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SYRIAN ARAB REPUBLIC

Final report: Medical stores

Acknowledgements

The Expert is greatly indebted to the many people who collaborated with him during the implementation of the activities described in this report and who offered advice and assistance. Special acknowledgements are due to:

Dr. Y. Kassab, Program Officer, UNDP
Dr. M.A. Shaaban, Project Manager, UNIDO
Dr. Z. Sawaf, Director General, Industrial Research and Development Center
Dr. D. Köttgen, Mechanical Production Engineer, Expert, UNIDO
Dr. M. Parkany, Expert in Instrumental Methods of Chemical Analysis, UNIDO
Mr. M.Todorovic, Food Canning Technologist, UNIDO
Mr. A. Louis, Engineer, Union of Food Industries
Dr. Z. Haj-Mustafa, Production Manager, Camelia Biscuit and Chocolate Factory.
Mr. H. Moussally, Production Manager, Modern Conserves and Agricultural Industries Corporation.
Mr. M. Awameh, Head of Food Processing Station, Karasta Institute

The Expert also wants to express his gratitude to his counterparts Miss Tamador Hakki and Mrs. Abeer Khasnadar for a very good cooperation during his stay.

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1. SUMMARY

The report is the final report from Food Industries Expert, Oscar Kvaale, for his assignment to the Industrial Research and Development Center (Project DP/SYR/72/006) from November 1974 to November 1975.

The report deals with findings and recommendations regarding the food laboratories at the Center and the Syrian food industries. The different activities are described as to counterpart relations, working facilities and program of industrial assistance. For some of the activities, special technical reports have been prepared. In some cases, these reports form a substantial part of the recommendations and they are, therefore, appended to this report.

Special recommendations are given to the further policy in methods of food analysis at the Center and to measurements for implementation of the recommendations given. The prospects of further UNIDO assistance to the food laboratories at the Center and of an extended UNIDO program of assistance to the food industries in Syria are also discussed.

2. INTRODUCTION

The expert was appointed as Food Industries Expert on a one year assignment from 10th November 1974. His terms of reference were:

1. Cooperate with the Union of Food Industries in assessing the general condition of the existing food processing plants and identify technical, quality control and standardization problems affecting their operation;
2. Provide technical advisory services to these enterprises for the solution of their problems;
3. Formulate recommendations aimed at the improvement of the plants' efficiency;
4. Undertake a work programme at the laboratories of the Center aimed at the improvement of the processes and better utilization of the local fruits and vegetables;
5. Participate in introduction of research results into industry (commercial development);
6. Take part in the establishment and/or further development of quality control systems in industrial standards;
7. Train local personnel in the above field;
8. Cooperate with other experts in undertaking techno-economic feasibility studies, to identify feasible projects in the food industries field.

The Expert was briefed in Vienna 10-12 November 1974 and arrived in Damascus 13th November 1974. Here, he was introduced by Dr. Shaaban, Project Manager, and Dr. Kassab, Project Officer, to Mr. S. Habib Ahmed, Resident Representative of UNDP, to Dr. Sawaf, General Director of the Center, and to counterparts for his assignment.

Following a briefing on the general situation at the Organic and Food Laboratories Department at the Center and on the general problems facing the food industries in Syria, the Project Manager, the Director General of the Center, and the Expert, decided upon the following schedule for his work:

- Beginning November 1974: Ordering of equipment for the Chemical and Microbiological laboratories at the Center.
- Beginning December 1974: Training of counterparts
- Beginning January 1975 : Visits to industrial companies
- Beginning February 1975: Special program of assistance to the food industry and special training in methods of food analysis at the Center.

At the time of the Expert's departure a number of the tasks expressed in his terms of reference have been completed. However, these terms cover a large field and comprise in their broadest interpretation most aspects of food technology. A one year assignment is a too short time to prepare detailed studies of all problems here involved. In many cases counterparts will have to finalise started activities, and recommendations for such extensions are given.

3. ACTIVITIES

According to his terms of reference, the activities of the Expert naturally fall in two parts:

- Work program inside the Industrial Research and Development Center (IRDC), and
- Work program outside IRDC to provide advisory services to the Syrian food industries.

The two fields of activities will here be mentioned separately.

3.1 Activities of assistance inside IRDC

3.1.1 Present stage of food laboratories

On the Expert's arrival, the laboratories intended for food analysis were almost without any equipment. Equipment for general chemical analysis was ordered, and during the assignment of the Expert most of it has arrived and has been installed. Instruments for special and advanced food analysis (gas chromatograph for analysis of fatty acids, spectrophotometer for determination of food preservatives, atomic absorption spectrophotometer for detection of mercury in food) were also present. By assistance of the Expert in Instrumental Methods of Chemical Analysis, Dr. M. Parkany, these instruments are now put in a working condition. However, the spectrophotometer and the atomic absorption spectrophotometer still lack accessories for food analysis. Equipment for microbiological food analysis was not ordered at the Expert's arrival and an order for suitable equipment for such purposes was prepared and sent to UNIDO in November 1974. At the end of the Expert's assignment, no item for this order has arrived. These circumstances have hampered the activities in food microbiology to a considerable degree.

The laboratory furnishings (dishes, sinks, fume hoods, etc.) make the laboratories well suited for microbiological and chemical food analysis. Facilities for washing and cleaning of glassware and other

laboratory equipment are, however, not provided for in a satisfactory manner. These operations are assumed to be carried out in sinks at the end of each laboratory desk. This is not in accordance with the sanitation conditions which have to be established in a laboratory for microbiological analysis.

3.1.2 Counterpart situation

When arriving to the IRDC the Expert was introduced to Miss Tamador Hakki and Mrs. Abeer Khaznadar as his counterparts. They are both well qualified chemists.

Miss Hakki has a basic education in chemistry from the University of Damascus and a very valuable post education in gas chromatographic and infrared spectroscopic food analysis of organic chemicals from studies in France and England. Besides, she has administrative experience from holding posts as Technical Director in the Union of Food Industries and as Head of the Department for Chemical Industries in the Ministry of Industry. Her knowledge of the Syrian food industries, is, therefore, very detailed.

Mrs. Khaznadar is educated in applied chemistry from the University of Damascus in 1972. She shows a sincere interest in laboratory food analysis and has a theoretical background that is very suitable for work in this field.

In the structure plan for the IRDC, the Chemical Section has four departments:

- Organic and Food Laboratories Department
- Inorganic Laboratories Department
- Special Laboratories Department
- Textile Department

The Organic and Food Laboratories Department is again divided in three divisions:

- Microbiological Division
- Chemical Division
- Special Instrumentation Division

Miss Hakki is appointed as Head of the Organic & Food Laboratories Department. She also holds a post as Assistant Head of the Chemical Laboratories Section. Up to present no final decision has been taken as to the manning of the different divisions at the food department. Mrs. Khazmadar and another chemist (M. Sawan) are performing the daily laboratory work irrespective to which division it may belong. One post as chemist, two as technical assistants and one as technical supervisor are still vacant in the Organic and Food Laboratories Department.

3.1.3 Training of counterparts

On the arrival of the Expert none of the counterparts had any extended experience in the principles of microbiological food analysis or in industrial food microbiology. Due to the equipment situation, very little advice and training could be given in practical microbiological laboratory analysis during the Expert's assignment. However, by supplementing of the IRDC library with suitable literature in this field, a background was established for a more theoretical training program. By lecturing and group working the Expert has enlightened the most important fields in food microbiology and in methods of microbiological analysis of food. The topics covered by special lectures go forth from Appendix 1. In fields where numerous methods of analysis exist and where a general view of the situation is not to find in the available literature, such surveys have been made. The surveys, appended to this report, are as follows:

Survey of Methods for the Enumeration of Microorganisms - Appendix 2.

Survey of Laboratory Methods for Detecting Spoilage in Food and Food Products. Appendix 3

Survey of Methods for Sanitation Control in Food Industries. Appendix 4.

Survey of Methods for Determination of Water Activity in Food. Appendix 5.

Survey of Methods for Determination of Fat in Solid Foods. Appendix 6.

Survey of Methods for the Determination of Texture in Foods. Appendix 7.

3.1.4 Preparing food standards

Up to now only a very few food standards have been approved by the Syrian General Standard Committee. Official standards exist for drinking water, beverage water and for some types of biscuits. Draft standards exist for more products, including chocolate and refined sugar, and a Technical Committee for Food Standard is, at present, working out drafts for still more food products.

Upon request from the IRDC, the Expert has prepared English language draft standards for some canned meat and fish products imported into Syria. Recommendations are given to Syrian standards for canned tuna, sardines, corned beef, luncheon meat and chopped meat. Special advice is also given for regulations for the content of mercury in fish products, as to acceptable limits for mercury in fish, sample preparation and basic procedures for the determination of mercury in sub-micro quantities. All of the suggested standards are in good accordance with the FAO/WHO Codex Alimentarius proposals for international standards for these particular products.

3.2 Activities of assistance outside the Industrial Research and Development Center

3.2.1 The total industrial production from state owned Syrian enterprises, amounted in 1973 to 898 million Syrian Pounds. Of this sum the Union of Textiles Industries contributed with 396 million Syrian Pounds, the Union of Food Industries 282 million Syrian Pounds and the Union of Engineering and Chemical Industries 220 million Syrian Pounds [1]. Thus,

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1) Antoine Guiné: 1975. Syria. Editions Delroisse, Boulogne, France

The food industries are the second largest Syrian industrial branch.

The food industries of Syria are very diversified, ameliorating a wide variety of raw materials. The main industries can be listed in the following categories: food canning, vegetable oil processing, sugar refining, dairy product manufacturing, biscuit and chocolate producing. The production volume of these industries go forth of the following statistics (figures lacking for the dairy industry):

Production of main food manufacturing industries 1970-1973 in 1000 tons

	1970	1971	1972	1973
Canning industry	5.5	5.7	5.8	4.7
Veg. oil industry	25.1	26.2	27.5	28.7
Sugar industry	123.7	130.4	137.2	142.0
Biscuit industry	2.3	2.3	2.4	2.8
Chocolate industry	0.9	0.9	1.0	1.0

Source: Syrian Central Bureau of Statistics:
Statistical Abstract 1974. C.B.S. Printing Press

The food industries are partly private owned and partly governmental enterprises administrated by the Union of Food Industries. The following table gives a picture of the importance of the state owned factories:

Per cent production from state owned food industries

	1970	1971	1972	1973
Canning industry	89	87	94	82
Veg. oil industry	98	99	99.9	99.9
Sugar industry	99.9	99.9	99.9	99.9
Biscuit industry	91	82	83	89
Chocolate industry	11	12	19	16

Source: as above.

