information, while maintaining this International Standard as the governing document through reference. Applications which are too specific should be avoided in order to limit the proliferation of detailed documents.

B.5 The guidance in this annex should be used by accreditation bodies and other types of evaluation bodies when they develop applications for their own purposes (e.g. accreditation in specific areas).

Bibliography

[1] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1 General principles and definitions*

[2] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[3] ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

[4] ISO 5725-4, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

[5] ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

[6] ISO 9000:—[1)], *Quality management systems — Fundamentals and vocabulary*

[7] ISO 9001:2000, *Quality management systems — Requirements*

[8] ISO/IEC 90003, *Software engineering — Guidelines for the application of ISO 9001:2000 to computer software*

[9] ISO 10012:2003, *Measurement management systems — Requirements for measurement processes and measuring equipment*

[10] ISO/IEC 17011, *Conformity assessment — General requirements for accreditation bodies accrediting conformity assessment bodies*

[11] ISO/IEC 17020, *General criteria for the operation of various types of bodies performing inspection*

[12] ISO 19011, *Guidelines for quality and/or environmental management systems auditing*

[13] ISO Guide 30, *Terms and definitions used in connection with reference materials*

[14] ISO Guide 31, *Reference materials — Contents of certificates and labels*

[15] ISO Guide 32, *Calibration in analytical chemistry and use of certified reference materials*

[16] ISO Guide 33, *Uses of certified reference materials*

[17] ISO Guide 34, *General requirements for the competence of reference material producers*

[18] ISO Guide 35, *Certification of reference materials — General and statistical principles*

[19] ISO/IEC Guide 43-1, *Proficiency testing by interlaboratory comparisons — Part 1: Development and operation of proficiency testing schemes*

[20] ISO/IEC Guide 43-2, *Proficiency testing by interlaboratory comparisons — Part 2: Selection and use of proficiency testing schemes by laboratory accreditation bodies*

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[21] ISO/IEC Guide 58:1993, *Calibration and testing laboratory accreditation systems — General requirements for operation and recognition*

[22] ISO/IEC Guide 65, *General requirements for bodies operating product certification systems*

[23] GUM, *Guide to the Expression of Uncertainty in Measurement*, issued by BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML

[24] Information and documents on laboratory accreditation can be found on the ILAC (International Laboratory Accreditation Cooperation): www.ilac.org

ICS 03.120.20

Price based on 28 pages

Designation: E 882 – 87 (Reapproved 2003)

Standard Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[1]

This standard is issued under the fixed designation E 882; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the essential aspects of an accountability and quality control program for a chemical analysis laboratory. The reasons for establishing and operating such a program are discussed.

2. Referenced Documents

2.1 *ASTM Standards:*

MNL 7 Manual on Presentation of Data and Control Chart Analysis[2]

2.2 *ANSI Document:*

ANSI/ASQC A1 Definitions, Symbols, Formulas, and Tables for Control Charts[3]

3. Significance and Use

3.1 An accountability and quality control system is established by laboratory management to improve the quality of its results. It provides documented records which serve to assure users of the laboratory's services that a specified level of precision is achieved in the routine performance of its measurements and that the data reported were obtained from the samples submitted. The system also provides for: early warning to analysts when methods or equipment begin to develop a bias or show deterioration of precision; the protection and retrievability of data (results); traceability and control of samples as they are processed through the laboratory; good communication of sample information between submitters, analysts, and supervision; and information on sample processing history. This guide describes such a system. Other accountability and quality control programs can be developed. Such programs can be equivalent to the program in this guide if they provide all of the benefits mentioned above.

4. Accountability

4.1 Accountability means assurance that the results reported refer directly to the samples submitted.

4.2 Prior to submitting samples to the laboratory, the prospective user should consult with laboratory personnel concerning his needs and the capability of the laboratory to satisfy them. It is the responsibility of the originator of the samples to select and identify proper samples for submission to the laboratory, to decide what information is required (especially, to define the use to be made of the information), and, after consulting with laboratory personnel, to submit the samples in suitable containers, properly labeled, and accompanied by written instructions identifying the samples, their nature, and the information sought through chemical analysis. This should be done formally, using a well-defined document for information transfer to initiate work in the laboratory.

4.3 Laboratory management establishes a written accountability system to be used throughout the laboratory at all times. This implies traceability and documentation of all reported results through the laboratory back to the submitted sample. This system should have the following general characteristics:

4.3.1 Each nonroutine job submitted by a user of the laboratory's services is assigned an internal laboratory identification number (ID), which is used to correlate all samples, work, time and cost accounting, consultation, and reports and other paperwork associated with that job. The final report that is returned to the originator will always bear the number (ID) for future reference. Moreover, it is convenient for laboratory data to be filed according to sequential ID numbers. For example, "86/0428" might identify the associated work as the 428th request submitted in the year 1986. The *Data Record* should provide all data generated during the analyses, names of persons performing the analyses, dates the analyses were performed, and any unusual occurrences that happened during the analyses. Accountability for production control samples is normally maintained separately from the nonroutine records because results from production control samples are usually reported on routine report forms, the samples being identified with the day, shift, run, or lot from which they were taken.

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[1] This guide is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.22 on Statistics and Quality Control.

Current edition approved Oct. 1, 2003. Published November 2003. Originally approved in 1982. Last previous edition approved in 1998 as E 882 – 98.

[2] ASTM Manual Series, ASTM, 6th Edition, 1990.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

4.3.2 Each sample, specimen, sample site, or other unique piece of material or container identified as a separate sample by the originator should be assigned a sequential item number (NN) for internal laboratory use. As soon as the samples are accepted by the laboratory, laboratory personnel will mark each sample or sample container with its own laboratory sample number (ID-NN) in such manner that the label is not likely to become separated from its sample or rendered unreadable during its residence in the laboratory. For example, the fifth sample on the above-mentioned request might be identified as "86/0428-05."

4.3.3 All laboratory work records, intermediate sample containers, data, and reports for a specific sample will be identified by the same laboratory identification and item number to avoid any opportunity for samples or data to be lost or intermixed within or between jobs.

4.3.4 The first and last steps in the accountability procedure are functions of technical supervision. Before any work is performed, the compatibility of the work requested with the physical condition of the samples and the capabilities of the laboratory must be verified. When the analysts have completed their work, the results must be reviewed to be certain that all information requested has been determined and that the work has been performed with the required care and precision. In this latter regard, quality control procedures prove invaluable both to the analysts performing the work and the reviewing supervisor. The supervisor also verifies that the results are calculated in units that are most meaningful to the submitter and that the units and basis on which the results are calculated are clearly stated.

4.3.5 Except for the most routine work, the original analyst's data book, a serial listing of laboratory identification numbers and descriptions, and a copy of each job report sheet are retained in the laboratory's records for the periods of time established by laboratory policy. Intermediate calculations and samples are normally discarded after the submitter has had a reasonable opportunity to submit questions concerning the results and request return of his samples. In some cases, customer specifications may dictate the records that must be retained and the retention times for both analytical records and laboratory samples.

5. Quality Control

5.1 Quality control of analytical methods provides the information needed to ensure that procedures, equipment, and personnel are performing at the levels of precision and accuracy required by the intended use of the data.

5.2 *General Characteristics*—The following factors have been found helpful in maximizing the effectiveness and minimizing the cost of quality control procedures:

5.2.1 Involve the operators or analysts who actually perform the work to the greatest possible extent.

5.2.2 Use the simplest, most direct statistical procedures that will provide the necessary degree of control. This means that graphical or simplified arithmetic procedures are preferred.

5.2.3 Perform the quality control measurements as early in the measurement process as possible. This prevents waste of analytical effort if the method is not initially in control. However, when a prolonged series of measurements is made, it is also necessary to verify that the method remains in control throughout the run.

5.2.4 Provide specific action limits and describe exactly what must be done when these limits are exceeded.

5.2.5 For each method (for each sample type), choose a control material that is known to be stable and homogeneous and has measured values within the range of interest. Any inhomogeneity in the control sample will add to the variance of the results. Any increase in variability that is not related to the measurement process will reduce the sensitivity of the quality control procedure to detect changes in the measurement process. Where possible, the control material should be similar to the samples to be analyzed. Obtain as large an amount of control material as can be prepared in a homogeneous state because considerable effort is required to prepare a new control. Always prepare a new control material well in advance of exhausting the old one so that the new chart is ready when needed. In situations where satisfactory control material cannot be obtained, alternative techniques (such as, retest by a senior analyst) may be substituted for the control chart approach.

5.2.6 Give analysts specific instructions concerning their response to an out-of-control condition. Supervision may decide that, if the analyst can correct the problem so that the control sample results again plot within limits, the process may continue without immediate contact with the supervisor. In other situations, the supervisor may need to become involved with each out-of-control incident. In either case, adjustments to the process should be recorded to explain each shift in the control measurements.

5.2.7 Provide for a periodic in-depth review by supervision and management of the overall effectiveness of the laboratory quality control system. Operating experience may indicate that methods should be added to, or dropped from the program, that the frequency of specific control samples should be increased or decreased, or that a different strategy might be more appropriate for control of a specific method. The interval for such reviews should be determined by the uniformity of the processes that generate the samples. Any anticipated or observed change in the character of the samples being analyzed should initiate at least a cursory review of the control procedures for the methods that apply to those samples.

5.3 *Laboratory Quality Control Strategies*—Control chart methods are suitable for laboratory quality control programs. The choice of which control strategy to use depends on circumstances: the type of instrument or laboratory procedure, the number of samples and frequency of the analyses, and the closeness of control required. The following are appropriate:

5.3.1 The $\bar{X}$- and R-chart method is most frequently used. The control sample is run two or more times during the run, batch, or shift. The average is plotted on the $\bar{X}$-chart and the absolute value of the difference between the high and low values, the range, is plotted on the R-chart. If the average falls between the upper and lower control limits and the range falls below the upper control limit, the process is considered to be in control. Fig. 1 shows the essential features of charts for averages and ranges.

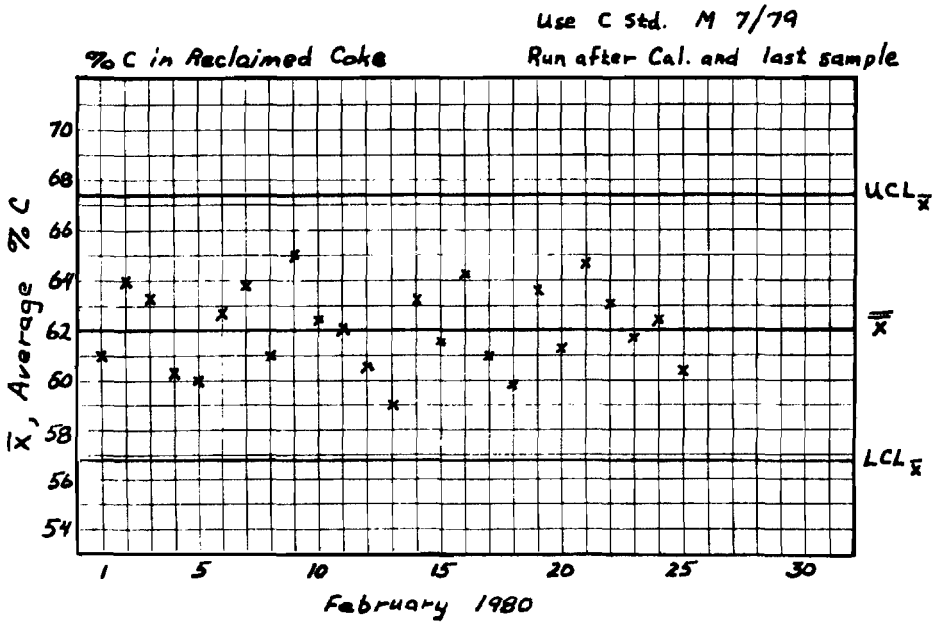

FIG. 1 Control Chart for Averages

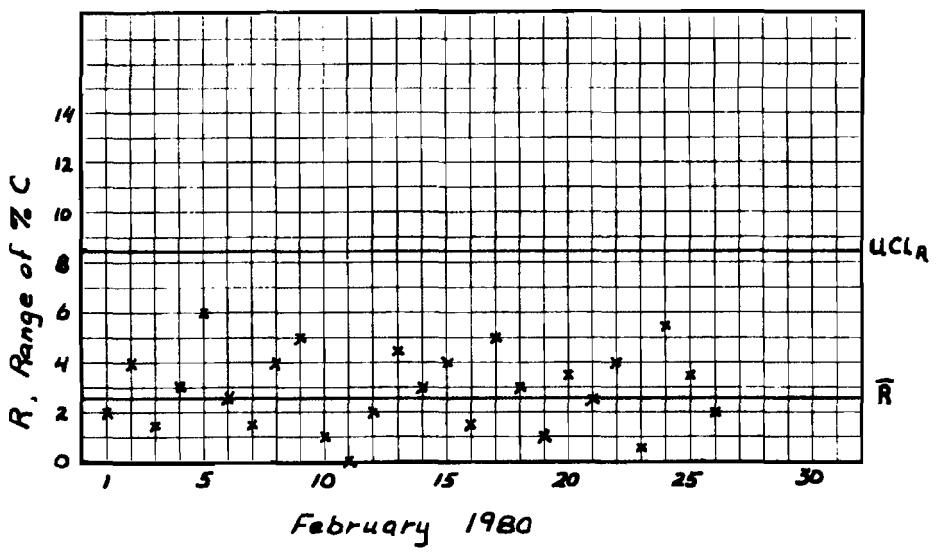

FIG. 2 Control Chart for Ranges

5.3.2 The *X*-chart method (often called the control chart for individuals) is useful for measurements that are made on a frequent or continual basis. It is appropriate for methods or instruments for which the usual mode of failure produces relatively large shifts in results and the cost of a determination precludes performing replicate analyses of control samples. Its main characteristic is that it responds rapidly to sudden relatively large changes in the analytical process, but it is not as sensitive to small changes as the $\bar{X}$- and *R*-chart method. Each time the control material is analyzed, its value is plotted on the *X*-chart. If the point plots between the upper and lower control limits, the analytical process is considered to be in control. Fig. 3 shows the essential features of charts for individuals.