The vegetable oil and the sugar industries are fully nationalized while about 10% of the canning and the biscuit producing industries are on private hands. Up to 1974 the Food Union shared only about 15% of the chocolate production. In the years 1970-1973 the proportion between state owned and private enterprises was relatively stable.

The Union of Food Industries has established the following companies for its food production:

- The Modern Conserves and Agricultural Industries Corporation
- The Syrian Industrial Company for Vegetable Oil
- The Arab Industrial Company for Oils and Soup
- Hama Oil Company
- Lattakia Oil Company
- The Syrian Arab Company for Dairy Products
- Al Chark Company for Food Products
- Damascus Company for Food Products
- Onion and Vegetable Dehydration Factory
- The Syrian Company for Biscuits and Chocolate
- The Syrian Sugar and Agricultural Product Company

The geographical sites of the factories belonging to these companies are shown on a map attached as Appendix 8.

According to statistics investments in the state owned food industries amounted to:

1970 - 160.6 million Syrian Pounds
1971 - 151.5 million Syrian Pounds
1972 - 116.9 million Syrian Pounds

The sugar industry has got the biggest share of this capital (38%), then the vegetable oil industry (26%) and the canning industry(9%).

In 1974 and 1975 contracts have been signed by the Food Union for several new food industries projects, comprising three canning factories (at Idlib, Hasakeh and Deir El Zoor), one macarony factory at Deraa and three sugar factories in the Hasakeh-Deib El Zoor region. Further plans involve one grapefruit processing plant and one milk sterilizing plant in Homs, one pea processing line in Mserib and five milk processing factories in the Euphrates region.

3.2.2 Assistance to the food canning industry

The food canning industry of Syria is entirely a fruit and vegetable industry. A great diversity of products are produced, the most important ones being tomato paste, apricot jam, canned peas, green beans, broad beans and artichokes.

Together with counterparts at the IRDC and with UNIDO Food Canning Technologist Mr. M. Todorovic visits were paid to all canning plants belonging to the Union of Food Industries. The findings from these visits are detailed in the final report from Mr. Todorovic's assignment.[1] The two experts, after having made an assessment of the general conditions of production and quality control, agreed upon a further cooperation of work according to following guidelines:

The Food Canning Technologist should, above all, concentrate his activities on technical aspects of assessing methods of process control, the accuracy and efficiency of the production machinery and the machine operators and to try to minimize the present frequency of swollen cans.

The Food Industries Expert should try to establish and get into function a system of in plant quality control for the canning industry and advise on suitable methods for process and product control and measurements to be taken for securing a good manufacturing practice in the canning industry.

The Damascus factory No. 1, Modern Conserves & Agricultural Industries Corporation, was found to possess most of the equipment necessary to perform an adequate internal quality control. The equipment was, however, stowed away and out

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1) Food Canning Technologist Mr. Milenko Todorovic: Technical Consultations for the Canned Fruit and Vegetable Industry, Syrian Arab Republic (IS/SYR/72/028/11-02-06). Final Report, UNIDO 1975.

of use, and nobody seemed to have a sufficient knowledge of its use. The quality control performed was almost negligible.

The Expert made a proper registration of all laboratory equipment present and recommended supplementary equipment necessary to perform an adequate quality control. Recommendations were also given for proper installation and maintenance of the laboratory equipment. He also prepared a recommended programme of in plant quality control and also a manual of laboratory methods for performing the control recommended, together with suitable record forms. A summary of this programme is attached to this report as Appendix 9. The program was appended in extenso to the Mid Term Report from the Expert. Through Project Manager and General Director IRDC it was presented to the Ministry of Industry and to the Union of Food Industries.

Work was started to initiate a training of local personnel in the use of the methods of quality control and in the use of existing laboratory equipment. In this connection, the counterparts of the Expert have started translation of parts of the quality control program into Arabic. During all this work, Mr. Hussain Moussally (Production Manager of the Modern Conserves & Agricultural Industries Corporation and counterpart to Mr. Todorovic) was an interested and most valuable contact person. During the Summer 1975 Mr. Moussally was transferred to a post outside the canning corporation. Temporarily this has hampered the implementation of the quality control program considerably.

During a visit to the canning factory at Jable, the management expressed ideas of seeking a production program in better accordance with raw materials locally available. In this connection the IRDC was asked for advice in production of orange marmalade. Upon this request, the Expert and his counterparts has prepared recommendations for such a production, as to suggestions for recipes, production procedures and control measurements.

The Management of Jable Canning Factory has uttered interest in

starting production experiments according to these principles when the orange season starts (October/November).

The general safety conditions are not satisfactory in any of the canning factories. The autoclaves for sterilizing of cans are badly maintained and are lacking most of the necessary instruments for control of pressure, temperature and time. Moreover, several autoclaves are not equipped with safety valves, and the existing safety valves are often rusty and apparently not in a good functioning state. This represents a potential danger for the operators and for other workers in the factory.

The management has strongly been recommended to adjust these faults and to equip the autoclaves in accordance with instructions given (see also Final Report from Mr. Todorovic)

Still the safety conditions are alarming. The autoclaves are old and are looking strained from wear and tear. A program of hydrostatic pressure tests should be run for each autoclave, and the Expert has suggested that the IRDC should initiate such a control program.

3.2.3 Assistance to the biscuit and chocolate industry

Together with counterparts visits were paid to the Biscuits and Chocolate Factory, Aleppo (Al-Chark Company for Food Products), the Camelia Biscuit and Chocolate Factory, Damascus (Damascus Company for Food Products) and the Ghraoui Biscuits and Chocolate Factory (the Syrian Company for Biscuits and Chocolates). In all of these factories the biscuit production is highly mechanised up to the process of biscuit filling and biscuit packaging. These operations are mainly done by hand, requiring a great number of female workers. The biscuit ovens run continuously and automatically and operate usually up to the installed capacities. Lack of proper instructions for running and maintenance has, however, in some cases (Camelia Biscuit and Chocolate Factory) caused heavy breakdown and damage in relatively new installations.

There have been some customers complaints about the quality of the biscuits, especially to the quality of the fat in use. For the bulk of the production a locally hydrogenerated vegetable

fat (vegetaline) is used as the fat component. This product sometimes gives biscuits with a propensity of going rancid. The management is aware of this problem, but so far it has been a national policy to use the locally produced hydrogenated fats, and so far the quality of this product has not been of a sufficient quality. The problem will be further dealt with later in this report.

The Federation of Food Industry is, as previously mentioned, only about 1% of the total Norwegian chocolate production. At present this production is at a standstill while the federation organisations are trying to sell out the stock production of 10%. There are two main reasons for this: the price of the products is too high and the quality is too low.

To reduce the product price some companies have substituted the cocoa butter with different hydrogenated vegetable fats. However, the companies are not satisfied with the quality of the chocolate made from this fat, and the price difference between cocoa butter has not been big enough to have a sufficient positive effect on the price of the final product.

Upon request from the management of the Oslo Biscuits and Chocolate Factory, the Expert and his counterparts, in cooperation with the factory technical management, have performed a series of experiments using different types of hydrogenated fats and different types of recipes to substitute for use of cocoa butter in chocolate. These experiments have included pilot plant trials both at the INBO and in the factory, organoleptic evaluations of the experimental products and physical-chemical analysis of the different fats in use.

Preliminary experimental results show that an acceptable chocolate can be produced both from a hydrogenated palm kern oil product (Crocklam, Holland) and a hydrogenated marine oil product (Sandaril, Norway). In both cases can the product be given a consistency hard enough not to melt under practical Norwegian commercial conditions. The melting point is, however, not as sharp as with cocoa butter and the chocolate is not likely to melt rapidly in the mouth. The aromatic characteristics should be improved and will have to be adjusted.

3.2.4 Assistance to the vegetable oil industry

Together with counterparts, the industrial companies for production of vegetable oils in Aleppo and Homs were visited and different aspects of assistance from the IRDC were discussed.

The main product for the Syrian vegetable oil industry is cotton seed oil extracted from locally produced cotton seed. Besides, sesam and sunflower oils are produced, and the factory in Homs is also producing coconut oil from imported raw materials. This industry is almost fully nationalized (99%), the remaining private sector is consisting of a number of small factories producing olive oil.

In most of the factories the equipment is of a rather old date requiring more than normal maintenance service. For this reason, the plants often operate below their capacities. A shortage of raw materials is also in some cases the reason for suboptimal production volumes.

Most factories have lines for production of crude oils and for refining of oils. The factory in Aleppo (the Syrian Industrial Company for Vegetable Oils) and in Homs (the Syrian Sugar and Agricultural Products Company) also have production facilities for hydrogenation of cotton seed oil. The hydrogenation and the deodorization processes are, however, not complete enough to make a product which can compete with other imported hydrogenated vegetable oil products.

Upon requests from the factories, the IRDC have carried out gas chromatographic analysis of samples from refined cotton seed oil and from the hydrogenated product "Vegetamine". The technical management has also been made familiar with the principles of hydrogenation of vegetable oils as described in a recent UNIDO publication. [1]

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1) UNIDO, Vienna: The Hydrogenation of Vegetable Oils and the Production of Vegetable Ghee. ID/124, UN Publication Sales No. E.74.II.B.7

Recently the Aleppo factory has purchased a new equipment assembly for hydrogenation of cotton seed oil. The machinery has arrived to the factory, but has, for some reason or another, remained uninstalled up to present. By using this equipment good conditions should exist for producing a first class hydrogenated vegerable oil.

3.2.5 Assistance to the onion dehydration industry

The Onion and Vegetable Dehydration Factory, Salamiyeh, was visited, also in company with counterparts.

The factory is a modern set up with continuous production lines for the production of dried onions. Because of a highly specified production, dehydration of one product only, the production has to be seasonal (about six months) and on a year base the factory is operating below its capacity.

No problems were reported as to the dehydration process, the quality of the final product or to the system of control of this quality. The factory possesses premises for an adequate quality control and reportingly, a qualified chemist has recently been appointed for this job.

The factory wants, however, to find some use for its rather big quantity of wasted onion peel. (in 1974 amounting to 2640 tons). An Egyptian expert visiting the factory has given recommendations for extracting pectin from the onion peel and has suggested a flow sheet for such a production. In the meantime, Harasta Institute, Damascus (Agricultural Research Institute, Ministry of Agriculture) has performed experiments to extract pectin fm onion peel and to evaluate its technological and organoleptical qualities.

On this background, the Union of Food Industries has recently put out on the world market tenders for know-how, equipment and technology, concerning a pectin plant based on onions or onion skin. So far, this inquiry has brought no response.

3.2.6 Assistance to the sugar industry

The Syrian sugar industry is 100% nationalized with 3 governmental plants in production, one in Damascus, one in Homs and one in Jisr Esh Shughur. During the Expert's appointment the factories in Homs and Damascus were visited.