5.3.3 A combination of the above two methods constitutes a useful strategy. A fixed number of control sample runs are made during a period that samples are being analyzed (such period could, for example, be a shift or a day in a continuous analysis process). Each individual value is plotted on the *X*-chart as the measurement is completed. Their average value and range are plotted on the $\bar{X}$- and *R*-charts. The additional effort to prepare and maintain both types of control charts may be justified in situations where erroneous assays would cause large economic losses. Other control chart techniques that may be appropriate for special circumstances may be found in the ANSI/ASQC document.

5.3.4 Comparison with standard reference materials (SRMs) is frequently the only strategy that can be employed for

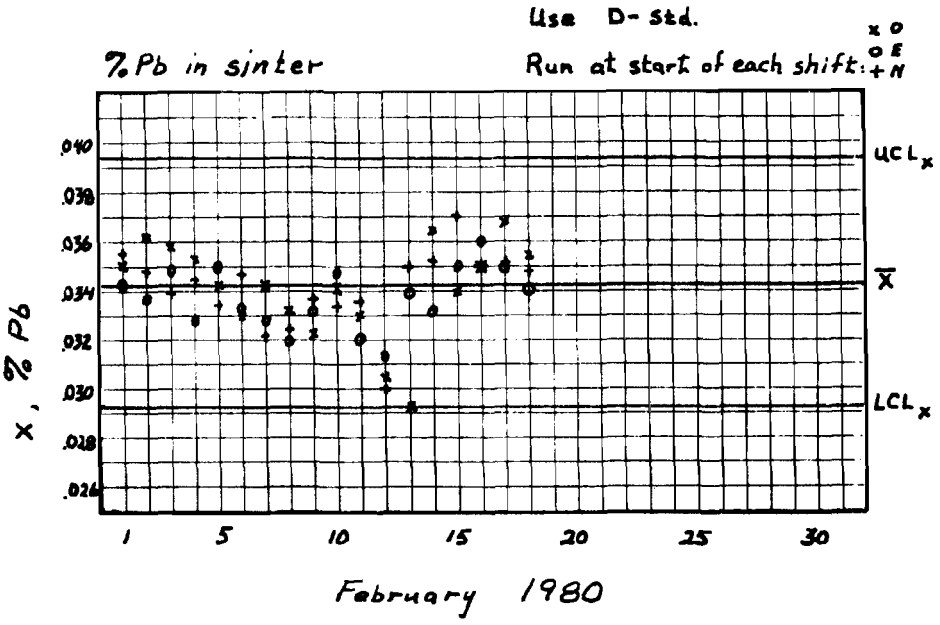

FIG. 3 Control Chart for Individuals

infrequently used analytical methods or for nonroutine sample types. If an SRM such as one from the National Institute of Standards and Technology (similar to the samples) is run along with the samples, comparison of the measured value against the known value of the standard provides a measure of confidence in the sample assays. Lacking an SRM, any previously analyzed material may be used. In all cases, it is important to retain as large a portion of such a material as possible and to tabulate the results, the method used, the date, and the analyst. Materials and data thus obtained may have important future statistical or control chart use.

5.4 *Definitions*:

5.4.1 *mean*:

$$\bar{X} = (X_1 + X_2 + \ldots . X_n)/(n) \quad (1)$$

where n = the number of analytical values.

5.4.2 *grand mean*:

$$\bar{\bar{X}} = (\bar{X}_1 + \bar{X}_2 + \ldots . \bar{X}_k)/(k) \quad (2)$$

where K = the number of individual means.

5.4.3 *range*:

$$R = X_h - X_l \quad (3)$$

where:

X_h = highest observed value, and

X_l = lowest observed value in the data being averaged.

5.4.4 *average range*:

$$\bar{R} = (R_1 + R_2 + \ldots R_k)/(k) \quad (4)$$

5.4.5 *estimated standard deviation*:

$$s = \sqrt{\frac{\Sigma(X_i - \bar{X})^2}{n-1}} \quad (5)$$

where:

x_i = the value of the n successive observations, and

Σ = the sum of the squares of the indicated differences.

5.5 *Control Chart Construction*—Calculate the central value and control limits. Prepare the control chart with the vertical scale labeled so that the central value is approximately midway on the graph. Select a scale factor to permit the results to be plotted accurately and easily. The horizontal axis is labeled by shift, day, run number, or run date, as appropriate. Draw and label the central line and the lines representing the upper and lower control limits. The central line may be the grand average of the values obtained during the base period described below, or it may be a specifed value based upon experience. Title the chart with the method, the control sample identification, and any special instructions. Show all of the original data points that were used to calculate the control limits.

5.5.1 *$\bar{X}$- and R-Chart*—At least two independent measurements must be made on the control material during each run. The average of the n values obtained on each run is plotted on the $\bar{X}$-chart and the range is plotted on the R-chart. To calculate the positions of the lines on the charts, obtain at least 20 groups of n values (that is, 20 runs) during a period when the method is believed to be performing normally.

Formulas for $\bar{X}$ and R Control Chart Lines

Number of Measurements per Run[A]	Chart for Averages, $\bar{X}$	
	Central Line	Control Limits
2	$\bar{\bar{X}}$	$\bar{\bar{X}} \pm 1.880\ \bar{R}$
3	$\bar{\bar{X}}$	$\bar{\bar{X}} \pm 1.023\ \bar{R}$
4	$\bar{\bar{X}}$	$\bar{\bar{X}} \pm 0.729\ \bar{R}$
	Chart for Ranges, R	
	Central Line	Upper Control Limit
2	$\bar{R}$	$\bar{R} + 3.267\ \bar{R}$
3	$\bar{R}$	$\bar{R} + 2.574\ \bar{R}$
4	$\bar{R}$	$\bar{R} + 2.282\ \bar{R}$

[A] See MNL 7 if more than four measurements are made per run.

5.5.2 *X-Chart*—Each individual result is plotted on the *X*-chart. To calculate the positions of the lines on the chart, obtain at least 20 values for the control material when the method is believed to be performing normally. These measurements should be taken at the same intervals to be used for control purposes.

Formula for *X* Control Chart Lines

Central Line	Control Limits
$\bar{X}$	$\bar{X} \pm 2.66\ \bar{R}$

where $\bar{R}$ (the moving range) is calculated as the average of the differences of each measurement from the preceding measurement, ignoring the sign. (Note that there will be one less such difference than the number of individual measurements.)

5.5.3 *Individuals Used as Grouped Data*—If the individual values used to produce the *X*-chart can be grouped in some logical way such that, *n* results are obtained each day, each shift, and so forth, then $\bar{X}$- and *R*-charts may be constructed by treating these grouped data by the formulas of 5.5.1. Each result is then plotted as it is produced on the *X*-chart for an immediate control decision. The average and range of the grouped data are plotted on the $\bar{X}$- and *R*-charts at the end of each period for detection of less abrupt drifts in the analytical procedure.

5.5.4 A complete discussion of control chart theory and practice (including examples of each procedure) is included in MNL 7.

6. Limitations of Control Chart Methods

6.1 In common with all statistical procedures, control chart methods depend upon certain basic assumptions. For the purposes of this guide, the most important of these assumptions is that the analytical method is capable of yielding consistent results in the hands of those who use it. If the performance of the method itself is erratic, or if the personnel who apply it do not exercise sufficient care, then no period of time will exist when the data collected can predict future behavior.

6.2 The limitation discussed above is not serious in the practical laboratory environment. The reason is obvious: If the method or the way it is employed is actually out of control (another way of saying erratic), that fact will become apparent because the calculated initial control limits will be unacceptably wide.

6.3 A second limitation of the control chart method is that it only indicates whether or not the analytical method is performing as expected. When it is not, the control chart gives no direct indication of the reasons for the poor performance. An analytical chemist must exercise his chemical skills and ingenuity to discover and correct the causes of the problem. The control chart will, however, provide analysts with quantitative indication of the effectiveness of their efforts.

7. Procedures for Improving Precisions or Accuracy of Analytical Results

7.1 Although complete coverage of the topic of improving the precision of analytical methods is beyond the scope of this guide, several aspects of statistical or supervisory control have the effect of improving precision or accuracy of analytical results without changes in the analytical methods.

7.1.1 Replication—It is a simple statistical fact that averaging a larger number of independent measurements improves the precision of the average reported values. In general, for readings randomly obtained from a normally distributed population of readings, the expected standard deviation of the average of *n* readings is just $\frac{1}{\sqrt{n}}$ times the standard deviation of a single reading. A second important fact is that the first few replications improve the precision much more than would the same number of additional readings beyond, say 30. The improvement from averaging four observations is to reduce the standard deviation of the reported result by one half; nine replicates reduces it by two thirds, and so forth. The nature of chemical analyses is such that we must be careful when we seek to improve results in this manner. First, it is generally not difficult to justify making two or even four replicate observations for an important measurement. It is rarely justified to perform the same assay more than nine times. The second consideration is even more important. Truly independent replications are obtained only by replication of the *entire* analytical process. Replication of less than the entire analytical process (which includes sampling and sample preparation) usually produces much less reduction in the error of the final reported value than would be expected from the square root law. By way of illustration, the average of two separate samples taken from a rail car containing 100 tons of ore will probably be closer to the true content of that car than the average of two successive readings of an atomic absorption spectrometer while it is aspirating the solution obtained from one of those samples. Yet, because sampling variability is not included, the observed precision of the latter seems to be the better of the two.

7.1.2 *Improvements in Instruments and Personnel*—Proper maintenance of equipment with periodic checks on its performance against standards is necessary to minimize the contribution of instrumental errors. Proper training of personnel in good techniques and in understanding the significance of each step performed is necessary if a laboratory is to approach the accuracy that methods and equipment are capable of delivering. The attitude of pride in good work and confidence in one's ability to perform well can be increased by involving all laboratory personnel in the planning and implementation of a quality control program. After all, the analyst will be the first one to see how his own performance looks on the control chart.

7.1.3 *Standards*—This last factor is included because it is crucial to accurate and meaningful laboratory results. A method or an instrument can be no more accurate than the standards with which it is calibrated. Not only must the standards be correct for the method and characterized with the accuracy demanded by the ultimate analysis to be performed, but the standardization process must be performed in such a manner

that this accuracy is transferred to the instrument or solution being calibrated. More care needs to be taken in the calibration process than for any other single measurement. In general, each calibration observation is subject to the same measurement error as each sample observation. Thus, it makes no sense to draw a calibration curve from single observations on standards and then to measure each sample four times. The random error for the calibration measurement would appear as a perceptible bias in all sample measurements. No general rule can be given for the proper emphasis to be placed on calibration. The guide to proper effort in this area is to combine common sense with an awareness that each calibration measurement is usually no more precise than any other measurement made with the same equipment.

8. Keywords

8.1 accountability; chemical analysis; control charts; quality control

Principles of Method Validation and Measurement Uncertainty in Chemical Analysis

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UNCERTAINTY

Uncertainty is an estimate attached to a test result which characterises the range of values within which the true value is asserted to lie (EurochemCITAC, QUAM 2000). Uncertainty of a measurement is a parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand. In other words, uncertainty is an expression of dispersion of measurement results and it can not be removed.

Uncertainty of measurement comprises many components. In general there are 2 types of uncertainty:

Type A: Those can be evaluated from the statistical distribution of the results of series of measurements and can be characterised by standard deviations. (Uncertainties from Validation Parameters)

Type B: Those can be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information. (Uncertainties from calibration certificates, certificates of reference materials used etc.)

VALIDATION

Validation is the confirmation by examination and the provision of objective evidence that particular requirements for a specific intended use are fulfilled (ISO/IEC/17025). In other words, validation is systematical test/measurements and statistical evaluation of a method to determine the performance of it.

Every laboratory has to validate their methods to provide consistency in their application of methods, comparability between their laboratories and other laboratories and to be suitable for the legal requirements. Besides, we ethicaly need validation to establish fitness-for-purpose on customer's behalf and to make good science, commercialy to provide "due care" in product liability/responsibility.

Laboratores have to validate their methods to verify their own ability to match published data in their own laboratory conditions. There are also several reasons to validate a method:

- If you develop a method
- If you change of application / working environment / analysts
- If you notice changes in your results during your Quality Control (QC) checks
- To show the similarities between two methods
- If you standardize a method

Validation Stages

There are several stages to validate a method. Before starting a validation, you have to define your analytical requirements and you develop or identify a candidate method. Then you plan a validation experiment. Your validation plan has to cover sample preparation and sampling. After you make the plan, you carry out the experiments. After you finish the experiments you have to assess the results in terms of fitness-for-purpose. Then you check the results if they met with analytical requirements. If everything is suitable, then you prepare the validation report. After statement of fitness-for-purpose, your method is validated. The validation stages are summarized in **Figure 1.**