The production lines consist of equipment of a very diversified origin and age which makes repair and maintence complicated and difficult. The factories are partly (60 days early) ameliorating local crops of sugar beets, and partly refining imported raw sugars. In the refining process, the Damascus factory is applying the principle of phosphatation while the two other factories apply the carboratation process.

The factories are in deficiency of sufficient store rooms for the imported raw sugars. Very often this is done in open air, with unfortunate phenomena of crystallization as a frequent results. In the Damascus factory the filtration process is a bottle neck because of an excessive flock formation following the phosphatation.

Due to shortage of skilled trained operators the enforcement of measurements of process and product quality control is very moderate, which makes steering and optimising of processes very difficult. The factories want to use the IRDC to further develop their control systems and to verify their own contol results.

3.2.7 Assistance to the milk sterilizing plants

Pasteurized fresh milk is not available on the Syrian market. However, sterilized, bottled milk is produced and plants for such production exists in Damascus (2), Homs (1), and Aleppo (1). Together with counterparts the biggest of these plants (Damascus) was visited. This plant was founded by contributions from UNICEF about eight years ago. It is a modern set up with a good flow line for production of sterilised milk and it also possesses good premises for laboratory examination of milk and dairy products. It operates, however, far below capacity,

normally producing 5.5 tons sterilized milk per shift from a capacity of 12 tons.

The Expert and his counterparts were presented to specific problems in which the plant management wanted assistance from the IRDC:

- recommendations for an easy method to detect if raw milk from farmers is mixed with rehydrated dry milk powder.
- recommendations for methods to determine rest concentration of chlorine in water
- recommendations for rapid methods to detect traces of antibiotics in raw milk
- recommendations for available literature on flavoured milk.

Detailed advices on each of these topics have been prepared and forwarded to the plant management.

3.2.8 Assistance to factories producing food packaging materials

Each of the Syrian canning factories is set up with a small producing unit for empty cans. To secure a sufficiently high standard of quality, this necessitates a comprehensive system of quality control on each production site. On the arrival of the Expert no such control system existed.
During his assignment the Expert has prepared technical specifications and methods of quality control for tinplates, can laquers and can lid linings. He has also recommended a system for routine control of can seams. The control program is included in "A Recommended Program of in Plant Quality Control for the Modern Conserves and Agricultural Industries Corporation", here attached in summary as Appendix 9.

At the moment the Union of Food Industries are looking into the feasibility of the present system of production of empty cans to see if a central factory for automatic production would be preferable. The Expert has supplied the Union with information as to production capacity and equipment cost for different types of factories (automatic, semi-automatic) for production of empty cans. Tenders have been sent out on the world market and at the moment the different offers are being estimated.

4. RECOMMENDATIONS

Most of the recommendations given by the Expert during his assignment have been dealt with in the preceeding chapter. In the following recommendations are given for further initiatives to complement and to extend his activities.

4.1 Further activities inside the Industrial Research and Development Center (IRDC)

4.1.1 Supply of equipment

In spite of several efforts to speed up the delivery of UNIDO equipment for the microbiological laboratories this has still not arrived. To go on with the program of counterpart training these laboratory facilities are absolutely necessary and no further activity or extension of assistance program is recommended until the ordered items are delivered.

Most of the equipment is relatively simple to install and needs no expert advice in this respect. The autoclave will need connection to water supply and possibilities for convenient drainage. Preferable, this instrument should be placed in a room specially intended for preparing of culture media and washing and sterilization of glasswares, and preferable it should be placed under a fume hood.

Special experience is needed to operate some of the instruments in question. This applies in particular to the Howard Mould Count Cell, the Ultra-K-apparatus and the Ellab Automatic Fo-value computer. Recommendations to overcome these difficulties will be given in a following chapter (see point 4.1.4)

4.1.2 Methods of food analysis at IRDC

Numerous methods have been proposed for the evaluation of food quality. Some of these methods have been adopted by national and international associations as official reference methods while

others serve as convenient routine methods where official methods may be not so suitable.

In the opinion of the Expert, the choice of methods of analysis is a very difficult and a very important problem for a food laboratory. For this reason, detailed recommendations are here given for a series of suitable methods for use at the IRDC. In each case reference is given to an officially recommended method, and, in some cases, to the special surveys of methods prepared by the Expert and referred to under point 3.1.3. The recommendations are appended as Appendix 10.

Many of the methods here recommended are not easily available in handbooks or other scientific literature. Therefore, a manual of "Supplementary Laboratory Methods of Food Analysis for Use at the Industrial Research and Development Center" has been prepared. It is attached to this report as Appendix 11.

4.1.3 Implementation of food standards

Work on a variety of new food standards is now in the process of being taken up by the Syrian General Standard Committee and will be approved and come into force before long. However, the implementations of these standards needs extra regulations, preferably enforced by law. This is especially the case for imported canned products.

It is recommended that before being released from the Customs, all canned food products which are imported into Syria and which are covered by general or special Syrian standards of product quality, should be tested and approved by an official instance specially authorized hereto. This task should be undertaken by the IRDC.

Each importer should, if possible before the arrival of the lot, inform the IRDC about quantity and type of product, harbour or town of import and anticipated date of arrival, and invoice showing the value of the product.

The IRDC should, at its earliest convenience, draw the necessary numbers of samples and at once submit these to the prescribed control measurements, whereupon the importer and the Custom authority should be informed of the control results. Products

which do not fulfill the Syrian requirements of quality should not be allowed to be imported into the country. The same should apply to canned food products which are not labelled in accordance with Syrian regulations for labelling.

4.1.4 Program of training abroad

It is recommended that Mrs. Abeer Khaznadar should get possibilities of training in food analysis abroad and that this program of a six month duration should be organized as UN Fellowship. The program should comprise:

a. A study in microbiological, chemical, biological and physical methods of evaluating the quality in food. This study may be performed at the Norwegian Food Research Institute (NINF) situated on the university campus at Aas, 40 km outside Oslo. This institute is dealing with quality studies in all kinds of food, except cereals and dairy products. It is a new and modern institute, advanced equipped and with a competent staff. Here training also could be given in use of the Howard Mould Count Cell, the Ultra X apparatusg and the Ellab Automatic F_0-value computer (see point 4.1.1)

b. A study in analytical methods for control and testing of dairy products. This part of the fellowship could be carried out at the Institute of Dairy Research, Agricultural College of Norway, also situated at Aas.

For the next two years, the Expert will be the administrative head of the Norwegian Food Research Institute, and in a good position to facilitate all arrangements concerning Mrs. Khaznadar's study and to supervise her work and arrange her program to suit her future stay at the IRDC.

4.1.5 Further UNIDO assistance for the IRDC food laboratories

Due to lack of sufficient laboratory equipment it has not been possible for the Expert to fulfill the duties of assistance to the IRDC expressed in his terms of reference. To get the laboratories in optimal operation, a short time assignment (2 months) for a UNIDO Expert in food analysis is recommended. His terms of reference should be:

- Make a proper installation of equipment for microbiological and chemical analysis at the IRDC laboratories.
- Training of counterparts in food analysis according to the recommendations for suitable methods of food analysis at IRDC (Appendix 10).
- Undertake a work program at the laboratories aimed at the improvement of the standards and qualities of the Syrian food industries.

If the fellowship of Mrs. Khaznadar can be arranged in summer/autumn 1976, it is recommended to await initiating of the extended program of assistance until her return to the IRDC. Under no circumstances should such an assistance take place during Mrs. Khaznadar's absence.

4.2 Further activities outside the Industrial Research and Development Center

4.2.1 Implementation of program and standards for industrial quality control

The recommended program of quality control for the canning industry is recommended implemented as soon as possible. So far, this has not been the case.

To perform a quality control according to the guidelines given, qualified chemists or food technologists are needed. In the Syrian food industries there is a scarcity of people qualified in these fields and in many cases such people hold pure administrative posts in the companies. The Union of Food Industries is recommended to point out people with a chemical or technological background for in plant quality control work. These people should be given supplementary training and advice in performing the program of work, partly at the IRDC and partly in the factories. (See also recommendations in Final Report from Food Technologist M. Todorovic)

During the assignment of the Expert there has been a frequent change in technical management in the food industries. Key people are being transferred from one job to another in a high degree. The counterpart of

Food Technologist M. Todorovic is no longer engaged by the Modern Conserves and Agricultural Industries Corporation, and it has been difficult to brief his successor in all advices given by Mr. Todorovic. Such incidents have made implementation of the program of quality control somewhat complicated, and a better stability of technical management is recommended to be able to accomplish this work.

4.2.2 Implementation of regulations for good manufacturing practice

To make a high quality food product an adequate program of quality control is not sufficient. The whole infrastructure of the processing plant should be taken into consideration and regulations for a good manufacturing practice should be laid down. For the processing of low acid foods in hermetically sealed containers, recommended Syrian regulations have been prepared and presented to the technical managementm at the Modern Conserves and Agricultural Industries Corporation. These recommendations are in good accordance with similar regulations laid down by the US Food and Drug Administration[1)] and with Norwegian regulations in this field. The proposed Syrian regulations are appended (Appendix 12). To implement the proposals it is recommended to enforce the regulations by law.

4.2.3 Safety measurements in the factories

Official measurements to approve the safety conditions in the canning factories are strongly recommended. The regulations for good manufacturing practice (Appendix 12) state requirements for all operations in the thermal processing room. Specifications should also be given on how to run hydrostatic pressure tests on the autoclaves, at what intervals such tests should be repeated and in what way to implement the test results.

Recently a report has been prepared by the IRDC (Dr. Eng. D. Köttgen, Eng. G. Al-Saleh and Eng. G. Makhoul) with proposals for a steam boiler safety control system in Syria. Here it is recommended that setting up of safety regulations, codes, etc., for steam boilers should be one of the responsibilities of the Standard Department of the IRDC, and that safety inspectors

1) ~~F.D.A. Department~~ of Health, Education and Welfare: 1973. Federal Register, Vol. 38 No. 16, Jan. 24.

should be appointed to control and authorize the installments. This program should be extended to include control and inspection of all pressurex vessels, and it is recommended for IRDC to initiate a legal base for retort safety regulations and to enforce such regulations as soon as possible.

4.2.4 Further assistance from the IRDC to the food industries

In connection with the activities mentioned under the Expert's findings, there are some immediate problems in which the food industries want further assistance from the IRDC.

When the orange season starts (October/November) the IRDC should contact Jable Canning Factory and start experiments making orange marmalade following the recipes and process recommendations given by the Expert.