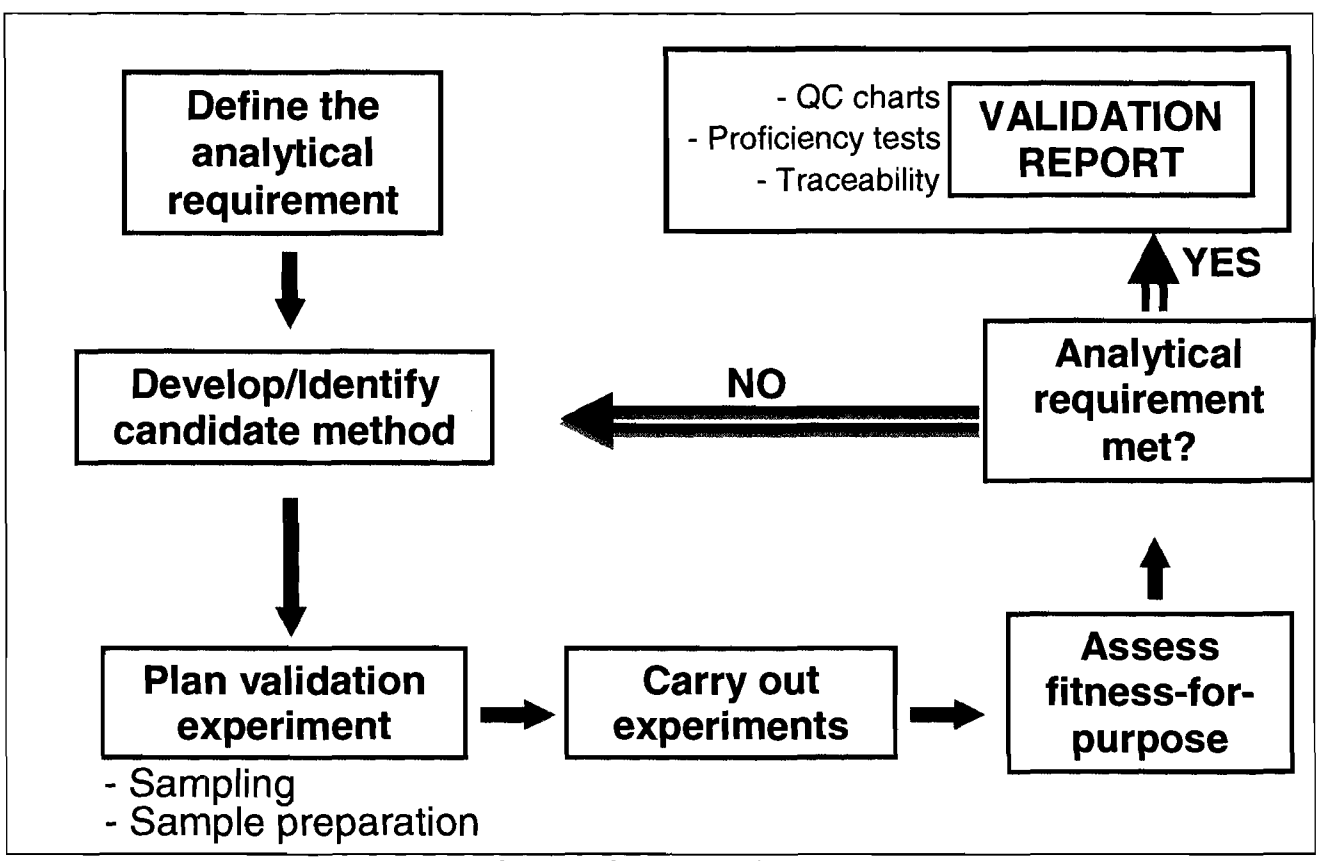


Figure 1. Validation Stages

Validation Parameters (Performance parameters) of a method

The validation parameters are the evidences to show that the results produced by the method are fit-for-purpose. Validation studies for quantitative analytical methods typically determine some or all of the following parameters:

- Bias, recovery
  - These parameters show that how much the results are close to the "right" answer
- Precision (repeatibility, reproducibility)
  - These parameter shows that how much the results of replicate measurements made on the same sample are close to each other.
- Working & Linear Range (Limit of Detection (LOD), Limit of Quantification (LOQ), Linearity)
- Specificity/ Selectivity
  - These parameters are calculated to show if there are any interferences in the method.
- Robustness/Ruggedness
  - These are the parameters to control necessaries for each stage of the procedure

Bias, recovery

The bias of an analytical method is usually determined by study of relevant reference materials or by spiking studies. Bias is difference between mean value of analytical results and referance value (Figure 2). The determination of overall bias with respect to appropriate reference values is important in establishing traceability to recognised standards. Bias may be expressed as analytical recovery (value observed divided by value expected). Bias should be shown to be negligible or corrected. Bias is a measure of trueness.

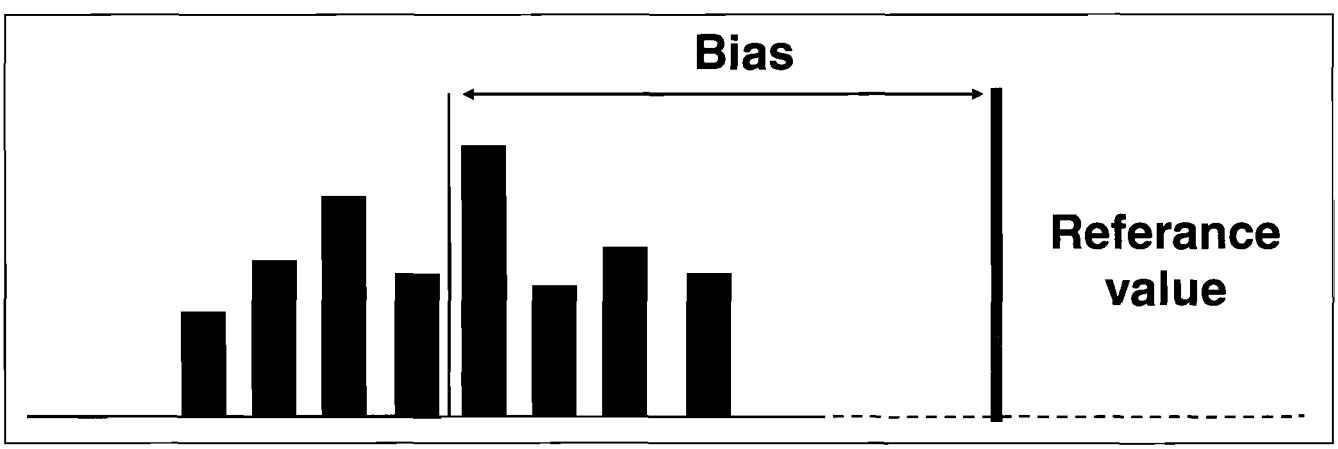


Figure 2. Bias

The methods that you use don't always measure the component that you are interested in. Mostly other components effect the results possitively or negatively. To avoid this problem, recovery has to be calculated for the method to evaluate its efficiancy for measuring the component that you are interested in. Recovery of a method can be calculated by study of referance materials or by spiking studies.

Precision (Repeatibility, reproducibility)

The principal precision measures include repeatability standard deviation, reproducibility standard deviation (ISO 3534-1) and intermediate precision. The repeatability indicates the variability observed within a laboratory, over a short time, using a single operator, item of equipment etc. Repeatibility may be estimated within a laboratory or by inter-laboratory study. Interlaboratory reproducibility standard deviation for a particular method may only be estimated directly by interlaboratory study; it shows the variability obtained when different laboratories analyse the same sample. Intermediate precision relates to the variation in results observed when one or more factors, such as time, equipment and operator, are varied within a laboratory; different figures are obtained depending on which factors are held constant. Intermediate precision estimates are most commonly determined within laboratories but may also be determined by interlaboratory study. The observed precision of an analytical procedure is an essential component of overall uncertainty, whether determined by combination of individual variances or by study of the complete method in operation.

Limit of Detection (LOD), Limit of Quantification (LOQ), Linearity, Working Range

During method validation, the detection limit is normally determined only to establish the lower end of the practical operating range of a method. Limit of Detection (LOD) is the minimum concentration that can be detected by the method in the laboratory condition. It is the lowest concentration of analyte in a sample that can be detected but not necessarily quantified. Limit of Quantification (LOQ) is the lowest level that you can quantified at which uncertainty is acceptable. Standard deviation of 10 replicates of the whole method for a blank sample or a lowest spiked sample can be used to

calculate the LOD and LOQ. Successive dilution (dilution until approximately %50 of results indicate no analyte present) is also used for determination of LOD and LOQ.

The linearity of an analytical method is its ability to elicit test results that are directly proportional to the concentration of analytes in samples within a given range or proportional by means of well-defined mathematical transformations. Linearity may be demonstrated directly on the test substance (by dilution of a standard stock solution) and/or by using separate weighings of synthetic mixtures of the test product components, using the proposed procedure.

The range of an analytical method is the interval between the upper and lower levels (including these levels) that have been demonstrated to be determined with precision, accuracy and linearity using the method. The range is normally expressed in the same units as the test results (e.g., percentage, parts per million) obtained by the analytical method.

Specificity/ Selectivity

The terms selectivity and specificity are often used interchangeably. The term specific generally refers to a method that produces a response for a single analyte only, while the term selective refers to a method that provides responses for a number of chemical entities that may or may not be distinguished from each other. If the response is distinguished from all other responses, the method is said to be selective. Since there are very few methods that respond to only one analyte, the term selectivity is usually more appropriate.

Selectivity in liquid chromatography is obtained by choosing optimal columns and setting chromatographic conditions, such as mobile phase composition, column temperature and detector wavelength. Besides chromatographic separation, the sample preparation step can also be optimized for best selectivity.

Robustness / Ruggedness

Robustness tests examine the effect that operational parameters have on the analysis results. For the determination of a method's robustness, a number of method parameters, for example, pH, flow rate, column temperature, injection volume, detection wavelength or mobile phase composition, are varied within a realistic range, and the quantitative influence of the variables is determined. If the influence of the parameter is within a previously specified tolerance, the parameter is said to be within the method's robustness range.

Ruggedness is a measure of reproducibility of test results under normal, expected operational conditions from laboratory to laboratory and from analyst to analyst. Ruggedness is determined by the analysis of aliquots from homogeneous lots in different laboratories.

QUALITY CONTROL of MEASUREMENT

Tools have to be identified to determine the quality of the results. There are 3 levels of Quality Control (QC)

1. Level QCs

These QCs are internal controls made by analysts in the laboratory daily. There are several tools which can be used for 1. level quality control:

- Analysis of a blank sample before working with a real sample
- Checking the calibration curve by an extarnel standard
- Using Control samples or referance material (Quality Control Charts)
- Parallel analysis

- Spiking of Standard, using an internal standard
- Cross check with another method

Quality Control (QC) charts are the most important tools those can be used for checking the performance of the method. Qualtiy Control charts (Figure 3) have to be prepared for whole of the method.

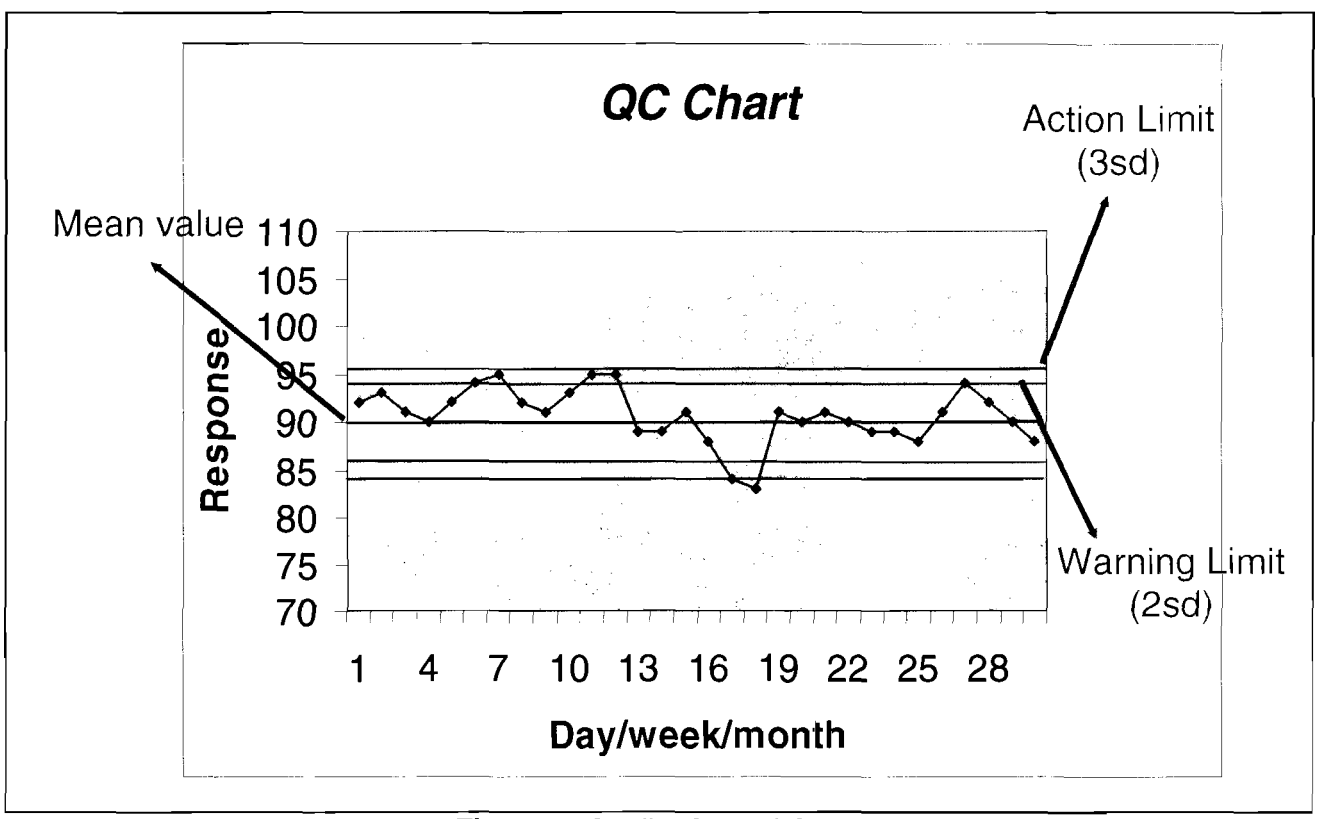


Figure 3. Quality Control Chart

You can use different criterias to evaluate a quality control chart. It can be suggested that at least following four criterias have to be used to evaluate a QC chart. If these following situations are observed, the analyst can begin to think that some of the validation parameters should have been changed.

Evaluation criterias:
- 11 sequential responses are on the same side of mean
- 2 responses in last 3 responses are out of 2sd (Warning limit)
- 1 response is out of 3sd (Action limit)
- tendency (increasing or decreasing) in 7 sequential responses

2. Level QCs

These QCs are internal controls made by quality manager of a laboratory periodicaly. The quality manager can prepare or provide a blank sample and/or referance material for the analyst to analyze. Then quality manager evaluate the results of the laboratory by comparing them with referance values.

3. Level QCs

These QCs are external controls. You can join to proficiency testing organized by an independent authority or to interlaboratory comparision testing

REFERENCES:

1. ISO/IEC/17025 on "General requirements for the competence of testing and calibration laboratories"

2. EUROCHEM Guide, The Fitness for Purpose of Analytical Methods, A Laboratory Guide to Method Validation and Related Topics, 1998

3. EURACHEM/CITAC, Second Edition, 2000 (QUAM:2000. Quantifying Uncertainty in Analytical Measurement

4. ISO 3534-1:2006, Statistics - Vocabulary and symbols - Part 1: General statistical terms and terms used in probability

5. ISO 5725-1:1994(E). Accuracy (trueness and precision) of Measurement Methods and
6. Results-Part 1: General Principles and Definitions.

7. ISO 5725-2:1994, Accuracy (trueness and precision) of Measurement Methods and Results-Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method.