The experiments trying to substitute cocoa butter in chocolate should continue. The aim of the management has been to accomplish a 100% substitution. This is a very difficult task, and food research laboratories all around the world are working on this problem [1]. A partial substitution is easier, and it is recommended to continue experiments using a mixture of cocoa butter, Cro klan and Sandarit (the latter being the far cheapest). Detailed advice for such experiments are given. An ultimate goal should be to substitute the cocoa butter with locally produced fats. When better refined, the hydrogenated cotton seed oil (Vegetamine) would represent an interesting alternative. In the meantime the use of shea$ butter (karité) could be tried.

Before starting up a full scale production of pectin from onion peel, the feasibility of this investment should be further investigated. Scale economics indicate that, in Europe and North America, a pectin factory should have a minimum capacity of 500 to 600 MTPY in order to be marginally operable.[2]

1) Wilhand, H.: 1972. Cocoa and Chocolate Processing Food Processing Review No. 27, Noyes Data Corporation, New Jersey, USA
2) Van den Ent, E.: 1975. Personal communication.

Moreover, the capacity of pectin processing in the world is presently on a high level, production being substantially less than capacity, with the resulting pressure on price, and with an imminent danger of dumping. At the present, no pectins are manufactured in Europe or North America on the basis of onions or onion skins, and several problems concerning such production need better clarification, i.e.: quality of the product (jellying power, strength), pectin content in onion peel over a longer period of time and under different storage conditions, elimination of onion flavor, etc.

If the Union of Food Industries decides to go on with this project, recommendations should be made for the IRDC to assist in solving the problems here mentioned.

To be able to give a continuing proper assistance to the food industries, the contact between the IRDC and the industries needs to be strengthened. The industries need to be aware of the capacities of the Center, and the Center needs to be familiar with all actual problems in the industries. More direct contacts between the management of the industries and the management of the Center is recommended as well as between the technical staff in industries and at the Center.

The personnel at the Organic and Food Laboratories Department lack experience from industrial food production. A special food industries consultant with good theoretical background and with a broad experience from food production should be appointed to the Center,. This would further secure a continuing good contact and also giving the Center proper problems to work upon.

4.2.5 Extended UNIDO program of assistance to the food industries in Syria

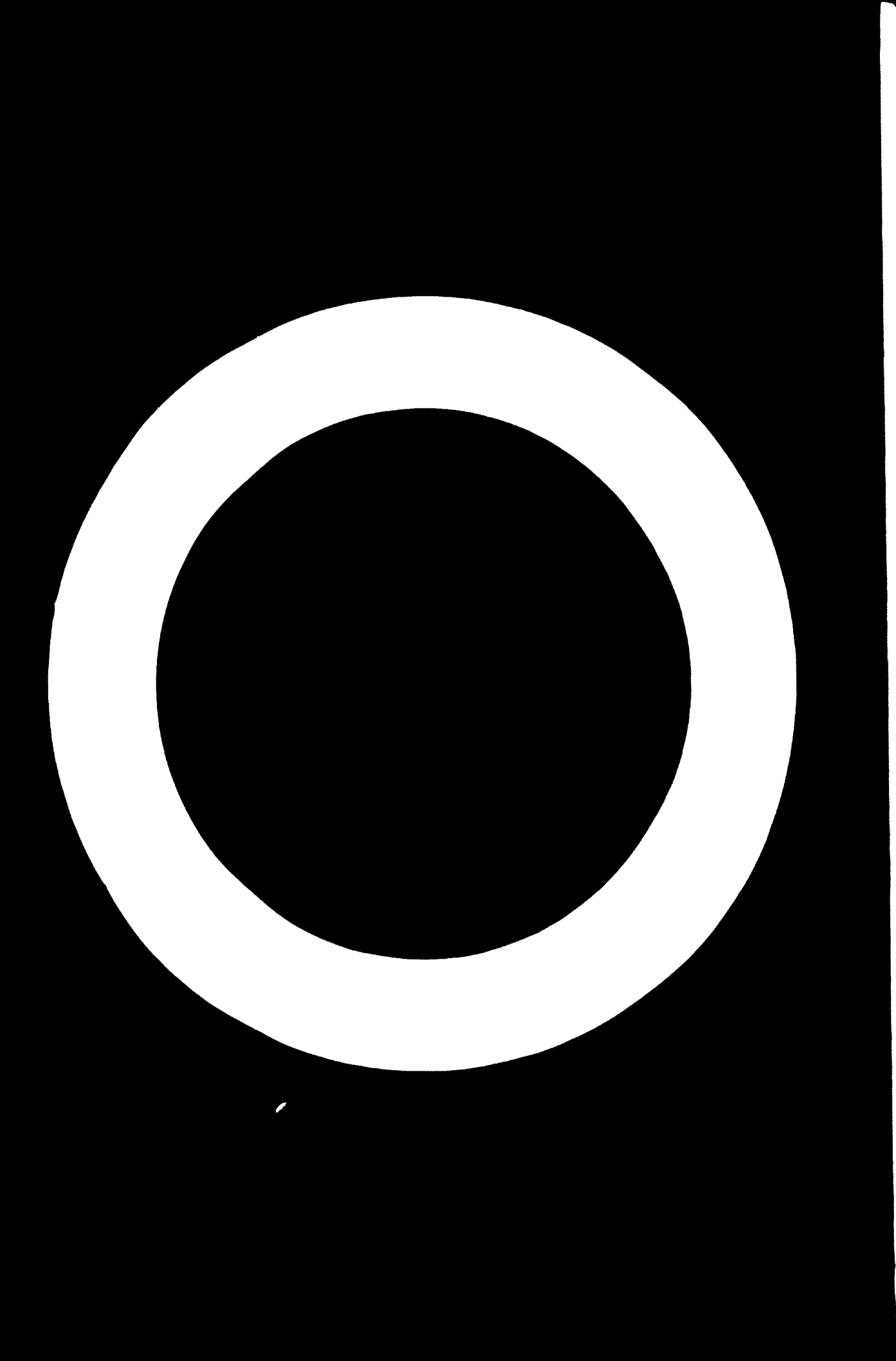
Before preparing a large scale project as a follow up assistance program to the Syrian food industries (ref. terms of reference for Mr. Todorovic for his second assistance mission to Syria), the following matters should be given a careful consideration:

The development plans for the Syrian food industries as outlined under the Expert's findings will, to a certain extent, change the structure of this industrial branch. The structure of the industry will reflect the need for assistance in trouble shooting and development work, and a long time plan for industrial development should be carefully studied before any assistance program is decided on..

The future role in food research of the Haraeta Institute should be clarified. Today, this institute performs a considerable research on agricultural food raw materials: Selecting of suitable varieties of fruits and vegetables for processing, advising on proper harvesting with regard to quality, etc. The institute possesses a pilot plant for preservation experiments and is also working on establishing quality standards for canned food products.

The future role of the IRDC in food research should also be elaborated. Already, the Center has laboratory premises and staff sufficient to cover most need for research in the fields of establishing systems of quality control, standardization, trouble shooting and other technological consultancy services, technical and economical feasibility studies and laboratory services for chemial and microbiological analysis. Supplied with a pilot plant, the IRDC will also cover the need of research as to process and product development and of problems in packaging food products.

In the Expert's opinion these matters should be considered before taking any decision on establishing a new institution in Syria analogous to the Food Processing Industry Center now being planned in Iraq.



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Microbiology of water
Bacteriological examination of [illegible]
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Microbiology of milk
Bacteriological examination of [illegible] milk
Preservation of milk and milk products
Microbiology of [illegible] dairy products
Microbiology of meat
Preservation of meat and meat products
Microbiology of fruits and vegetables
Preservation of fruits and vegetables

...LY OF METHODS FOR THE ENUMERATION OF MICROORGANISMS

1. Introduction

Growth of microorganisms is generally measured by estimating increases in numbers in relation to time. Enumeration may be carried out according to several different methods, but the results are, at best, estimates having mainly statistical value and are approximately correct only within certain limits of cell numbers.

2. Direct Methods

2.1 Hemacytometer cell counts

Principle

By counting the individual cells in a counting chamber of exactly known volume, the numbers of organisms per unit of sample is computed.

Remarks

The method is applicable to any suspension of microscopic particles. It gives a total count of live and dead organisms. Numbers of the order of at least 1×10^7 per ml of suspension are requisite for statistical validity of the count.

2.2 Smear counts

Principle

An exact volume of the sample is smeared over an exact area on a slide, stained with an appropriate dye and counted.

Remarks

The method is simple and little time-consuming. It gives a total count of live and dead organisms, different morphological types may be distinguished and the slide may be kept as a record. The method is applicable to any suspension of microscopic particles that can be visualized under the microscope.

2.3 Comparative counting method

Principle

An exact volume of sample is mixed with an exact volume of fluid with a known number of characteristic particles in it and the amount of microorganisms are compared with the amount of particles as seen under the microscope.

Remarks

The method gives the total number of live and dead cells. It is a total estimate. In food microbiology it is not much used.

2.4 Membrane filter counts

Principle

Measured samples of fluid are passing through sterile, porous-membrane filters which trap the microorganisms. The organisms may be counted directly or grown for colony counts (see later method)

Remarks

All microorganisms in a relatively large volume of fluid may here be collected on a small dish without handling lots of tubes or flasks with large volume cultures. The method is also well suitable for detecting special indicator organisms in water, milk, etc.

2.5 Electronic Counter

Principle

Samples with microorganisms are passing an electronic beam that traverses a space between two closely adjacent electrodes. Each particle causes an interference with the electron beam and this interruption is taken up by instruments and recorded electrically.

Remarks

All particles - living and dead substance is registered and impurities of the samples may give false results. The method should, therefore, be applied only to estimation of bacteria in homogen solutions free of other particles.

Instruments with high speed scanning beams that can count enormous numbers of colonies on agar plates are now available. With such automatic bacterial colony counters a technician can determine at a glance the number of colonies growing on an agar surface. The colonies are scanned, the count is registered digitally and the colonies are marked and shown on a vidicon screen. In routine examination of milk this technique is now introduced with great success.

3. Indirect Methods

3.1 Determination of total number

Principle

A standard volume of culture is centrifugated at a standard speed for an exactly measured time. From a knowledge of the average volume of the individual cells an estimation of numbers is possible.

Remarks

The method gives a total estimate of bacteria. In a modified form it is used in medical diagnostic studies to measure the total volume of blood corpuscles in a hematocrite determination. In food microbiology it is not much in use.

3.2 Turbidometric methods

Principle

The turbidity or scattering of light in a culture due to accumulation of evenly dispersed cells suspended is measured, and turbidity data are recorded as an expression of rate of microbial growth.

Remarks

The method is subject to errors due to variation in size and shape and clumping of the cells as well as to different degrees of translucency of various species and other materials in cultures. However, the method is one of the quickest and simplest and is reasonable accurate. It should be brought in mind that turbidity data are not numbers of bacteria and cannot correctly be used as such in calculations based on exponential expressions of cell numbers.