8. ISO 7870:1993. Control Charts – General Guide and Introduction

9. ISO 7873:1993. Control Charts for Arithmetic Average with Warning Limits

Principles of method validation and measurement uncertainty in microbiological analysis

There are only two absolute certainties in life: death and taxes! Whatever task we undertake, no matter how menial or how sophisticated, we are faced with a lack of certainty in the outcome. It is therefore essential to have a common understanding of what is meant by uncertainty.

Accuracy is defined (ISO3534–2.2003) as "*the closeness of agreement between a test result or a measurement result and the true value.*" Accuracy is a combination of trueness and precision (a combination of random components and systematic error or bias components). "*Accuracy*" is essentially "absence of error"; the more accurate a result the lower the associated error of the test. It is important to note that the term "accuracy " applies only to results and can not be applied to methods, equipment, laboratories or other general matters. "***Trueness***" is defined (ISO, 2003) as, "the closeness of agreement between the average value obtained from a large series of test results and an accepted reference value". *Trueness* is equivalent to an absence of "***bias***", which is the difference between the expectation of the test results and an accepted reference value and is a measure of total systematic, but not random, error. *Trueness*, unlike *accuracy*, may correctly be contrasted with *precision.* "***Precision***" is defined as the closeness of agreement between independent test results obtained under stipulated conditions. *Precision* depends only on the distribution of random errors and does not relate to a true value or a specified value. *Independent test results* means results obtained in a manner not influenced by any previous results on the same or similar test object.

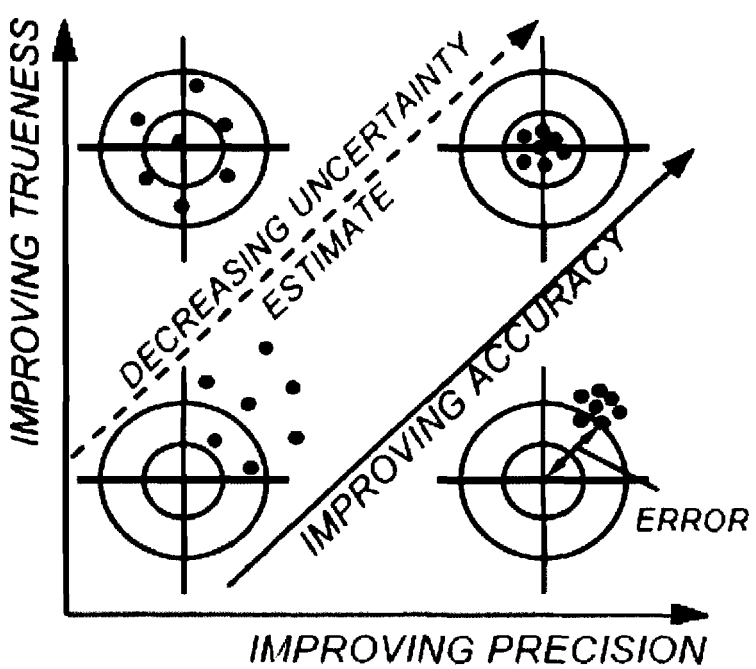


Fig 1. Relationships between trueness, accuracy, precision and uncertainty in analytical results (AMC, 2003).

The validation of microbiological test methods should reflect actual test conditions. This may be achieved by using naturally contaminated products or products spiked with a predetermined level of contaminating organisms. The analyst should be aware that the addition of contaminating organisms to a matrix only mimics in a superficial way the presence of the naturally occurring contaminants. However, it is often the best and only solution available. The extent of validation necessary will depend on the method and the application.

In microbiology the test result is usually derived from several **observed values** involving many measurements. The term **measurement** is used loosely to denote both a single observed value and the result calculated from a combination of them. **Uncertainty** of measurement according to ISO (Anon 1995) is a parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand. (Measurand is a general term for any particular quantity subject to measurement.)The most natural expression of uncertainty of microbiological test results is usually the **relative standard uncertainty**,

For quantitative data (e.g. colony counts, MPNs.) a measure of uncertainty may be any appropriate statistical parameter associated with the test result. Such parameters include the standard deviation, the standard error of the mean or confidence interval around that mean.

A standard deviation (standard uncertainty) is calculated from a series of *n* independent parallel measurements *x*1, *x*2,...,*x*n of the test quantity using the conventional statistical formula for experimental standard deviation:

$$s(x) = s_x = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

($\bar{x}$ is the arithmetic mean)

Arithmetical mean, *x* mean of a list of numbers is the sum of all of the list divided by the number of items "n" in the list.

Measures of repeatability and reproducibility are the corner stones of estimation of analytical uncertainty. They are defined (ISO 2004) as:

Repeatability is "a measure of variability derived under specified ***repeatability conditions"***, i.e. independent test results are obtained with the same method on identical test items in the same laboratory by the same analyst using the same equipment, batch of culture media and diluents, and tested within short intervals of time.

Reproducibility is "a measure of precision derived under ***reproducibility conditions"*** i.e. test results are obtained with the same method on identical test

items in different laboratories with different operators using different equipment. A valid statement of reproducibility requires specification of the conditions used.

Quantitative microbiological test methods; the specificity, sensitivity, relative trueness, positive deviation, negative deviation, repeatability, reproducibility and the limit of determination within a defined variability should be considered and, if necessary, quantitatively determined in assays. The differences due to the matrices must be taken into account when testing different types of samples. The results should be evaluated with appropriate statistical methods.

Qualitative microbiological test methods; such as where the result is expressed in terms of detected / not detected and confirmation and identification procedures, should be validated by determining, if appropriate, the specificity, relative trueness, positive deviation, negative deviation, limit of detection, matrix effect, repeatability and reproducibility

The total uncertainty of a test result typically consists of several components. In microbiology, at least three factors are always involved: the uncertainty of the inoculum volume, random scatter due to particle statistics, and the uncertainty of reading the result. Uncertainty of dilution is frequently a fourth factor. Other components (e.g. sample stability and sample preparation) cannot be measured directly and their contribution cannot be evaluated in a statistical manner but their importance to the variability of results should be considered also.

In microbiological laboratory practice, we can identify many causes of variability, for instance:

- The ability of an isolate to give typical reactions on a diagnostic medium;
- The use of the incorrect ingredients in a culture medium;
- The consequence of changing brands of commercial media;
- Use of non-standard conditions in the preparation, sterilization and use of a culture medium;
- Equipment and human errors in weighing, dispensing, pipetting and other laboratory activities;
- The tolerance applied to the shelf life of test reagents;
- The relative skill levels of different technicians;
- The relative well-being of any technician who is undertaking analyses;
- and so on, and so on....

According to ISO 17025; Reference materials and certified reference materials provide essential traceability in measurements and are used, for instance;

- to demonstrate the accuracy of results,
- to calibrate equipment,
- to monitor laboratory performance,
- to validate methods, and
- to enable comparison of methods.

If possible, reference materials should be used in appropriate matrices.

Reference cultures are required for establishing acceptable performance of media (including test kits), for validating methods and for assessing/evaluating on-going performance. Traceability is necessary, for example, when establishing media performance for test kit and method validations. To demonstrate traceability, laboratories must use reference strains of microorganisms obtained directly from a recognised national or international collection, where these exist. Alternatively, commercial derivatives for which all relevant properties have been shown by the laboratory to be equivalent at the point of use may be used.

Internal quality control Internal quality control consists of all the procedures undertaken by a laboratory for the continuous evaluation of its work. The main objective is to ensure the consistency of results day-to-day and their conformity with defined criteria. A programme of periodic checks is necessary to demonstrate that variability (i.e. between analysts and between equipment or materials etc.) is under control. All tests included in the laboratory's scope of accreditation need to be covered. The programme may involve:

- the use of spiked samples
- the use of reference materials (including proficiency testing
- scheme materials)
- replicate testing
- eplicate evaluation of test results

The interval between these checks will be influenced by the construction of the programme and by the number of actual tests. It is recommended that, where possible, tests should incorporate controls to monitor performance.

Laboratories should regularly participate in proficiency testing which are relevant to their scope of accreditation; preference should be given to **proficiency testing** schemes which use appropriate matrices. In specific instances, participation may be mandatory.

References:

Seppe I. Niemela, 2002, Uncertainity of Quantitative Determinations Derived by Cultivation of Microorganisms.

EA-04-10 Accreditation for Microbiological Laboratories.

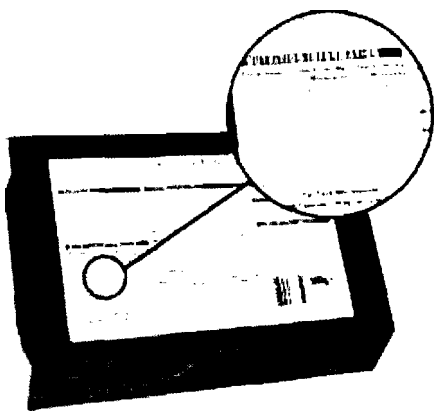
19036- Uncertainity Associated with Microbiological Analysis

Lynne I.Fosster, 2003, Measurement Uncertainity in Microbiology.

Food Labelling

Regulations, Importance, Applications

Consumers today expect a great deal of information about the food products they purchase. Providing this information on food labels helps consumers to make informed choices, and can help people to choose between different types, brands and flavours of foods.



Why do we need food labelling?

Can you think of any reasons for having food labelling? Would you buy a tin of food from a supermarket that had no label on it? How would you know what was in it?

Food labels have many different functions, for example:

1. Providing information for the consumer, such as a description of the food
2. Making a product stand out from others, thereby promoting its sale
3. Ensuring consumer safety, for example by showing food storage and cooking information

Can you think of any others?

What's on a label?

In addition to information that helps consumers choose between products, European Union (EU) law requires that certain information is displayed on a food label. This acts as a safeguard for the consumer.

Information required by European Law

The name of the food

The name of the food must be clearly displayed on all pre-packed foods. This is one of the most important pieces of information on a label. For example, it is not sufficient to just say 'cheese', because there are so many different types of cheese. Other foods have made-up names which give no information about what is in the food. In such cases, a clear description of the food must be given, for example "M&M's" – milk chocolate covered peanuts in a crisp coloured shell. The name must also include information about any processing the food has undergone, for example *dried* apricots, *salted* peanuts or *smoked* mackerel. It must also describe the differences between apparently similar products. For example a yogurt labelled 'fruit yogurt' must be flavoured with real fruit, whereas a yogurt labelled 'fruit flavoured yogurt' can be flavoured with artificial flavourings.

Weight or volume

The weight or volume of pre-packed foods must be shown on the label. The symbol e indicates an average quantity i.e. the average pack is at least the weight declared. Comparing the weight with the price of different brands enables consumers to make choices between brands based on value for money.

Ingredients

Ingredients must be listed in descending order of weight, according to the amounts used to make the food. The ingredient names must be given in the language of the country where the food is to be sold. All additives must be stated on the ingredients list.

Allergens

A new European Union (EU) labelling rule introduced in 2004 requires 12 food ingredients that may cause adverse reactions in some individuals - milk, eggs, peanuts, nuts from trees (e.g. walnuts), fish, crustaceans, soya, wheat, celery, mustard, sesame and sulphur dioxide – to be clearly labelled on food products. This is particularly helpful to people with food allergies and food intolerances, who may need to avoid these specific food ingredients.

Date and storage conditions

Information must be provided on how long a product will keep, and how the product should be stored. 'Date marking' provides an important safeguard against foods which may be unsafe to eat. Following storage instructions can prevent food from spoiling too quickly, and helps to ensure that food looks and tastes its best when eaten. Perishable foods that spoil quickly, such as meat and fish products, have a 'Use by' date. 'Use by' provides clear instruction that a food should be used by the end of the date shown on the label. Remember though that freezing a product can extend a perishable product's shelf life, although not indefinitely. Products suitable for freezing sometimes indicate the extended shelf life attributable to freezing, if products are frozen according to the instructions on the label. Other foods which can be kept safely for a longer period of time (e.g. cornflakes) have a 'Best before date'. After this date foods may still be safe to eat, but they may not be at their best with regards to their flavour, colour and texture.

Preparation instructions

Where relevant, instructions on how to prepare and cook a food must be given on the label. These instructions help to ensure that a food tastes its best when eaten, and that it will be thoroughly heated to a core temperature of 75°C to minimise the risk of food poisoning.

Name and address of manufacturer or seller

The name and address of the manufacturer or seller must be stated on the label. For branded products, this must be the name of the manufacturer. For 'own label' this is the name and address of the retailer.

Place of origin

'Place of origin' must be included on the label if it is unclear where the food has come from. For example some Greek yogurts are made in France and it might mislead the consumer if this is not properly labelled.

Lot or batch number

The lot or batch number is a code that can identify batches of food in the event that they have to be recalled by the manufacturer, packer or retailer.

Additional information

Nutrition labelling

Nutrition labelling can help consumers to make healthy choices and can provide clear consistent messages to consumers. Nutrition labelling is only required by EU Law where a nutritional claim about a product is made (e.g. low fat, rich in calcium), but as consumers' knowledge about health and diet grows, many manufacturers are choosing to display nutrition information on food labels. Manufacturers who voluntarily provide nutrition information, or do so because they make a nutrition claim, must follow the rules of the EC Nutrition Labelling Directive 1990. This directive ensures that nutrition information is presented in a standard way, allowing consumers to easily compare the nutrient content of one food with another. The legislation states that:

- The energy value of the food in kilojoules (kJ) and kilocalories (kcal) must be provided
- The amount of protein, carbohydrate and fat in grams (g) must be provided
- Optionally (unless a nutrition claim is made) the amounts of sugars, saturates, fibre and sodium can be provided, if the first four nutrients have been provided.

Nutrition information must always be given as values per 100g or per 100ml of food. Values for a portion or serving can be given as well, provided that the number of servings contained in the pack is shown on the label. The legislation also allows inclusion of information on the amounts of other fatty acids, cholesterol and some specified vitamins and minerals (if they are present in significant amounts) contained within the food. This is optional unless a claim is made about one of these nutrients, in which case it is obligatory that information on the particular nutrient is provided.