3.3 Dilution methods

Principle

A sample of exactly known size is eventually after homogenizing - diluted with sterile distilled water (or 0.9% salt solutions) 1/10, 1/100, 1/1000 and so on. The enumeration of bacteria in each dilution may follow according to the principle of Most Probable Number where growth or no growth is observed is in two-fold, five-fold or ten-fold series, and where existing tables show the most probable number of bacteria calculable from all possible combinations of results in such series. The enumeration may also follow as Colony Counts where duplicate samples from each dilution is plated on suitable media and then incubated at a suitable temperature before counting.

Remarks

Dilution methods are widely used to estimate numbers of viable bateria in foods. The most probable number calculation is much used in the examination of water where special tables are available. (Standard Methods for the Examination of Water and Wastewater. 1971. 13th Ed. American Public Health Association).

Different methods can be used for obtaining colonies for counting. Most commonly the outgrowth is performed in Petri Dishes. Whatever method used, each colony represents, theoretically, one single cell from the original incubation. Actually, several organisms, if stuck together in a clump, will give rise to only a single colony. However, the errors in colony-count methods are fairly well known, and such methods are the most useful for enumeration of microorganisms in foods. It should, however, be brought in mind that it measures only organisms viable under the conditions of growth (medium, temperature, etc.) provided.

4. References

A detailed information on the methods mentioned in this survey may be found in:

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SURVEY OF LABORATORY METHODS FOR DETECTING
SPOILAGE IN FOOD AND FOOD PRODUCTS

Meat and Meat Products

1. Introduction

After slaughter a series of chemical changes take place in the animal muscular tissues. Existing compounds are broken down and new compounds are formed in processes which are accelerated by enzymes of internal or external origin. The internal enzymes, still active, accelerate the metabolic processes in the live animal. Their action can be slowed down by low temperature storage of the meat, but it cannot be totally prevented. The external enzymes originate from microorganisms brought into the meat during slaughter and post-slaughtering processes. Hygienic handling of the meat will to a great extent prevent this enzymatic activity and thus extend considerably the keeping quality.

Numerous microbiological, chemical, physical and biochemical tests have been prescribed to reflect changes occurring in meat and perishable meat products during storage. Recently these methods have been reviewed by Pearson (21) and by Simonsen (29). Some of these methods are described in Recommended Methods for the Microbiological Examination of Foods (1) and will not be mentioned here. A short survey of the different other methods will be given.

2. Microbiological methods

2.1 Contact plate methods

Principle

Microbial contamination on meat can be demonstrated by the direct surface agar plate method described by Angelotti et al (2,3). A solidified sterile medium is exposed to the meat surface; the medium is incubated and microbial colonies counted.

W[illegible] this technique on ox and pig carcasses, [illegible] found the colonies showed confluence and were difficult to count. He would, therefore, not [illegible] this method. It is indicated, however, that the method can be used on samples with less contamination, particularly on cooked meat (29).

Leistner (19) has described a technique in which he combines the contact plate and a replica method and by which he claims to be able to transfer and to count 2/3 of the bacterial present on the meat.

2.2 The Agar sausage method

Principle

Agar is filled into artificial casings so as to make agar sausages. These are sterilized and the agar is allowed to solidify. When doing the test, the end of the sausage is cut off with a sterile scalpel, the agar extracted from the casing by slight pressure and the exposed agar pressed against the meat surface to be tested.

Remarks

In Scandinavia this method is now widely used to evaluate the surface contamination on carcasses as well as on different vacuum-packed meat products. A detailed description of applying the agar sausage method to examination of meat is given by Ølgaard (33). By examination of meat products only a pre-storage condition can be measured. According to Zeuthan (32) 100 colonies on each slice of agar-sausage would equal 2500 microorganisms per gram in the product, and by storing the meat the microbial population would soon multiply so as to make this form of evaluation impossible.

2.3 The filter paper method

Principle

A membrane filter or an ordinary filter paper is exposed to a contaminated surface, whereafter the filter is transferred to a suitable diluent for plating with an appropriate culture medium.

Remarks

Using a multipore filter, Silliker et al (26) found slightly less bacteria with this method than with a conventional method. Baltzer and Dalhoff (6) have shown that the results are in accordance with conventional methods as long as the meat surface is wet. When the surface is drying, this technique becomes unreliable.

2.4 Swab methods

Principle

A meat surface of known size is carefully rubbed with a sterile swab, which is then transferred into a sterile diluent, whereafter the rinse fluid is plated with an appropriate culture medium.

Remarks

By measuring the keeping quality of chilled beef, Ayres (5) has found this method to give results comparable with those of conventional methods. Ojala had the same experience while examining cattle and pig carcasses (19). Mossel and Buchli (17) have further developed this method to make it particularly suitable for detecting of Entero-bacteriaceae. This is done by using alginate swabs over comparatively large surfaces.

2.5 **Rinse tests**

Principle

An area of known size is immersed in a sterile fluid which is then agitated either manually or mechanically. To detect the micro-organisms, the fluid is plated with an appropriate culture medium.

It was reported by Williams (31) that this method would give 10% or less difference in bacterial count compared with a method using wet swabs. Ayres (5), however, found rinse tests rather impractical for measuring the bacterial load on meat, and that it was necessary to rinse samples at an accurate measure of 40 cm² in order to get reliable results.

2.6 Microscopic examination

Principle

The bacterial population of a meat sample of known size is transferred to a diluent by mixing or stirring. A drop consisting of 1/100 ml of the fluid is placed on a microscopic slide, dried, fixed with an appropriate dye. By examination of an appropriate number of microscopic fields the total bacterial population on the slide can be measured and, hence, the bacterial count of the whole sample.

Remarks

Above all, this is a very rapid method for detecting bacteria on meat. It can be applied to every kind of meat product and it can also be used to check the quality of raw materials in cooked or processed products. In Germany this is a recommende method for examination of imported canned meat products (13). If, by this test, more than 30 bacteria are found in 20 microscopic fields, the product is subjected to an extensive bacteriological examination.

2,7 Anaerobic bags for detecting clostridium sp.

Principle

The sample is placed in a plastic bag, which is then filled with a suitable agar medium and then sealed and incubated at a convenient temperature for growth of bacteria.

Remarks

This technique, originally described by Bladel and Greenberg (7), avoids the usual measurements for anaerobic growth of bacteria. It is, however, as time consuming as conventional techniques. The recovery of Clostridium botulinum has proved to be significantly higher by this method than by other method

2.8 The can method for detecting Clostridium sp.

Principle

The meat sample is mixed with a sterile diluent and filled into 10 cans, which are sealed and then heat-processed according to the following schedule: 3 cans are boiled for 15 minutes, 3 cans for 30 minutes, 3 cans for 45 minutes and 1 can for 60 minutes. The cans are placed in an incubator or an oscillating table where they are stirred for one hour daily in 5 days. If, after 5 days, 6 to 7 cans are blown, the probable number of Clostridia will be 0.5 - 1 per gram.

Remarks

In canning of meat product it is always of great importance to have an estimation of the amount of heat resistant organisms in the raw materials. By this method, developed by Madsen (16), this can be done without much laboratory equipment. However, the method is not less time consuming than the method for detecting spore-forming organisms previously mentioned.

3. Chemical Methods

3.1 Determination of volatile bases

Principle

Many different techniques have been described for this determination (21). Most of the methods involve either macro- or semi-micro distillation, micro-diffusion, aeration or colorimetry. According to a common procedure, a filtrate from a meat macerate is distilled and the volatile nitrogen is absorbed in boric acid (20).

Remarks

Compared to fish, meat contains trimethylamine only to a very limited extent, and the volatile nitrogen consists almost entirely of ammonia. The results would, therefore, be similar to those obtained by a direct ammonia determination, and according to Turner (30) one would only get clear answers when the sample is of a very bad quality.

3.2 Determination of ammonia

Principle

The ammonia formed by the breakdown of meat protein can be determined by the method of Eber (quoted in 25) or Nessler (quoted in 30).

Remarks

In Germany, determination of ammonia by the method of Eber has been widely used. This has, however, been criticized, partly because of the difficulties in determining small concentrations and partly because of its limitations in determining only free ammonia (25). The Nessler method is recommended, since this method shows a more distinct reaction, particularly in bad quality meat. This is in accordance with the findings of Turner mentioned earlier (30).

3.3 Determination of hydrogen sulphide

By microbiological decomposition of meat, hydrogen sulphide may be formed, and this can be determined by adding lead acetate. Formation of lead sulphide will then be an indication of microbiological spoilage.

Remarks

Microbial activity is not always associated by formation of hydrogen sulphide, and even sterile meat can give a positive lead sulphide reaction (29). Nevertheless, satisfactory results of determining quality have been obtained by exposing lead acetate impregnated filter-strips to chilled packages of meats (14).

3.4 Determination of free amino acids

Principle

The amount of free amino acids can be determined by formol titration of a filtrate from a water extraction of the meat sample.

Remarks

In USA evaluation of meat quality by formol titration is described as an official method (4). Turner (30), however, found this method most unsatisfactory, as the sample had to be almost completely spoiled before the amount of free amino acids showed a significant increase.

3.3 Determination of nitrate decomposition

Principle

A 0.1% nitrate solution is added to the sample, whereafter it is incubated at 30°C for 4 hours. If nitrite is formed, this is an indication of the presence of large amounts of nitrite-reducing bacteria, especially Escherichia coli.

Remarks

The method is suggested as a rapid way of determining the hygienic quality of meat (18). It is, however, limited to evaluating secondary contamination in fresh meat (29).

4. Physical methods

4.1 Measuring pH

Principle

Due to excessive formation of lactic acid, there will be a rapid fall in the pH of meat after slaughtering. Extended storage may lead to a subsequent rise, mainly due to bacterial growth on the meat surface. pH-measurements can be done with a suitable pH-indicator or with a pH-meter.

Remarks

The practical advantage of this method lies in its simplicity and rapidity. Fresh meat should have a pH lower than 6,0. Fresh meat with a pH value between 6.0 and 6.5 should be rejected and submitted to a further examination (21). A pH value higher than 6.5 is an indication of the animal being physically exhausted or suffering from a long and tiresome sickness before slaughtering. A great disadvantage from using the method is the fact that results are strongly dependent upon the type of the bacterial flora and also on the storage conditions of the meat. According to changes in bacterial composition, packed in plastic bags the meat will show a lower pH than otherwise (29).

4.2 Determination of extract release volume (ERV)

Principle

When mixed and homogenized in water or in a buffer solution, a meat sample of bad quality will give a more viscous mixture than would a sample of good quality. After filtration, therefore, a homogenate from meat with high bacterial load will give less filtrate than would a sample of good quality.