Nutrition and health claims

Food manufacturers often want to draw attention to the amount of nutrients present in a food, outside of the nutrition information panel on the label. Nutrition and health claims provide an opportunity for manufacturers to highlight any specific nutritional benefits a food many have, beyond the provision of nutrients for general health and well being. Nutrition claims refer to any statement outside of the nutrition information panel, which declares or implies that a food contains one or more nutrients. In Europe, many nutrition claims are defined in legislation (e.g. 'source of calcium') , but there is currently no specific legislation relating to health claims e.g. 'can help reduce blood cholesterol' etc. (although the law says that any claim made must be true and not misleading). Nutrition is a fast-moving subject area, but European legislation relating to nutrition and health claims has not kept up with product development. This has led to manufacturers across Europe developing their own criteria for nutrition and health claims, often causing consumer confusion. Some countries have also established voluntary systems by which manufacturers can request assessment of health claims, e.g. the Joint Health Claims Initiative in the UK (http://www.jhci.co.uk/). The European Commission is currently looking at harmonising the different national laws relating to nutrition and health claims, in order to protect consumers and promote innovation in food. For more information on this topic, see the Nutrition and Health Claims web feature.

Where does nutrition information come from?

We have seen why food labelling is important, and what information is displayed on food labels, but where do manufacturers get the nutrition information for their products from? Nutrition information for use on food labels can be obtained from two main sources.

1. By direct chemical analysis

   The 'gold standard' for manufacturers to obtain nutrition information for their products is to chemically analyse samples of their products in a laboratory. For example, the protein content of a food can be determined by measuring the food's nitrogen content in the laboratory, and this value used to calculate the protein content of the food. The EC Nutrition Labelling Directive 1990 provides energy conversion factors which manufacturers must use to calculate how much energy is in the food. For example, 1g of protein provides 4 kcal of energy and 1g of fat provides 9 kcal. While chemical analysis of a food provides

accurate information on the food's nutrient content, it is often expensive, and many manufacturers lack the facilities and expertise to analyse their products in-house. A more cost effective and equally acceptable way for manufacturers to calculate nutrition information for their products is to use officially published food composition data.

2. Using food composition data to inform food labelling

Food composition databases (FCDBs) contain information on the nutrient content of a range of different foods and ingredients. Typically, FCDBs provide values for the amount of energy, protein, fat and vitamins & minerals that 100g of a particular food or ingredient contains. The nutrient values for the foods and ingredients listed in the database are usually derived by chemical analyses of the foods and then compiled into databases. Manufacturers may then use the nutrient values from these official databases to calculate the nutrition information for their products. Where the manufacturer's product itself has not been analysed by the database compilers (e.g. a cook-chill lasagne), the manufacturer can calculate the nutrient content of the product from the nutrient values of the individual ingredients that make up the product. For example, in the case of the cook-chill lasagne, the nutrition information can be calculated by adding the nutrient values of the correct amounts of pasta, minced beef, tomatoes and other ingredients that make up the lasagne.

Reference

- http://www.eurofir.net/public.asp?id=2241

- European laws covering food labelling (e.g. EU Directive 2000/13/EC)
http://europa.eu.int/comm/food/food/labellingnutrition/nutritionlabel/index_en.htm

TÜBİTAK
MAM

INTERNATIONAL WORKSHOP AND STUDY TOUR ON: FOOD LABORATORIES MANAGEMENT AND PRACTICE

UNISWORK VIII

TUBITAK

MARMARA RESEARCH CENTRE

FOOD INSTITUTE

LABORATORY APPLICATIONS

Gebze- Kocaeli, TURKEY

16-20 November 2009

TÜBİTAK
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UNIDO International Training Programme on
Food Laboratory Management and Practice
16–20 November 2009

Draft Programme for the UNISWORK VIII
LABORATORY APPLICATIONS:

10:00-11:15	**LABORATORY APPLICATIONS:** Microbiological analyses (Total viable count, *E.coli*, *S.aereus*) (GroupA) Nutrition analyses (protein, Vit C and Vit A analyses) (Group B)
11:15-12:30	**LABORATORY APPLICATIONS:** Microbiological analyses (Total viable count, *E.coli*, *S.aereus*) (GroupB) Nutrition analyses (protein, Vit C and Vit A analyses) (Group A)
12:30-14:00	
14:00-15:15	**LABORATORY APPLICATIONS:** Food chemistry analyses (total sugar, invert sugar, salt, HMF, total acidity) (Group A) Mineral and heavy metal analyses (Ca, Zn, Pb, Cd,) (Group B)
15:15-15:30	Coffee break
15:30-16:45	**LABORATORY APPLICATIONS:** Food chemistry analyses (total sugar, invert sugar, salt, HMF, total acidity) (Group B) Mineral and heavy metal analyses (Ca, Zn, Pb, Cd,) (Group A)
09:30-10:30	**LABORATORY APPLICATIONS:** Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group A) Oil/Fats analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group B)
10:30-11:00	Coffee break
11:00-11:45	**LABORATORY APPLICATIONS:** Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group A) Oil/Fats analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group B)
11:45-13:00	
13:00-14:45	**LABORATORY APPLICATIONS:** Sugar components, aflatoxins and deoxynivalenol analyses by HPLC (Group B) Oil analyses (free fatty acid, peroxide, fatty acid composition by using GC (Group A)

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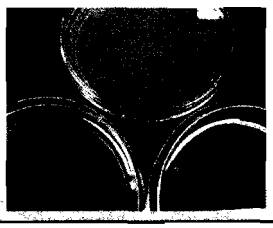
Microbiological analysis
Total viable count, *E-coli*, *S.aureus*



UNIDO
International Training Programme
November 16-20, 2009
Food Institute

N. Aslı Kısıkkaya ÖNCÜ (MSc.)

MAM

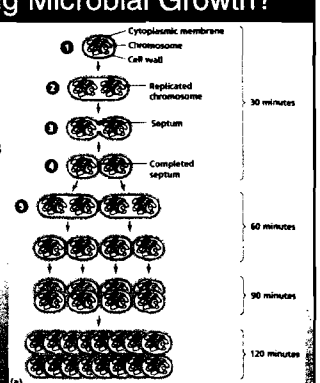
- Provide Clean,Safe, Healtful Food to Consumer
- ❖Food Permits Growth
- ❖Control of Microbial Growth

- Prevent Food Spoilage.
- Prevent Food Borne Illness

MAM

Factors Influencing Microbial Growth?

- ❖ *Nutritional* requirements
- ❖ Oxygen requirements
- ❖ Temperature
- ❖ pH

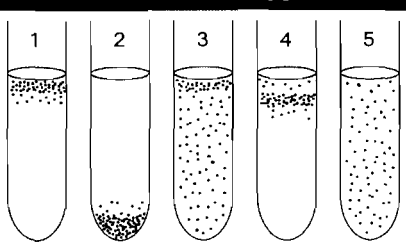


Nutrition-General

- Organisms use a variety of nutrients for their *energy needs* and to *build organic molecules* and *cellular structures.*

- Most common nutrients – those containing necessary elements such as oxygen, nitrogen, and hydrogen.

Microbes & Oxygen

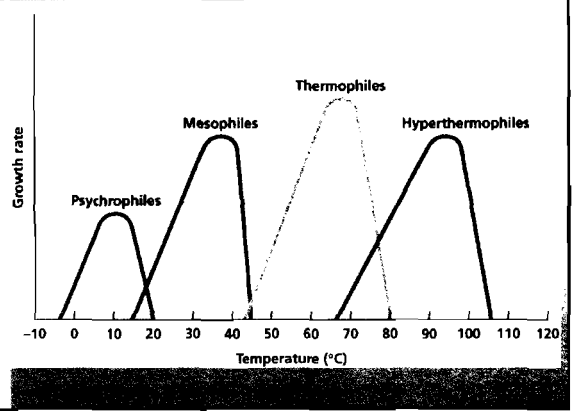


1-Obligate aerobic bacteria gather at the top of test tube to absorb .

Microbes & Temperature

- Three-dimensional shape because of the temperature sensitive hydrogen bonds.
- These bonds will usually break at higher temperatures, and protein become.
- Denatured proteins lose function.
- Become *brittle* if temperature is too low.

Categories of Microbes Based on Temperature Range



Growth Media

Media = Mixtures of nutrients that the microbes need to live.

Also provides a surface and the necessary moisture and pH to support microbial growth.

Growth Media

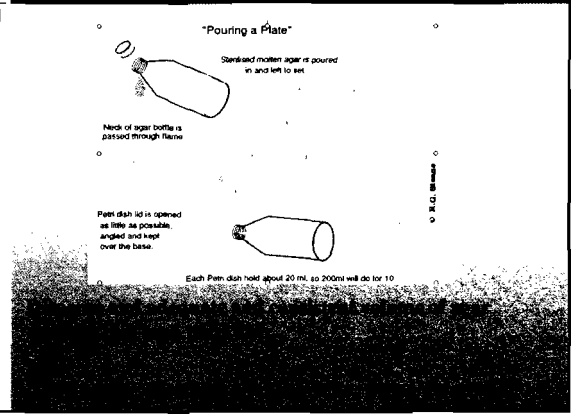
- Bacteria and other microbes have particular requirements for growth.
- In order to successfully grow bacteria in lab, we must provide an environment suitable for growth.
- Growth media *(singular = medium)* are used to cultivate microbial growth.
- Tryptic Soy Agar (TSA) is the media that most

How is made media?

- When lab personnel make media they measure out a quantity of dry powdered nutrient media, add water and check the pH. (1 N NaOH / 1 N HCL)
- Why adjust pH? It is very important to verify and adjust the pH of culture media.
- They dispense the media into bottles, cap it and autoclave.
- This is a process similar to home canning techniques in food preservation.
- The autoclave exposes the media to [illegible] and pressure

Preparing and Storing Culture Media

- Flask, weighing instruments and all utensils must be clean.
- Culture media must be prepared using water purified by distilation. (Never use tap water)
- Always try to time your work so that culture media are autoclaved promptly.
- Always allow sterilizied media to cool before adding heat-sensitive supplements or dispensing into petri dishes.



Storage of Culture media

- Protect media from contamination during and after pouring.
- Incorrect storage will ruin the best quality culture medium.
- Always store plated media in the refrigerator.
- Proper storage of media ingredients is also very important to the performance of the finished culture medium.

Enumeration Methods

- Pour Plate Method (Total bacteria counts, Enterobactericia.)
- Spread plate method (*S.aureus*)
- Tube MPN procedures (*E-coli*, Coliforms)

How to dilute?

- Weigh the sample (10 gr or 25 gr)
- Add *diuent (Peptone dilute solution etc)*
- Homegenize as appropraite to the sample.
- This proces is the initial 10^{-1} dilution of the original sample.
- *Then transfer by pipette 1 ml of the homogenizate to a* sterie 9 ml dilution blank. This results in a 10^{-2} dilution of the original sample.
- Then use fresh pipette to transfer 1 ml of the 10^{-2} dilution into another 9 ml dilution blank, producing a 10^{-3} dilution of the original sample.

Pour Plate Method

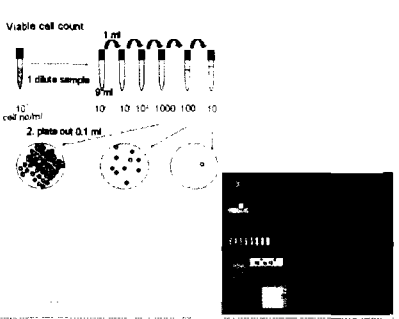
- Prepare a sample homogenate.
- Dilute, pipette 1 ml volumes into Sterile Petri dishes.

- Pour molten agar into each dish.(The temperature of the melted Agar should be 44-45 C°because higher Temperature can lead to cell Shock or cell injury and give a lower than true count.

Total viable count

- Using seperate sterile pipets, prepare decimal dilutions of 10^{-2} , 10^{-3} , 10^{-4} ..etc as appropriate, of food homogenate transfering 25 ml of previous dilutions to 225 ml of diluent .
- Pipet 1 ml of each dilution into seperate, duplicate, appropriately marked Petri plates.
- Add 15 ml of Plate Count Agar (cooled to 45 ±1 C°) to each plate.
- Immediately mix sample dilutions and agar medium thoroughly and uniformly.

Total viable count



Spread plate Method

- The most common use of the method is for enumarating "*Staphylococcus aureus*"
- Spread plates can also be used for yeast&mold enumeration, The reason; for this is that most molds are aerobes (need oxygen for growth) and grow best on the surface of a culture medium instead of being

Spread Plate Method

- Examine each plate carefully before inoculation.(The surface of the agar should not have any drops of moisture on it.)
- Spread the inoculum promptly. Spread the liquid uniformly andThoroughly over the surface.
- Incubate plates inverted.

Staphylococcus aureus

- The term "Staphylococci"informally describes a group of small, spherical, gram positive bacteria.
- They are catalase positive, have typical gram positive cell.
- The genus Staphylococcus is subdivided into >23 species and subspecies.
- Several species of Staphylococcus, including both coagulase-negative and coagulase-postive isolates, can produce staphyloccal enterotoxins.
- Although several species can cause gastroenteritis, nearly all staphylococcal food poising is attributed to *S.aureus*.

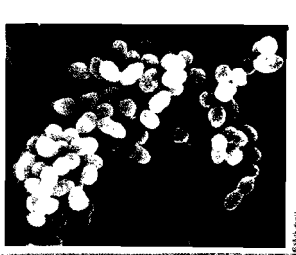
Staphylococcus aureus

- Spread plate method is the most common use method for enumarating "*Staphylococcus aureus*"
- Using seperate sterile pipets, prepare decimal dilutions of 10^{-2} , 10^{-3}, 10^{-4}..etc as appropriate, of food homogenate transfering 25 ml of previous dilutions to 225 ml of diluent .
- Asseptically transfer 1 ml sample suspension to 3 plates of Baird Parker medium, distributing 1 ml of inoculum equitably to 3 plates (e.g., 0.4 ml, 0.3 ml, and 0.3 ml).
- Invert the plates and incubate 45-48 h at 37 °C.

Staphylococcus aureus

- Select plates containing 20-200 colonies and count number of colonies and records counts.
- Colonies of S.aureus are circular, smooth, convex, moist, 2-3 mm in diameter on uncrowded plates, gray to jet-black, frquently with ligth-colored (off-white) margin, surrounded by opaque zone and

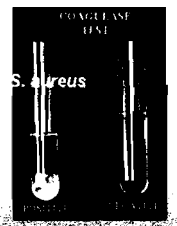
Electron micrograph of ***S.aureus*** cells



Identification tests of *S.aureus*

- Transfer suspect S.aurues colonies into small tubes containing 0.2-0.3 ml Brain Hearth Infusion Broth (BHI).
- Then inoculate agar slant of suitable medium. For instance; Trypticase Soya Agar (TSA)
- Incubate BHI culture suspention and TSA petri dish 24 hours at 35 C°.
- **Retain slant cultures at room temperature for repeat test in case coagulase test results are questionable.**
- **Add 0.3 ml reconstituted coagulase**

COAGULASE TEST



S.aureus

- Catalase test: Use growth from TSA slant for catalase test on glass slide or spot plate and iiuminate properly to observe production of gas bubbles.
- **Gas bubble indicate positive test.**

- ***'Staphylococcus aureus"***

Tube MPN Methods

- MPN tables are built on a three dilution series.
- You must prepare 10^{-1} sample homogenate and then make a 10^{-2} and 10^{-3} dilutions.
- Use replicate 1 ml volumes from each dilution.
- **Place the rack of tubes into the incubator.**

Reading and Confirming MPN tubes

- Each tube can be examined simply for growth (For instance; color change due to an enzymatic reaction in the medium or for gas produced as a result of sugar fermentation.
- Gas is detected by including in each MPN tube a small inverted test tube also known as a Durham vial, which traps any gas that migth be generated by the microbes during incubation.
- A confirmed positive tube is usually defined as one [illegible]

Coliforms, Fecal coliforms and *E-coli*

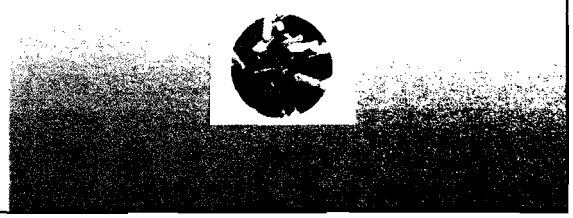
- Gas bubbles provide the diagnosite clue for this method.
- Subculture each tube in which gas has been trapped in the Durhan vial by transfering on drop of liquid to each of two other liquid media.
- Brillant Green lactose Bile Broth (BGLB), to [illegible]

Coliforms, Fecal coliforms and *E-coli*

- BGLB is incubated at 35-37 C°and tubes in which gas has been produced are considered by defination to contain coliforms.
- Incubate EC broth at 44.5 C°or 45 C°and examine each tube for gas production.
- EC tubes in which gas has been produced [illegible]

E-coli

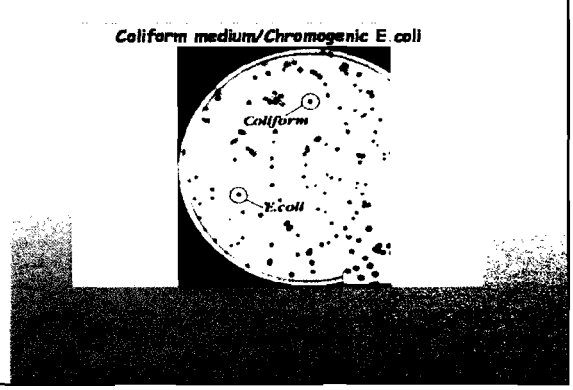
- To confirm *E-coli* from a positive EC tube, streak a loopful of liquid to Levine's Eosin Methlene Blue Agar (EMB) after incubation, subculture two suspect colonies from each positive EMB plate for IMVIC patterns.



IMVIC test

- *Indole production*. Inoculate tube of tryptone broth and incubate 24 ± 2 h at 35°C. Test for indole by adding 0.2-0.3 mL of Kovacs' reagent. Appearance of distinct red color in upper layer is positive test.
- *Voges-Proskauer (VP)-reactive compounds*. Inoculate tube of MR-VP broth and incubate 48 ± 2 h at 35°C. Transfer 1 mL to 13 x 100 mm tube. Add 0.6 mL α-naphthol solution and 0.2 mL 40% KOH, and shake. Add a few crystals of creatine. Shake and let stand 2 h. Test is positive if eosin pink color develops.
- *Methyl red-reactive compounds*. After VP test, incubate MR-VP tube additional 48 ± 2 h at 35°C. Add 5 drops of methyl red solution to each tube. Distinct red color is positive test. Yellow is negative reaction.
- *Citrate*. Lightly inoculate tube of Koser's citrate broth; avoid detectable turbidity. Incubate for 96 h at 35°C. Development of distinct turbidity is positive reaction.
- [illegible]. Inoculate a tube of LST and incubate 48 ± 2 h at [illegible]

Coliform /Chromegenic *E-coli*




MAM
Thank you for your attention!

NUTRITION ANALYSES (PROTEIN, VIT C AND VIT A ANALYSES)

PROTEIN DETERMINATION

(Kjeldahl method, AOAC 1995)

Chemicals:

Sulphuric acid, Kjeltab, sodium hydroxide, boric acid, bromcresol green, methyl red, methanol, hydrochloric acid, oxalic acid, sodium carbonate.

- Sulphuric acid: technical quality (no nitrogen included)
- Kjeltab: 3.5g potassium sulfate, 0.0035g selenium
- Alkaline solution: sodium hydroxide with technical quality at a concentration of %35-40 (weigh 3700g sodium hydroxide with technical quality and adjust the volume to 10L with 10L distilled water) (1M) or adjust all calculations for 1L.
- Receiver solution: dissolve %1, 10L boric acid solution (100g boric acid) in 10L water and %1 boric acid solution is obtained. Onto it add 100mg bromcresol green (bromcresol green solution which is prepared by dissolving 100mg bromcresol green in 100ml methanol). Then add 70ml methyl red solution (100mg mthyl red is dissolved in 100ml methanol) into the main solution. Shake well. Take 25ml from boric acid solution. Put into receiver tube. Add 100ml distilled water into the solution. If the color is still red, add 0.1M sodium hydroxide (take 100ml from the solution of 1M sodium hydroxide prepared above and complete to 1L with distilled water and 0.1M sodium hydroxide is obtained). Titrate the solution till the color of the solution turns into natural gray. Amount of the titrant is the amount for adjustment of 10ml, %1 boric acid solution and it is calculated by putting it into the formula.

ml of 1.0M alkaline=ml titrant x 40

or all calculations are adjusted for 1L.

- Acid solution: 0.1N hidrochloric acid.
- The solution of sodium hydroxide: weigh 4g sodium hydroxide and dissolve it with distilled water. Adjust the volume to 1L with distilled water (0.1N sodium hydroxide solution) titrate it with oxalic acid to obtain the factor of sodium hydroxide. And this factor is used for obtaining the factor of hidrochloric acid. The correction which is made by sodium carbonate is made also with this application.
- Standard acid solution: prepare 0.1M hidrochloric acid solution with using %37 hidrochloric acid (weigh 8.28ml, %37 hidrochloric acid and adjust the volume to 1L with distilled water). For the aim of controlling the molarity and factor of the solution add primer standard material (sodium carbonate). Weigh 10g from dried sodium carbonate.Dry it at 265°C in an oven for an hour. Take it in a desicator and protect it in a bottle not to gain any moisture.
- Indicator solution: take 0.1g methyl red and 0.1g bromcresol green. Dissolve them together in 100ml methanol.
- Weigh 0.4g of sodium carbonate standard substance(it is the weight W1). Add 40ml distilled water. Add 10 drops indicator solution. Titrate it with hidrochloric acid solution. Note the volume of the titrant when the color of the solution become pink (it is the volume A1). Boil it a few sec. Cool it under the tap immediately to room temperature. Titrate it with the hidrochloric acid again. Note the volume of the titrant when the color of the solution become pink again (it is the volume A2).

$$\text{Molarity (M)} = (18.686 \times W1) / (A1+A2)$$

Analysis:

Procedure:

- Make the sample homogeneous.
- Weigh 0.1-1g from the sample and put it the Kjeltec baloon.
- Add a Kjeltac tablet and 12ml, %97 sulfuric acid onto it.
- Put it on the heating unit. Boil it for an hour under the hood with vacuum. Sample b become clear when burned.
- Cool it to room temperature.
- Add 60 ml distilled water onto it.
- Ttitate it with using Kjeltch Auto 1030 Analyzer apparatus.

Calculations:

Nitrogen (%) = (1.4007 x M f x (A-B))

1.4007: the atomic weight of the nitrogen equivalent to 0.1ml, 0.1N hidrochloric acid

M: molarity of acidity (mol/L)

f: factor of standard Kjeldahl

A: the amount of titrant used (ml)

B: the control used (ml)

W: amount of the sample (g)

To translate the % nitrogen to % protein use factors.

Protein (%) = nitrogen (%) x factor of protein (vary with respect to the food)

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Protein factors of foods with respect to their nitrogen levels:

	F
• Milk and milk products:	6.38
• Cereal and cereal products:	5.80
• Soybean and soybean products:	5.71
• Oily seeds and fruits with hard shells:	5.30
• Mushrooms:	4.17
• Other foods (ex: meat and meat products, fish, fruit, vegetables,...etc)	6.25

Reference

AOAC Official Method 960.52

ASCORBIC ACID (VITAMIN C) IN VITAMIN PREPARATIONS AND JUICES (967.21/AOAC 1995)

Principle: Ascorbic acid reduces oxidation reduction indicator dye, 2,6 dicloroindophenol to colorless solution. At end point excess unreduced dye in rose pink in acid solution. Vitamin is extracted and filtration performed in presence HPO3-CH3COOH or HPO3-CH3Cooh-H2SO4 solution to maintain proper acidity for reaction and to avoid autoxidation of ascorbic acid at high pH.

Solutions: %6 HPO3 (60 g HPO3 weigh and dissolve in 1 L of distilled water), %3 HPO3 (Pipette 500 ml of HPO % 6 in a volumetric flask and complete the volume with distilled water to 1L), %0.025 2,6 dichlorophenol (weigh 50 mg of 2,6 dichloroindophenol and dissolve in 150 ml of hot distilled water. Add 42 mg of NaHCO3 and cool Complete the volume to 200 ml with distilled water. This solution can be stored at refrigerator about 1 week.

Sample preparation: Homogenized 200-30g of sample and same amount of %6 HPO. And mix. Homogenize. Take 10-30 g of sample in a volumetric flask. Adjust the volume with %3 HPO3 to 100 ml. Filtrate. Take 10 ml for titration.

Standard preparation: Weigh 100 mg of standard. Dissolve with %3 HPO3 and adjust the volume to 500 ml. Transfer 10 ml to an erlen. Add 5 ml of %3 HPO3. Titration to rose pink color. T= 1/ml of titrated indophenol

Calculation:

V= color volume of sample titrated
T= ascorbic acid equivalent to 1 ml indophenol standard solution
W= volume for titration of dilution
Sample: 200 g sample +200 g %6 HPO3
20 g mix+100 ml dilution
Used volume of indophenol for titration=10 ml
Ascorbic acid (mg/100g)= V X T X 100/W =200 X 20/400 X 100 = 1 g
T= 1/8.43= 0.119
V= 2 ml 2 X 0.119/1 X 100 = 23.80 mg/100g
Reference
AOAC Official Method 967.21

VITAMIN A (RETINOL) ANALYSIS METHOD

Purpose and Scope

The determination of vitamin A (retinol) in foodstuffs by High performance liquid chromatography (HPLC).

Application Methods

Equipment

HPLC

Column: MAXSIL 5 Slica 250*4.00 mm 5 micron P/NO 00G-0053-D0 phenomenex

Detector.Fluorescencedetector

Excitation:325nm

Emission:480nm

Mobile Phase: 98% n-Hexane, 2% Isopropanol

Flowrate:1ml/min

Injection volume: 50µl

- *Shaking water bath*

Maintaining 80±2°C, variable speed.

- *Glassware*

250 ml separatory funnels, volumetric balloon flasks.

- *Rotary evaporator*

Solutions;

- *Extraction solution (100 ml):*

0.5 g Ascorbic acid

4 ml distilated water

10 ml ethanol

100 ml methanol

% 50 KOH (100ml): 50 g KOH was mixed with distilled water up to 100ml.

- *Chemicals:*

n-Hexane

Diethyl ether

Propanol

Butylated hydroxytoluene (BHT)

Mobil phase solution:

980ml hexane

20ml propanol

2.3 Preparation of Samples and Standards

2.3.1. Sample Preparation

Homogenize test sample. Weigh 10-30g in the balloon flask (250 ml).
Add 50 ml extraction solution and 5ml %50 KOH solution in test portion and mix.
Add a spatula tip of BHT in to test portion in order to prevent from oxidation. Place flasks in 80°C shaking water bath for 30 min.
Remove flasks and place in ice 5 min, or until contents cool to room temperature.

2.3.2. Extraction

Quantitively transfer contents to separate 500 ml separatory funnels. Trasfer the solution at room temperature into a separatory funnel and over add 50 ml distillated water. Add 100 ml diethylether in to separating tunnel and extract it. When the two phases are appeared, take the lower phase into balloon flask. Take upper phase into a different erlen mayer or baloon flask. Repeat separation step 3 times. Take the collected upper phases into separating funnel and wash with water. Add 100-150 ml distillated water onto upper phase that was in the separating funnel and remove water. Repeat wash step 5 times. Add 3-5 drops of Phenol phytalein into a clean erlenmayer flask and titrate lower phase with it. End the washing step when the colour gets pink. Take the phase into balloon joje (200 ml), and bring test portion to 200 ml with diethyl ether. Add 3 spatule tip of sodium sulphate. Remove diethyl ether by a rotary evoporator.Take the remaining test into n-hexane. Filter the test with 0.42 µ fitler paper and take into vials.

HPLC

Inject standart or test solution into HPLC flouresence dedector, at;

Wave lenght ex: 325 em; 480

Flow rate: 1ml/min

Loop; 20 µl

2.3.3. Standart Preparation

Weigh 0.05 g vitamin A and follow same test procedure with test sample. Bring test portion to 200ml. Take 10 ml test portion, and evaporate at rotary evoporator. Bring volume to 100 ml (final concentration: 25 µg/ml).

Standarts;

1- Bring 0.25 ml to 25 ml
2- Bring 0.5 ml to 25 ml
3- Bring 1 ml to 25 ml
4- Bring 2 ml to 25 ml

Firstly give mobil phase to HPLC for 30 min, after give standard sample to HPLC.

At the end samples are analysed in HPLC.

Related Documents

Analytical Methods for Vitamins and Caratonoids in Feed. Animal Nutrition and Health Vitamins and Fine Chemicals Division. Roche 1998.U. Manz and K. Philipp.