Remarks

Jay (12) has developed a method for determining the extract release volume (ERV) after filtration of a meat/water homogenate. He found a good correlation between ERV and pH of a sample, and that a pH of 5.0 would give the maximum extract volume. Other investigators have confirmed this finding (9,23) claiming ERV to be more correlated to pH than to any other quality criterion, and having the same limitations as the pH value for measuring quality.

4.3 Determination of the oxidation-reduction potential

Principle

Bacterial growth will reduce the oxidation-reduction potential in stored meat. Measurement of this potential has been suggested as an evaluation of meat quality.

Remarks

Usually it is considered difficult to determine the redox potential in meat by direct measurements, although such methods have been suggested (10). However, many of biochemical methods for determining meat quality use a redox indicator with a change in colour according to the potential value.

4.4 **Determination of water-holding capacity**

Principle

The water-holding capacity (WHC) is the ability of meat to hold its own or added water during the application of pressure or mincing. WHC has its highest value immediately after slaughtering and is gradually decreasing during storage (11) and several methods for measuring it has been suggested.

Remarks

Determination of WHC is widely recommended and used for evaluation of meat quality(11). It is demonstrated, however, that reproducible results can only be obtained when the experimental conditions are fully standardised (9). In each case sample should be drawn from the same muscle. To avoid dehydration the determination should be carried out immediately after drawing the sample. These conditions are limiting the practical use of the method.

[illegible]

.1 Reduction of methylene blue

Principle

Bacte[illegible] (d[illegible]) [illegible] to the colourless leucomethylene blue. [illegible] present, the faster the reaction.

Remarks

The method is similar to the [illegible], and widely used in the quality [illegible]. [illegible] (26) [illegible] good co[illegible] and time of reduction. [illegible] with [illegible]. It is, [illegible], a comparatively slow method.

.2 Reduction of [illegible]in

Principle

Due to the [illegible], the blue coloured resazurin is reduced to the red [illegible] in and further to the colourless dihydro[illegible]. [illegible] of the reaction is a measurement of the [illegible] present.

Remarks

The method was first described by [illegible] (22) who found a good correlation between reaction time and organoleptic quality in meat [illegible]. Several investigators have later confirmed these results (9,27). Different techniques for measuring the resazurin reduction have been described. A Danish method using resazurin impregnated filter strips (28) has proved very convenient and is now widely used. It should be [illegible] in mind that if reducing substances (ascorbic acid) are added to the meat, this will disturb the reaction (9).

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Fruit and Vegetable Foods

1. Introduction

Research on methods for detecting spoilage in fruit and vegetable foods has been less than in foods of animal origin. The reason for this may be that fruits and vegetables are supposed to be of less potential health hazard than animal products. However, from the quality point of view the need for such methods is just as great.

The literature indicates only a few microbiological and chemical methods for this purpose. A review on chemical indicators for quality control of fruit and vegetable products has been given by Fields (7). Cowell and Morisetti (3) have summed up what has been done on microbiological techniques. Recommended methods for the Microbiological Examination of Foods (1) gives some methods for fruit and vegetable examination. These will not be mentioned here.

2. Microbiological Methods

2.1 The Howard Mould Count

Principle

The sample is placed in a "Howard Cell" consisting of a graded slide supplied with a cover glass. Filamentous fungi are counted by microscopical examination of at least 25 separate fields of view. Results are read as number of positive and negative fields (8).

Remarks

The reliability of the Howard Mould Count has been widely discussed (4). It is, however, generally agreed, that a relationship usually exists between the mould count and the proportion of spoiled, rotten or mouldy fruit used in preparing a product. Certain factors can operate and cause this relationship to fluctuate between wide limits. Such factors are: Nature of spoilage and variation in mycelium formation among mould species, as well as manufacturing circumstances and sanitation conditions on the processing site.

For the provision of consistent and reliable results, the method requires an experienced analyst. There is often lack of agreement between laboratories counting the same material (17).

In spite of its limitations the Howard Mould Count is, in many countries, an official method for evaluating the quality of tomato products.

2.2 The sample incubation method

Principle

A few hundred individual samples (fruits) are picked from a lot. Each fruit is surface sterilized with sulphur dioxide and incubated for a week to ten days. Visible infections are then counted.

Remarks

This technique for forecasting microbial spoilage is reported to have been used in apples and grapes (14) where numerous controlled tests have shown remarkable consistency between forecast and actual delay which developed in the sampled lot during three to five months chilled storage. The method is used at fruit processing plants to pick out strongly infected lots for rapid use.

3. Chemical Methods

3.1 Determination of acetylmethylcarbinol (acetoin) and diacetyl

Principle

Many microorganisms can form acetylmethylcarbinol from acetaldehyd, while diacetyl may arise in the fermentation of sucrose via acetylmethylcarbinol. Both compounds give positive tests with Voges-Proskauer reagents, which are 1) alpha-naphtol dissolved in ethyl alcohol and 2) creatine dissolved in potassium hydroxide. When these reagents are mixed with either acetylmethylcarbinol or diacetyl, a red colour develops (10).

Remarks

Acetylmethylcarbinol and diacetyl have been used as chemical indicators of microbiological quality in many fruit products. Fields (5) reports it as a valuable indicator in evaluating quality of apple juice, and Holck and Fields (13) have shown that it also can be used as an index of quality in apple jelly. It is also reported that it has a potential for use as an indicator of microbiological quality in orange juice (11). One disadvantage of Voges Proskauer reaction as quality indicator is that some rots have been found to contain little or no acetylmethylcarbinol while orange juice from organoleptically sound oranges has been found to give a positive reaction (6,2). Bacterial flora, level of oxygen and type of carbohydrate present may also influence the results (7).

The method described by Fields (5) involves large samples and a somewhat complicated distillation technique. For routine industrial quality control, a fuchsin-SO_2 test for detecting spoilage (15) should be considered.

3.2 Determination of ethyl alcohol

Principle

Ethyl alcohol is a by-product of many bacteria, yeasts and moulds. Determination of the alcohol formed can be used as an indication of microbiological quality.

Remarks

Hill and Fields (12) have tried this principle for evaluation of the quality of apple juice. Here, a good correlation between % rot (determined by weight) and amount of ethyl alcohol formed was demonstrated. The major disadvantage of the method is the low boiling point of ethyl alcohol (7). Also, alcohol formed in the raw material, very easily can be lost during processing. Moreover, most techniques for estimating small amounts of ethyl alcohol require a rather complicated distillation procedure.

3.3 Determination of volatile and non-volatile acids

Principle

In fruit and vegetables, succinic, lactic, acetic and formic acids are formed as a result of microbial biosynthesis. Besides, galacturonic acid may be formed as a breakdown product of pectin. In principle, therefore, all these acids can be used as indicators of microbial quality.

Remarks

Harris (9) has shown that the increase in galacturonic acid in apples is due to galacturonase of microbial origin. In rotten fruit he found the amount of galacturonic acid to be about 20 times higher than in sound fruit. Mills (16) suggested galacturonic acid as an index of quality in strawberry juice. Succinic, acetic, formic and lactic acids have all been considered as chemical indicators of quality in different fruit and vegetable products (7). In most cases, however, correlation with microbial quality is not yet established. The possibility of losing some or all of the volatile acids during processing also limits their potential as chemical indicators of microbial quality.

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1. Introduction

Proper hygienic conditions are an absolute requirement for producing food of a good quality. At every stage of production, necessary steps must be taken to prevent outgrowth of microorganisms and spoilage and the hygienic conditions will have to be controlled by means of adequate and suitable methods. In most cases, a visual inspection is insufficient to evaluate the hygienic conditions. The inspector needs some extra tools for his work. He needs methods to detect the microorganisms eye cannot see. Recently, several methods have been announced for this purpose, and several authors have been reviewing this subject (12, 13, 14, 26).

In the following, a short survey of the most applicable methods for sanitation control in the food industries will be surveyed.

2. The swab-rinse technique

21 Use of cotton swabs

Principle

A surface of known size is carefully rubbed with a sterile cotton swab, previously moistened in sterile water. The tip of the swab is then aseptically placed in a tube containing a sterile diluent, the tube is vigorously shaken and the rinse fluid is plated with an appropriate culture medium.

Remarks

In many countries this technique is today widely used for estimating surface contamination. It has, however, some disadvantages. Experience shows that it is a poor correlation between the microbiological contamination present and that recovered by this method. (11). Moreover, two chemists will not use the swab in exactly the same way with respect to speed and pressure, and this will influence the results.

To get an idea of size sampling, a metal stencil with a square design of known size is commonly used. Standard Methods for the Examination of Dairy Products (1) suggests using the same swab on 5 areas of 8 square inches each.

Most surfaces have a rather uneven distribution of bacteria. Therefore, it has been suggested to rub off 10 different areas of 10 cm^2 each, using different swabs (17). The bacteria removed would then be transferred to the surface of a rigid agar slant by wiping the exposed sides of the swab directly onto the agar. After incubation, the slants would be grouped according to the numbers of colonies formed, and the average number of colonies with its standard deviation would be determined by plotting the distribution on a special probability paper.

2.2 Use of calcium alginate swabs

Principle

Swabs composed of calcium alginate wool will dissolve in distilled water or in Ringer solution. The microorganisms entrapped on the swab will thus be freed and the rinse fluid may be plated as previously described.

Remarks

When first described (18), this method was said to give higher recovery than by using cotton swabs. Other investigators (3,5) have not been able to confirm this findings. There are also results showing that the alginate may be inhibitory to some microorganisms (27).

The swab methods have other incorveniences. With respect to speed and pressure two people will not use the swab in exactly the same way. To eliminate this weakness, Reuter (23) has developed a sort of trigger consisting of a cylinder with a piston connected to a spring. A piece of alginate wool is placed under the piston. By delivering the piston, the wool is brought in contact with the sampling surface at a constant pressure. Using this technique, Coretti (11) recovered 85% of a known bacterial population on metal surfaces.

3. Rinse tests

Principle

An area of known size is immersed in a sterile fluid which is then agitated either manually or mechanically. To detect the microorganisms, the fluid is plated with an appropriate culture medium.

Remarks

The method may, within limits, be applied for use on large or stationary surfaces only. For sanitation control in food industry, therefore, it is of minor importance. Some investigators have compared this method with a direct surface agar plate method, and found it to give rather high but not so accurate recovery results (2). Clark (9,10) has developed a rinse equipment by which the rinse fluid is collected in glasses, whereupon plating is done out of these glasses.

Different methods are proposed to avoid the rather laborious traditional dilution and plating technique. Adjusted scopps may be used to plate exact quantities on solid media, or the rinse fluid may be membrane - filtered (3,15). On heavily infected surfaces, even direct microscopic examination may be carried out (8).