AOAC, 2000 Official Method 992.06., AOAC, 2000 Official Method 985.3

FOOD CHEMISTRY ANALYSES (TOTAL SUGAR, INVERT SUGAR, SALT, HMF, TOTAL ACIDITY)

DETERMINATION OF APPARENT REDUCING SUGARS AND APPARENT SUCROSE

1. SCOPE

The method can be applied to all honey samples.

2. DEFINITION

'Apparent reducing sugars' are defined as those sugars which reduce a Fehling's reagent under the conditions specified. 'Apparent sucrose' is defined as 0.95 of the difference in 'apparent reducing sugars' before and after the prescribed hydrolysis procedure.

3. PRINCIPLE

This method is a modification of the Lane and Eynon procedure, involving the reduction of Soxhlet's modification of Fehling's solution by titration at boiling point against a solution of reducing sugars in honey using methylene blue as an internal indicator. The difference in concentrations of invert sugar is multiplied by 0.95 to give the apparent sucrose content.

4. REAGENTS

a) Soxhlet's modification of Fehling's solution:

Solution A: dissolve 69.28 g of Copper sulphate pentahydrate ($CuSO4•5H2O$ in water, MW = 249.71) and make up to 1000 ml. Keep one day before titration.

Solution B: dissolve 346 g sodium potassium tartrate (C 4 H 4 NaO 5 •4H2O, MW 282.23) and 100 g sodium hydroxide (NaOH) with distilled water to 1000 ml. Filter through prepared asbestos.

b) Standard invert sugar solution (10 g/L)

Weight 9.5 g pure sucrose, add 5 ml hydrochloric acid (ca. 36.5 % w/w pure HCl) and dilute with water to about 100 ml. Store this acidified solution for several days at room temperature (ca. 7 days at 12°C to 15°C or 3 days at to 20°C to 25°C) and then dilute to 1000 rnl. (NB acidified 1 % invert sugar remains stable for several months). Neutralize a suitable volume of this solution with 1 M sodium hydroxide solution (40 g/L) immediately before use and dilute to the required concentration (2 g/L) for the standardization.

c) Methylene blue solution

Dissolve 2 g in distilled water and dilute to 1 litre.

d) Alumina cream

Prepare cold satured solution of alum (K_2SO_4 Al_2 $(SO_4)_3$ $\bullet 24H_2O$) in water. Add ammonium

hydroxide with constant stirring until solution is alkaline to litmus, let precipitate settle and wash by decantation with water until wash-water gives only slight test for sulphate with barium chloride solution. Pour off excess water and store residual cream in stoppered bottle.

e) Hydrochloric acid (6.34 M, aqueous), for apparent sucrose only

f) Sodium hydroxide (5 M aqueous), for apparent sucrose only

5. PROCEDURE FOR REDUCING SUGARS

5.1 Preparation of test sample, first procedure

(applicable to honeys which may contain sediment)

1. Transfer an accurately weighed sample of approximately 25 g (W 1) from the homogenized honey to a 100 ml volumetric flask, add 5 ml alumina cream, dilute to volume with water at 20°C and filter.

2. Dilute 10 ml of this solution to 500 ml with distilled water (diluted honey solution).

5.2 Preparation of test sample, second procedure

(honeys with no sediment)

1. Weigh accurately a representative quantity of about 2 g (W 2) of the homogenous honey sample, dissolve in distilled water and dilute to 100 ml in a calibration flask (honey solution)

2. Dilute 50 ml of the honey solution to 100 ml using distilled water (diluted honey solution)

5.3 Standardization of the modified Fehling's Solution

Standardise the modified Fehling's solution A so that exactly 5 ml (pipette), when mixed with approximately 5 ml of Fehling's solution will react completely with 0.050 g invert sugar as 25 ml dilute invert sugar solution (2 g/L).

5.4 Preliminary titration

The total volume of the added reactants at the completion of the reduction titration must be 35 ml. This is made up by the addition of a suitable volume of water before the titration commences. Since the compositional criteria specify that there should be no more than 60 % reducing sugars (calculated as invert sugar), a preliminary titration is necessary to establish the volume of water to be added to a give sample to ensure the reduction is carried out at constant volume. The volume of water to be added is calculated by subtracting the volume of diluted honey solution consumed in the preliminary titration (X ml) from 25 ml.

Pipette 5 ml Fehling's solution A into a 250 ml Erlenmeyer flask and add approximately 5 ml Fehling's solution B. Add 7 ml distilled water, a little powdered pumice or other suitable antibumping agent, followed by about 15 ml diluted honey solution from burette. Heat the cold mixture to boiling over a wire gauze, and maintain moderate ebullition for 2 minutes. Add 0.2 % aqueous methylene blue

solution whilst still boiling and complete the titration within a total boiling time of 3 minutes, by repeated small additions of diluted honey solution used (X ml).

5.5. Titration

Calculate the amount of added water necessary to bring the total volume of the reactants at the completion of the titration to 35 ml by subtracting the preliminary titration (X ml) from 25 ml. Pipette 5 ml Fehling's solution A into 250 ml Erlenmeyer flask and add approximately 5 ml Fehling's solution B.

Add (25-X) ml distilled water, a little powdered pumice or other suitable antibumping agent and, from a burette, all but 1.5 ml of the diluted honey solution volume determined in the preliminary titration. Heat the cold mixture to boiling over a wire gauze and maintain moderate ebullition for 2 minutes. Add 1 ml 0.2 % methylene blue solution whilst still boiling and complete the titration within a total boiling time of 3 minutes by repeated small additions of diluted honey solution until the indicator is decolourized. Note the total volume (Y ml). Duplicate titration should agree within 0.1 ml.

6. PROCEDURE FOR APPARENT SUCROSE

6.1 Sample preparation

Prepare honey sample as in 5.1: dilute 10 ml of this solution to 250 ml distilled water or acc.to 5.2.

Hydrolysis

The honey solution (50 ml) is placed in a graduated flask, together with 25 ml distilled water, heat the test sample to 65 o C over a boiling water bath. The flask is then removed from the eater bath and 10 ml of hydrochloric acid is added. The solution is allowed to cool naturally for 15 minutes, and then brought to 20 o C and neutralized with sodium hydroxide, using litmus paper as indicator, cooled again, and the volume adjusted to 100 ml (diluted honey solution).

Titration

As in Sections 5.4 and 5.5.

7. CALCULATION AND EXPRESSION OF RESULTS

7.1. Reducing sugars

Where the first procedure has been used:

C = (25/W1) *x*(1000/Y)

Where the second procedure has been used:

C = (2/W2) *x*(1000/Y2)

Where C = g invert sugar per 100 g honey

W1:weight (g) of honey sample according to 5.1

W 2: weight (g) of honey sample according to 5.2 Y1:volume (ml) of diluted honey solution consumed in the determination carried out acc. to 5.1 Y2: volume (ml) of diluted honey solution consumed in the determination carried out acc. to 5.2

Notes on the procedure

It is essential to the accuracy and repeatability of the determination that the volume of water necessary to bring the reactant mixture to a total volume of 35 ml be determined for each individual sample; the following table gives typical volumes which may be encountered at the preliminary titration stage for the increment contents of invert sugar shown, assuming the test sample (first procedure) weighs about 25 g or test sample (second procedure) weighs about 2 g.

Invert Sugar content (%)	Volume of distilled water to be added
60	8.3
65	9.6
70	10.7
75	11.6

7.2. Apparent sucrose

Calculate percent invert sugar (g invert sugar per 100 g) after inversion using the appropriate formula as for percent invert sugar before inversion in section 6.1

Apparent sucrose content :(invert sugar per 100 g honey after inversion) minus (sugar content before inversion) x 0.95

The result is expressed as g apparent sucrose/100 g honey

REFERENCES

1. Bogdanov., S. (2002) Harmonanised methods of the International Honey Commission.

DETERMINATION OF FREE ACIDITY

1. SCOPE

The method can be applied to any sample of honey.

2. DEFINITION

The free acidity of honey is the content of all free acids, expressed in milliequivalents /kg honey, determined by this method.

3. PRINCIPLE

The sample is dissolved in water, the pH measured and the solution titrated with 0.1M sodium hydroxide solution to pH 8.30

4. REAGENTS

Distilled, carbon dioxide - free water. Buffer solutions for calibration of the pH meter at pH 4.0, pH 7.0 and 9.0. 0.1M sodium hydroxide solution, accurately standardised (e.g. Titrisol).

5. EQUIPMENT

pH meter, accurate to 0.01 units.Magnetic stirrer.Burette 10 ml, 25 ml or automatic titrator. Beaker, 250 ml

6. PROCEDURE

Calibration of the pH meter

The meter should be calibrated at pH 4.0, 7.0 and 9.0.

Sample preparation:

6.1-Sampling

The sample to be analysed should be representative of the honey lot. All honey samples should beprepared in the following way before analysis.

6.1.1 Liquid or crystallised honey free from extraneous matter.

Homogenize the laboratory sample by stirring thoroughly (at least three minutes). Be careful that as little air as possible is stirred into the honey, especially if the sample is to be used for determination of hydroxymethylfurfural. If the honey is crystallised in a hard and compact mass, it can be previously softened by heating it in stove or thermostatic bath at no more than 40°C

6.1.2 Liquid or crystallized honey containing extraneous matter.

Remove any coarse material, subsequently stir the honey at room temperature and pass through a 0.5 mm sieve. Gently press crystallised honey with a spatula through a 0.5 mm sieve.

6.1.3 Comb honey.

Uncap the comb. Drain the comb through a 0.5 mm sieve without heating in order to separate honey from the comb.

6.2 Determination

Ensure the sample is representative. Dissolve 10 g sample in 75 ml of carbon dioxide-free water in a 250 ml beaker. Stir with the magnetic stirrer, immerse the pH electrodes in the solution and record the pH. Titrate with 0.1M NaOH to pH 8.30 (a steady reading should be obtained within 120 sec of starting the titration; in other words, complete the titration within 2 minutes.). Record the reading to the nearest 0.2ml when using a 10ml burette and to 0.01ml if the automatic titrator has sufficient precision.

7. CALCULATION AND EXPRESSION OF RESULTS

pH - Report to two decimal places.

Free acidity, express as milliequivalents or millimoles acid/kg honey

= ml of 0.1M NaOH x 10. Express the result to one place of decimals.

REFERENCES

1. Bogdanov., S. (2002) Harmonanised methods of the International Honey Commission.

DETERMINATION OF HYDROXYMETHYLFURFURAL AFTER WINKLER

1. SCOPE

The method can be applied to all honey samples.

2. DEFINITION

The method determines the concentration of 5-(hydroxymethyl-)furan-2-carbaldehyde, defined as

the constituents of honey which are capable of combining with barbituric acid and p-toluidine under the conditions of the test.

3. PRINCIPLE

This method describes the determination of hydroxymethylfurfural in honey and is based on the original method of Winkler (1955).

To aliquot parts of a honey solution, solutions of p-toluidine and barbituric acid are added and the resultant colour is measured against a blank in 1-cm cuvettes at 550nm.

4. REAGENTS

p-toluidine-solution

NOTE: p-toluidine is carcinogenic and presents a risk to health. Contact with the reagent should be avoided. Dissolve 10.0 g p-toluidine in 50 ml 2-propanol by gently warming on a water bath. Transfer with a few ml of 2-propanol to a 100 ml volumetric flask and mix with 10.0 ml glacial acetic acid. After cooling to ambient temperature, fill to volume with 2-propanol. Store in the dark for at least 24 hours before use. Discard after three days or if there is undue coloration.

Barbituric acid solution.

Transfer 500 mg barbituric acid as quickly as possible to a 100 ml volumetric flask with about 70 ml water. Dissolve by warming the stoppered flask gently on a water bath. Cool to ambient temperature and dilute to volume.

Carrez solution I: dissolve 15 g of potassium hexacyanoferrate(II), K4Fe(CN)6□3H2O in water and make up to 100 ml.

Carrez solution II: dissolve 30 g of zinc acetate, Zn(CH3.COO)2.2H2O in water and dilute to 100 ml with water.

5. EQUIPMENT

Spectrophotometer for measuring absorbance at 550 nm. 1 cm cells. Volumetric flasks, 50 and 100 ml. Test tubes. Beaker. Filter paper, analytical grade.

6. PROCEDURE

Sample preparation

Sample preparation:

6.1 Sampling

The sample to be analysed should be representative of the honey lot. All honey samples should beprepared in the following way before analysis.

6.1.1 Liquid or crystallised honey free from extraneous matter.

Homogenize the laboratory sample by stirring thoroughly (at least three minutes). Be careful that as little air as possible is stirred into the honey, especially if the sample is to be used for determination of hydroxymethylfurfural.

6.1.2 Liquid or crystallized honey containing extraneous matter.

Remove any coarse material, subsequently stir the honey at room temperature and pass through a 0.5 mm sieve. Gently press crystallised honey with a spatula through a 0.5 mm sieve.

6.1.3 Comb honey.

Uncap the comb. Drain the comb through a 0.5 mm sieve without heating in order to separate honey from the comb.,

6.2 THE METHODS.

6.2.1 Preparation of the sample solution

Weigh about 10g of honey to the nearest mg. Dissolve in about 20 ml water and quantitatively transfer to a 50 ml volumetric flask. Add 1 ml of Carrez I, shake well, add 1 ml of Carrez II, shake once more, dilute to volume with water and mix once more. A drop of ethanol prevents possible foaming. Filter the solution through filter paper. Discard the first 10 ml of the filtrate. Complete the rest of the analysis immediately. In the case of ***very clear samples***, clarification with Carrez' reagents is not necessary.

6.2.2 Determination

Pipette 2.0 ml of the sample solution to each of two tubes and add 5.0 ml p-toluidine solution to

both. Add 1.0 ml of water to one tube (blank value) and 1.0 ml of barbituric acid solution to the other with gentle shaking. Carry out without delay and complete in 1 - 2 minutes. Measure the absorbance of the sample against the blank as soon as the colour intensity has reached a maximum (3 – 4 minutes after adding the barbituric acid solution), using 1cm cells at 550nm.

7. CALCULATION AND EXPRESSION OF RESULTS

The content of HMF is calculated as follows:

HMF=(192xAx10)/(Weight of honey in ... grams)

Where

A = Absorbance,

192 = Factor for dilution and extinction coefficient

Express results in mg/kg to 1 decimal place

REFERENCES

1. Bogdanov., S. (2002) Harmonanised methods of the International Honey Commission.

DETERMINATION OF SALT IN CHEESE

1. SCOPE

The method can be applied to food samples.