4. Agar contact methods

4.1 Contact plate methods

Principle

Microbial contamination on surfaces may be demonstrated in situ by a direct surface agar plate method (DSAP) described by Angelotti et al (2,3). A sterile agar medium is poured on the surface area to be sampled and left to solidify, the agar slab being protected from additional contamination by a suitable sterile cover. Upon incubation, the colonies at the agar contact areas are counted.

Remarks

In food industries, most surfaces for examination are fixed and difficult to incubate at proper temperatures. Application of the method is, therefore, somewhat limited. Moreover, the method can only be applied for use on plain surfaces. A modification has been described where a

piece of gauze is placed in a Petri dish, ends out on each side, so that the solidified agar may be moved out of the dish by handling the gauze ends. After being exposed to the surface of examination, the agar is brought back into the dish for incubation and examination (24). In another technique (21) the agar is poured into plastic bags, the bags sealed and the agar allowed to solidify. When sampling, the bag is cut open in one corner and the agar exposed to the contaminated surface.

4.2 The agar syringe method

Principle

An open-ended syringe is filled with agar, which is exposed to the sampling surface by pressing the piston. The contaminated agar surface is then pressed a little more forward and an agar slice of about 1 cm thickness is cut off. The new agar section is again exposed to the sampling surface and the procedure is repeated. The agar slices are collected in Petri dishes for incubation and examination.

Remarks

The method, originally described by Lisky (cited by Walther (29)) is a precursor to the agar sausage method. It is disadvantageous because of the need for a series of syringes which makes practical sanitation control somewhat complicated and expensive.

4.3 The agar sausage method

Principle

Agar is filled into artificial casings to make agar sausages. These are sterilized and the agar is allowed to solidify. When used, the end of the sausage is cut off with a sterile scalpel, the agar extracted from the casing by slight pressure, and the exposed agar pressed against the surface to be sampled. A thin slice, 0.25–0.50 cm, is cut off and transferred to a Petri dish which is then incubated. Bacterial colonies on the surface of the agar slant are counted.

Remarks

The agar sausage method was originally developed and described by ten Cate (7). In the last 8-10 years the method has had a widespread application. In Scandinavia it is used for routine sanitary inspections in slaughterhouses and in meat processing plants (19, 20, 30) and Dutch references are also reporting its usefulness (6,22). Agar sausages containing selective bacterial culture media (Mac Conkeys substrate, plate count agar, mannitol salt agar, sabouraud maltose agar) are now commercially available (4).

4.4 The swab pressure method

Principle

A piece of cloth is stretched on a cylinder which is wetted and thereupon rolled over the sampling surface. The contaminated cloth is thereafter rolled over the surface of a suitable agar medium and the agar incubated and examined.

Remarks

The swab pressure method has not given more reliable results than the different contact plate methods previously mentioned. Greene (16) who describes the method is nevertheless of the opinion that it should be recommended for control of sanitation in hospitals and institutions of similar kinds.

4.5 The "sticky tape" method

Principle

A piece of sticky tape is pressed against the sampling material whereafter the tape is transferred to a suitable agar substrate for incubation and examination.

Remarks

This technique is proposed for dermatological examinations (28). Comparisons of this method and alginate swab and agar sausage method have shown good correlations between "sticky tape" and agar sausage methods, while the alginate swab technique gave a comparatively much higher recovery count. (22).

4.6 The filter paper method

Principle

A membrane filter or an ordinary filter paper is exposed to a contaminated surface, whereafter the filter is transferred to a suitable diluent for plating with an appropriate culture medium.

Remarks

When first developed, this method was intended for counting microorganisms on meat surfaces (25). Besides, it is stated that it would also be a suitable method for sanitation control of different equipment in the food industries. However, as such it has not yet had any significant importance.

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SURVEY OF METHODS FOR DETERMINATION OF WATER ACTIVITY IN FOOD

1. Introduction

The water activity (AW) represents one of the main factors determining the degree of microbiological activity in food. Each microorganism has its particular Aw-limits for growth, and by changing the Aw, the food can be preserved or made more perishable (4). A sufficient low Aw will effective preserve the food irrespective of factors like pH and temperature. According to Norwegian official regulations a food is considered perishable if its Aw is above 0,90, and U.S. Food and Drug Administration exclude from the definition "low acid food" any product with a Aw of 0.85 or less (1).

It is, therefore, of great importance to be able to measure the Aw in food. Several methods for such measurements are described (7), including determination of vapour pressure in vacuum and air, determination of osmotic pressure and measuring depression of freezing point or rising of point of boiling. For practical purposes measurements of vapour pressure is most convenient. A short survey of different methods will be given.

2. Isopeistic equilibration method (Landroch & Proctor) Principle

When two samples with different vapour pressure are placed in a closed system, vapour is distilled from one sample and condensed on the other until equilibrium. In this method loss or gain in weight in a series of samples are registered when placed in a closed system over sulphuric acid solutions with known Aw (3). When applying this principle the samples must all have the same weight, the same humidity and the same surface area.

Remarks

During many years this was the most common method for Aw measurements in food. It needs only simple equipment and give results after about 20 hours of equilibration. However, it is rather laborious and not very convenient for routine analysis of series of samples. It requires a sample material of at least 200 grams and the sample must be homogenized as to give portions with equal weight, humidity and surface area.

When calculating the results, a graphical interpolation method is commonly used. By this means very exact answers can be interpolated. The accuracy of the method is, however, dependent on how accurate one can regulate the Aw of the sulphuric acid solution. Experiments (9) indicate that it is very difficult to establish stable and accurate relative humidities at high levels and that equilibration methods are not well suited for Aw above 0.95. The results from research on the graphical interpolation method show that this method gives accurate and reproducible results only when Aw is below 0.90. Most foodstuffs have a water activity above this limit.

3. Filter strip hygroscopic method (Kvaale & Dalhoff)

Principle

A saturated aquous solution of a salt will, at a given temperature, maintain a constant humidity within a closed space. By proper selection of salts a humidity range from Aw = 0.20 to Aw = 0.99 can be covered. In this method, strips of filter paper are impregnated with a series of salts. The papers are left together with the sample in a closed space for a preset period of time. After this exposure the state of the test-strips (whether wet or dry) is used as an direct indication of the Aw of the sample (2).

Remarks

The method is simple to carry out, it is inexpensive, reproducible and sufficient accurate for routine Aw measurements in food ($\pm$ 0.01). Its simplicity is convenient for large scale experiments and industrial quality control and it is not

destructive to the sample.

Changes from dry to wet state is usually easy to observe. Precautions should be taken, however, not to use salts which are able to crystallize with different amount of water of crystallisation. In such cases the filter strip may contain a mixture of salt crystals and the test becomes uncertain. Originally this method was developed for Aw measurement in the region Aw 0.90 - 1.00. Selection of other salts will make it possible to use the same technique at any humidity interval and to cover the Aw scale from 0.20 to 1.00 (2).

4. Mechanic Hygrometric Method (Rodel & Leistner)

Principle

Mechanic hygrometers are instruments which mechanic register dimensional changes in natural and/or synthetic materials caused by changes in humidity. Rödel & Leistner (8) have developed a principle where a bimetallic thermometer and a mechanic hygrometer is fitted inside the lid of a metal box giving space to about 100 grams of samples. After incubation until equilibrium, the Aw of the sample is directly read from a scale in the lid. A correction table makes it possible to perform measurements in the range of 17-25°C.

Remarks

The method is convenient for routine measurements. The equipment needed is rather inexpensive. The measurements are easy to perform and experienced operators are not needed. Fore mor accurate laboratory analysis the method has some inconveniences (10). Reproducible results are not obtainable with more than two decimals and calibration of the instrument is somewhat difficult to perform. It is mentioned (8) that a calibration every month would be sufficient. Experience has shown, however, that this is not enough and that one calibration every week may be needed (10).

In spite of such deficiencies, the instrument may be a good investment for industrial quality control.

5. Electrical Hygrometric Method (Sina Equi-hygroscope)

Principle

Electrical hygrometers are hygrometers which depend for their action on the changes in electrical parameters of certain substances accompanying variations in relative humidity. A typical sensor has a lithium chloride hygroscopic film coating a bifilar-wound, noble-metal resistance element (6). An instrument for measuring of Aw in food according to this principle has been developed.

Remarks

The instrument in question is Swiss made and marketed under the name Sina Equi-hygroscope. The price is about US $ 5000.

Norwegian testing of this equipment has not given result in accordance with the statements given by its producers (10). The instrument is very sensitive to small changes in temperature in the air around the sample. This is critical because a 1°C change in temperature gives a 5% change in relative humidity. The equilibration takes 4 hours which limits its capacity to a few samples per day. According to the same tests, reproducible results are not obtainable with Aw of more than two decimals.

6. Dew Point Measuring Method

Principle

Dew point is the temperature at which the partial pressure of water in a gas is equal to the saturated vapour pressure of water. A further release of moisture into the space, or a decrease of temperature, would cause condensation, or "dew" to form. The dew point of a gas is thus a measure of the moisture in the gas in terms of the partial pressure of the water vapour.

Remarks

For measurements of Aw in food this principle was of little importance until in 1972 Rödel and Leistner developed a special test chamber for an American equipment (Dew Point Hygrometer Model 880, produced by EG & E International, Inc.).

The measurements with this equipment are comparatively accurate and reproducible but this method also shows a few inconvenience. Of greatest importance is to be able to read accurate temperatures as well as the test chamber temperature as the dew point temperature. On the present available equipment, the scale for the dew point thermometer is to small and narrow for exact readings. The capacity of the equipment is limited. Northolt (5) has, however, suggested a modification which allows several samples to be examined at a time.

For research purposes, where an accuracy of three decimals in Aw-measurements are required, this method seems to be the best one (10).

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APPENDIX 2

SURVEY OF METHODS FOR DETERMINATION OF FAT IN SOLID FOODS

1. Introduction

Various methods for the determination of fat in solid foods are reported in the literature. The different techniques comprise gravimetric, volumetric and methods based on changes in physical properties due to presence of fat in a selected solvent. In most laboratories the fat content in a food sample is determined gravimetrically according to Soxhlet method. The Association of Official Agricultural Chemists states this method as official for determination of crude fat in meat, fish, cereal foods, cacao bean and its product and grain and stock feeds (2).

Although this method is accepted as a reliable reference method, it is rather complicated and time consuming. From a routine control point of view this is of great importance, and for such purposes several more rapid and simple methods have been developed. Most of such methods have, however, strict limitations for use, and the choice of method should in each case depend upon the grade of accuracy required as well as of the personnel and equipment that could be made available. In the following a short survey of suitable methods for determing fat in solid foods will be given.

2. Gravimetric Methods

2.1 Soxhlet method

Principle

A food sample is accurately weighed and then dried in an air oven at 100°C. The dried sample is placed in a Soxhlet apparatus, extracted with petroleum or ethyl ether, whereafter the solvent is distilled off, the

sample dried at 100°C to constant weight and then weighed. The difference in weight gives the amount of fat in the amount of foodstuff analyzed.