2-DEFINITION:

In the Mohr method, the chloride is titrated directly by a standard silver nitrate solution using potassium chromate as an indicator.

3. PRINCIPLE

This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

$Ag^{+}_{(aq)} + Cl^{-}_{(aq)} \rightarrow AgCl_{(s)}$

The indicator used is dilute potassium chromate solution. When all the chloride ions have reacted, any excess silver nitrate added will react with chromate ions to form a red-brown precipitate of silver chromate. This procedure is known as Mohr's method.

$2Ag^{+}_{(aq)} + CrO_4^{2-}{}_{(aq)} \rightarrow Ag_2CrO_{4(s)}$

The procedure described here for seawater may also be applied to water samples from various other sources to determine the relative concentrations of chloride ions in, e.g., stream water, river water, estuary water.

3. REGEANTS

Silver nitrate solution:(0.1 mo/l L): If possible, dry 5 g of AgNO3 for 2 hours at 100°C and allow to cool. Accurately weigh about 4.25 g of solid AgNO3 and dissolve it in 250 mL of distilled water in a conical flask. Store the solution in a brown bottle.
Potassium chromate indicator solution (approximately 0.25 mol/L) :Dissolve 1 g of K2CrO4 dissolved in 20 mL distilled water).

4. EQUIPMENT

Buret and stand

10 and 20 mL pipettes

100 mL volumetric flask

250 mL conical flasks10 mL and

100 mL measuring cylinders

6. PROCEDURE

1. Dilute samples by pipetting a 20 mL sample into a 100 mL volumetric flask and making it up to the mark with distilled water.

2. Pipette a 10 mL aliquot of diluted seawater into a conical flask and add about 50 mL distilled water and 1 mL of chromate indicator.

3. Titrate the sample with 0.1 mo/ L silver nitrate solution. Although the silver chloride that forms is a white precipitate, the chromate indicator initially gives the cloudy solution a faint lemon-yellow colour. The endpoint of the titration is identified as the first appearance of a red-brown colour of silver chromate.

4. Repeat the titration with further aliquots of diluted samples until concordant results (titres agreeing within 0.1 mL) are obtained and a stronger red-brown colour results.

NB: The titration should be stopped when the first trace of red-brown colour is observed. Using an incompletely titrated reference flask for comparison is a helpful way to identify the first appearance of red-brown colouration.

7. CALCULATION AND EXPRESSION OF RESULTS

1. Determine the average volume of silver nitrate used from your concordant titres.

2. Calculate the moles of silver nitrate reacting.

3. Use the following reaction equation to determine the moles of chloride ions reacting.$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$4.

Calculate the concentration of chloride ions in the diluted samples

5. Calculate the concentration of chloride ions in the original undiluted samples.

6. Calculate the concentration of sodium chloride in the food in mol/L, g/L and g/100 mL (%).

Additional Notes

1. Silver nitrate solution will stain clothes and skin. Any spills should be rinsed with water immediately.

2. Residues containing silver ions are usually saved for later recovery of silver metal. Check this with your teacher.

3. The Mohr titration should be carried out under conditions of pH 6.5 – 9. At higher pH silver ions may be removed by precipitation with hydroxide ions, and at low pH chromate ions may be removed by an acid-base reaction to form hydrogen chromate ions or dichromate ions, affecting the accuracy of the end point.

4. It is a good idea to first carry out a "rough" titration in order to become familiar with the colour change at the end point.

5. The Mohr titration is sensitive to the presence of both chloride and bromide ions in solution and will not be too accurate when there is a significant concentration of bromide present as well as the chloride. However, in most cases, such as the sample, the bromide concentration will be negligible. For this reason, the method can also be used to determine either the total concentration of chloride and bromide in solution, or the concentration of bromide when the chloride concentration is known to be negligible.

REFERENCES

http://www.outreach.canterbury.ac.nz/chemistry/documents/chloride_mohr.pdf.

MINERAL AND HEAVY METAL ANALYSES (Ca, Zn, Pb, Cd)

DETERMINATION OF ZINC AND CALCIUM IN FOOD SAMPLES BY FLAME-AAS

Reagents

- Zinc (standart, 1000 mg/L)
- Calcium (standart, 1000 mg/L)
- % 65 nitric acid (HNO_3),
- % 35 hydrogen perokside (H_2O_2),
- % 37 hydrochloric acid (HCl)
- Multielement standart (Merck 109493)
- Deiyonize water, Milli-Q Gradient A10 (18 MΩ.cm)

Peraperation of Stock Standarts

Zinc stock standart 1 (10mg/L): take 1 ml solution from zinc stok standart solution (1000mg/L) to 100 ml volumetric flask and dilute with 1% nitric acid solution.

Calcium stock Standart 1 (100 mg/L) take 2.5 ml solution from calcium stok standart solution (1000mg/L) to 25 ml volumetric flask and dilute with 1% nitric acid solution.

Working Standarts: take 1.0 ml, 2.0 ml, 4.0 ml from stock standart solutions 1 (10 mg/L) to three different 50 ml volumetrik flask and dilute with 1% nitric acid solution.

	Zinc	Calcium
Working standart 1:	(0.2 mg/L)	(2 mg/L)
Working standart 2:	(0.4 mg /L)	(4 mg/L)
Working standart 3:	(0.8 mg /L)	(8 mg/L)

Peraperation of Samples

Weigh approximately 0.5 g samples to teflon sample vassels. Add 6 ml % 65 nitrik asit (HNO_3) and 1 ml %30 hydrogen perokside. Close the sample vassels and place the sample rotor into the microwave digestion system and start the oven program. After digestion. Sample solution is transfered to 50 ml volumetric flask by rinsing sample vassel with deionized water.

Measurement of Zinc with AAS

Place the zinc lamb to AAS machine and turn on the lamb. Fill the sample info file. Run the blank solution, working standarts and samples. Measurement wavelength is 213.9 nm and the slit is 0.7

Measurement of Calcium with AAS

Place the Calcium lamb to AAS machine and turn on the lamb. Fill the sample info file. Run the blank solution, working standarts and samples. Measurement wavelength is 422.7 nm and the slit is 0.7

DETERMINATION OF LEAD AND CADMIUM IN FOOD SAMPLES BY GF-AAS

Reagents

- Lead (standart, 1000 mg/L)
- Cadmium (standart, 1000 mg/L)
- % 65 nitric acid (HNO_3),
- % 35 hydrogen perokside (H_2O_2),
- % 37 hydrochloric acid (HCl)
- Multielement standart (Merck 109493)
- Deiyonize Water, Milli-Q Gradient A10 (18 MΩ.cm)

Peraperation of Stock Standarts

Lead stock standart 1 (2mg/L): take 200 ul solution from lead stok standart solution (1000mg/L) to 50 ml volumetric flask and dilute with 1% nitric acid solution.

Cadmium stock Standart 1 (1 mg/L) take 100 ul solution from cadmium stok standart solution (1000mg/L) to 25 ml volumetric flask and dilute with 1% nitric acid solution.

Working Standarts: take 50 ul, 100 ul ml, 200 ul from stock standart solutions 1 (10 mg/L) to three different 50 ml volumetrik flask and dilute with 1% nitric acid solution.

	Cadmium	Lead
Working standart 1:	(1 ug/L)	(2 ug/L)
Working standart 2:	(2 ug /L)	(4 ug /L)
Working standart 3:	(4 ug /L)	(8 ug /L)

Peraperation of Samples

Weigh approximately 0.5 g samples to teflon sample vassels. Add 6 ml % 65 nitrik asit (HNO_3) and 1 ml %30 hydrogen perokside. Close the sample vassels and place the sample rotor into the microwave digestion system and start the oven program. After digestion. Sample solution is transfered to 50 ml volumetric flask by rinsing sample vassel with deionized water.

Measurement of lead with AAS

Place the Lead lamb to AAS machine and turn on the lamb. Fill the sample info file. Run the blank solution, working standarts and samples. Measurement wavelength is 283.3 nm and the slit is 0.7

Measurement of Cadmium with AAS

Place the cadmium lamb to AAS machine and turn on the lamb. Fill the sample info file. Run the blank solution, working standarts and samples. Measurement wavelength is 193.7 nm and the slit is 0.7

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SUGAR COMPONENTS, AFLATOXINS AND DEOXYNIVALENOL ANALYSES BY HPLC

SUGAR COMPONENTS (GLUCOSE, FRUCTOSE, SUCROSE) İN HONEY

Sample Extraction	Weigh 5g of sample into erlenmayer
	Disolve the honey in 40ml of distilled water by mixing
	Add 25ml methanol into a 100mL volumetric flask
	Transfer honey solution into the 100mL volumetric flask which contains 25mL methanol and shake by hand
	Fill the volumetric flask by distilled water up to 100mL and shake by hand
HPLC	Filter the solution through the 0.45µm membran filter
	Inject 10µL of filtred sample solution onto the HPLC

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AFLATOKSIN B_1, B_2, G_1, G_2 IN PEANUT

Sample Extraction	Weigh 50g of ground sample into the blendor jar
	Add 4g of NaCl
	Add 250ml methanol:distilled water (6:4, v/v) solution (150 ml methanol + 100ml distilled water)
	Blend it for 2 minutes in high speed
	Filter 25-50 ml of solution through the filter paper (Whatman No.4)
Capture of Aflatoxins	Transfer 5 mL of filtrate into 100ml erlenmayer flask, add 45 ml PBS (Phosphate Buffered Saline) into the flask. Shake the flask by hand
	Transfer the sample solution into the glass syringe barrel for passage through the immunoaffinity column (Immunoaffinity columns should be at ambient temperature before use. Remove the cap from the top of the column, cut off the sealed end and replace. Attach the column to the 10 ml glass syringe barrel)
	Remove the bottom cap from the column and pass the sample solution through the immunoaffinity column at a flow rate of 2-3ml/min (approx. 1 drop per second)
Washing	Add 10 ml of distilled water into the glass barrel and wash the column by passing the distilled water through the immunoaffinity column at a flow rate of 5ml/min (approx 2 drops per second). (Repeat this step 2 times)
Elution	Place an amber vial under the column
	Pipette 1 ml of HPLC grade methanol into the glass barrel
	Elute aflatoxins from the column into the glass vial by slowly passing the methanol (eluant) through the column at a flow rate of 1 drop per second (Back flushing – reversing the direction of flow with eluant 3 times is recommended)
	Pipette 1 ml of distilled water into the glass barrel, pass through the column and collect in the amber vial
HPLC	Inject 100µl of sample solution onto the HPLC

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DEOXYNIVALENOL IN MAİZE

Sample Extraction	Weigh 25g of ground sample into the blendor jar
	Add 200ml distilled water
	Blend it for 2 minutes in high speed
	Filter 25-50 ml of solution through the filter paper (Whatman No.4). (Centrifuge the solution at 4000rpm for 5 minutes, if the turbidity of the solution is too high)
Capture of Deoxynivalenol	Transfer 2 ml of filtrate into the glass syringe barrel for passage through the immunoaffinity column (Immunoaffinity columns should be at ambient temperature before use. Remove the cap from the top of the column, cut off the sealed end and replace. Attach the column to the 10 ml glass syringe barrel)
	Remove the bottom cap from the column and pass the sample solution through the immunoaffinity column at a flow rate of 2-3ml/min (approx. 1 drop per second)
Washing	Add 10 ml of distilled water into the glass barrel and wash the column by passing the distilled water solution through the immunoaffinity column at a flow rate of 5ml/min (approx 2 drops per second)
Elution	Place an amber vial under the column
	Pipette 1.5 ml of HPLC grade methanol into the glass barrel
	Elute deoxynivalenol from the column into the glass vial by slowly passing the methanol solution (eluant) through the column at a flow rate of 1 drop per second (Back flushing – reversing the direction of flow with eluant 3 times is recommended)
HPLC	Evaporate the sample solution collected in the amber vial at 40°C under nitrogen until dryness.
	Dilute the residue by 1 mL mobile phase (acetonitrile:methanol:distilled water, 3:3:94, v/v)
	Inject 100µl of sample solution onto the HPLC

OIL/FATS ANALYSES (FREE FATTY ACID, PEROXIDE, FATTY ACID COMPOSITION BY USING GC

Preparation of Fatty acid Methyl Esters

Reagents

Heptane

Sodium Sulphate, anhydrous

Sodium Hydroxide, approximately 0.5 N methanolic solution

37 mixture of FAME standarts

Preparation of Samples

Weigh approximately 0.1 g of oil samples to glass tubes.(If the samples are not in oil form, they have to be treated with ether) .Add 5 ml of heptane and 0.5 ml of methanolic sodium hydroxide solution to sample, vortex about 2 minutes. Wait about two minutes for the sample to become clear, then take 1 ml from the heptane phase to the vial.

Measurement of the samples by GC

Firstly the 37 mixture of FAME standart is run in order to get the retention times. Then run the samples.

PEROXIDE VALUE

Reagents

Acetic acid-chloroform solution: Mix 3 parts by volume of glacial acetic acid, with 2 parts by volume of chloroform.

Potassium Iodide Solution : Saturated solution of KI.

Sodium Thiosulphate solution:0.1 N

Starch indicator solution

Procedure for Fats and Oils;

Weigh approximately 5 g of sample into a 250 ml Erlenmeyer flask and then add 30 ml of the acetic acid-chloroform solution. Swirl the flask until the sample is dissolved. Add 0.5 ml of potassium iodide.

Allow the solution to stand with shaking for exactyl 1 minute then add 30 ml of distilled water.

Titrate with 0.1 N sodium thiosulphate adding it gradually. Continue the titration until the yellow color almostly dissappeared. Add ca. 0.5 ml of starch indicator solution. Continue the titration, with thiosulphate until the blue color disappears.

Conduct the blank determination of reagents daily.

Calculation: (S-B)N*1000/weight of the sample

FREE FATTY ACIDS

Reagents

Ethyl alcohol

Phenolphthalein indicator solution

Sodium hydroxide solution

Procedure:

Samples must be well mixed and entirely liquid before weighing into an erlenmeyer. 5 grams of sample is weighed and 50 ml of neutralized alcohol is added to the sample. (alcohol is neutralized with 2 ml of indicator).

Titrate with alkali till the appearence of te permanent pink color. The color must persst 30 seconds.

Calculation: The percentage of free fatty acids in most types of fats and oils is calculated as oleic acid..

Free fatty acid as oleic%= ml of alkali*N*28.2/W