Remarks

As earlier mentioned, this method is generally accepted as a reliable reference method for the determination in foods. It has, however, a few inconveniences. It is rather time consuming: the predying of sample takes from 6 to 12 hours and for most foods the extraction will require about 12 hours.

The solvents that are used are extremely volatile and inflammable and it is essential that all joints of the apparatus are as tight as possible, and a good fume hood is required for the extraction and evaporation process.

Several factors determine the completeness of extraction, and the period of extraction depends upon them. Since the conditions of extraction for the most part cannot be definitely ascertained, the period of extraction is usually left to the discretion of the analyst (8). The method, therefore, requires skilled and experienced laboratory personnel.

2.2 Ultra-X-determination of fat

Principle

A special instrument, Ultramat (3) is developed for determination of moisture, fat, ash and protein in solid foods. For fat determination a sample is placed on a inbuilt balance under an infrared lamp and dried to constant weight. The dry material is then extracted with carbontetra-chloride, and the solvent is decanted off. This process is repeated five times whereafter the residual is weighed. The difference in weight between original sample and last weighing gives the fat content of the sample. By the use of a quick-ashing equipment, values for ash and for protein (by subtraction) can be obtained.

Remarks

The method is developed in Germany and is at present widely used as a rapid routine method in the quality control of meat and meat products. Although several workers have demonstrated good correlation between results from this method and Soxhlet method (4,5) it is not yet recognized as an official method of food analysis. It is a rapid method, usually the drying to constant weight takes about 15-20 minutes. The technique is almost entire mechanical and does not require highly experienced operators. Because of these conveniences, the method is especially suited for in plant quality control purposes, first of all for the determination of moisture but also for fat, ash and protein.

2.3 Gravimetric determination of fat in meat mixtures

Principle

Animal fat has a lower specific gravity than that of pure meat. The exact volume of a mixture of fat and meat of a known weight will be an indicator of the fat content of the sample. This volume can be measured by estimating the head space over the sample when placed in a closed container of constant volume. In this method the head-space is filled with water and the weight of the water is taken as an estimate of the fat content of the sample.

Remarks

Comminuted meat products are generally manufactured by chopping and grinding various batches of meat trimmings. It is not easy to estimate the fat content of each batch. Therefore, it may be difficult to arrive at the right fat content in the finished product. This method, developed at the Danish Meat Research Institute (9) is, above all, intended as a rapid, rough method for analysis of batches of meat trimmings.
The equipment needed for the analysis consists of a balance (up to 50 kgs.), a vacuum pump and a rigid container (20-30 l) supplied with a mechanism for air tight closure and a lid with a tap. The fat content is determined without previous mincing or homogenizing and the sample can go unspoiled back to its original lot after the examination. Diagrams are given showing the relationship between specifif gravity and fat content (9)

3. Volumetric methods

3.1 Gerber method

Principle

The sample is digested with sulphuric acid in a butyrometer, the fat is separated and volumetrically measured. Different types of butyrometers can be used for fat determination in solid foods. The procedure and the readings of results will, however, vary accordingly.

Remarks

This determination is recognized as a standard method for all kinds of dairy products (2,10) and special butyrometers have been developed for cheese, milk and cream. Adapted to other products, good results are reported from analysis of meat and meat products (12). One great advantage using this method is that series of samples can be examined at the same time. The method is, therefore, particularly suitable for in plant process control.

It is, however, not fully satisfactory for products containing cereals and spices. Such products require a long acid digestion which often gives a dark fat column, difficult to read on the scale.

3.2 Modified Babcock Method

Principle

The sample is digested with a reagent prepared by mixing equal volumes of CH_3COOH and $HClO_4$, whereupon the fat is volumetrically measured in a Paley-type Babcock cheese bottle.

Remarks

The Association of Official Agricultural Chemists states this method as official for fat determination in fish, applicable to raw, canned and frozen fish (2). The method is very similar to the Gerber Method, having its advantages and inconveniences. The Babcock cheese bottles are, however, not as easily available and as practical in use as are the butyrometers used in the Gerber Method.

4. Methods based on change in physical properties

4.1 Changes in refracting index in a solvent

Principle

When the fat containing food sample is dissolved in a suitable solvent, the refractive index is progressively reduced. The fat may be extracted from the sample by heating with the solvent before measuring the refractive index.

Remarks

According to this principle, methods have been developed for estimating the oil content of a variety of oil bearing seeds and fruits (8). A lot of solvents have been proposed for such determinations, of which Halowax oil (α- monochloronaphtalene) is found very suitable (13); the accuracy of the method depends on the accuracy with which the product and the oil is measured out and the accuracy with which the refractive index is read. Usually the results are in good agreement with those obtained by the ether extraction methods. The method is more rapid than most other methods and for this reason is now widely used in industrial control laboratories. Tables or charts for conversion of refractometer readings to fat contents are available for avocado oil, olive oil, coconut oil, sesame oil and sayabean oil (8).

4.2 Changes in specific gravity in a solvent

Principle

Total fat can be measured from change in specific gravity of a suitable solvent. Both carbon tetrachloride (7), dichlorobenzene (6) and trichlorethylene (11) have been proposed as such solvent. In the FOSS-LET modification the sample is extracted with tetrachlorethylene and specific gravity is measured by a thermostatically controlled electronic device with digital read-out. The reading is converted direct into oil or fat per cent using a conversion chart (1)

Remarks

The accuracy of this method is limited by the availability of the solvent, incomplete extraction, and by the magnitude of the decrease in specific gravity on solution of fat which is determined by difference between specific gravity of solvent and fat and nature of solution. The producers of FOSS-LET claim a standard deviation ± 0.21% from Soxhlet Method in the range of 4–14% fat. The determination is rapid, allowing 8–10 tests per hour (1). Total cost for necessary equipment (including balance with ± 0.19 accuracy, waste container and waste bin) is amounting to about S£ 20,000.

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APPENDIX

[illegible] METHODS FOR THE [illegible]MINATION OF TEXTURE IN FOODS

1. Introduction

In many foods texture is a primary criterion for consumers acceptance, and there is a need of methods for measuring food texture. Sensory assessments - organoleptic tests - may provide the evaluations closest to that used of the consumer, but such methods will always have the weakness of being dependent upon willingness and attitude of the tester. They may also be very expensive to run as routine tests.

There is, therefore, a need for objective methods of accurate and reproducible texture measurements, and a large number of instruments have been developed for such purposes (7, 9, 15). The instruments either measure the force required to cut, to compress, to shear, or the total work of compression or pulling the test material apart. A survey of the different methods will here be given.

2. Shear press methods

Principle

The consistency of a product is measured by first compressing and then shearing the sauple. This application of combined force stimulates the action of human teeth while chewing.

Equipment

Instruments as the tenderometer (8), the maturometer (11), and the Kramer shear press (6) work on this principle.

Procedure

a. The tenderometer.

The grid assembly of the tenderometer stimulates jaw action in that the lower and the upper sets of grids are hinged together. In contrast to mouth parts, here the lower set remains stationary while the upper grids rotate from the common hinge. The sample - placed between the two sets of grids - is first compressed and then sheared with parts of the material extruded ahead of the rotating grids. Power is provided by means of an electric motor and a hydraulic

system. The force required to shear the sample is shown by a pointer synchronized with counter weights.

b. The maturometer

A group of 25 rods travels through the mass of sample until they press through matching holes in the bottom of the cylindrical cup. Power is applied by hand through the rotation of a handle and gears with force down the cylinder, to the bottom of which the rods are attached. Force required is indicated on a hydraulic gauge which is attached to the top of the cylinder.

c. The Kramer shear press

The basic unit here consists of a hydraulic drive system for moving of a piston at any predetermined rate of travel. Power is obtained from a gear pump driven by an electric motor. Measurements of force is provided by the compression of a proving ring dynamometer, and different rings are available capable of providing ranges from a maximum of 100 pounds for relatively soft materials to 6000 pounds for hard products.

Remarks

The tenderometer is widely used all over the world (5, 12) to measure raw pea quality. It is precisious and gives a high correlation with alcohol-insoluble solids of the processed product. In many countries, at present, the price for peas to farmers is calculated according to the tenderometer value at the moment of harvesting (12). The maturometer is also developed for measuring the maturity in peas Its use, however, has been limited to Australia (10) and to South Africa (13).

The shear press is a more multipurpose instrument. It has been applied to the measurement of texture of a variety of products (14).

3. Penetration methods

Principle

The pressure required to cause a plunger to penetrate into a material is used as a measure of texture, and special penetrometers are constructed for such measurements.

Equipment

A complete penetrometer as delivered from Sommer & Runge KG, Berlin, consists of:

1. Penetrometer stand comprising: penetrometer table, bracket support, indicator assembly.
2. Penetration apparatus comprising: penetrator, plunger, load weights.

Procedure

The penetrator is adjusted on top of the sample surface, the switch clock is wound up and the lever released. By this procedure two operations will be controlled:

1. The penetrator will drop freely for the present period of time, and
2. at the end of penetration time there will be an automatic stop of the dropping motion.

As a general rule, the selected penetrator shall be allowed to penetrate into the sample under specified test conditions and over a specified period of time. The penetration depth is read in tenth of a millimeter from a graduated indicator dial (1).

Remarks

Penetrometers are widely used for the determination of fruit maturity (4). A specially designed Ridgelimeter has been developed to measure the firmness or "sag" of a jelly (2). Using a formula with specified proportions of ingredients, this method is used to determine the effect of pectin (pectin grade). The "sag" is then converted to grade strength of the pectin by reference to a graph calibrated against the apparatus.

4. INSTRON - measurements

Principle

The Instron apparatus for food textural measurements is an integgated test system that can be adapted to shear press, compression or penetration functions. By sensitive electronic force measuring and selectable testing speeds it can perform all common food texture tests.

Remarks

Unfortunately the Instron Model 1026 which the Center has purchased for textile testing cannot be used as a compression machine except by the use of special compression cages which are not suitable for food testing. Instron Limited recommends a special model (Instron 1140) for food testing. This is a compression machine supplied with Kramer Shear Cell, Magness Taylor Probe, compression Anvils, Back Extrusion Cell and Warner Bratzler Meat Shear. The results are recorded on a built-in synchronously driven recorder. Compared with other tests, Instron gives the most accurate and reproducible results. The incorporation of electronic load weighing with strain gauge load cells allows forces occurring throughout sample deformation to be accurately sensed. The recorder gives a complete plot of specimen behaviour rather than a single peak value.

In food research Instron measurements are recognized as a reference method for evaluation of texture and today most food research institutes are equipped with this instrument. (3)

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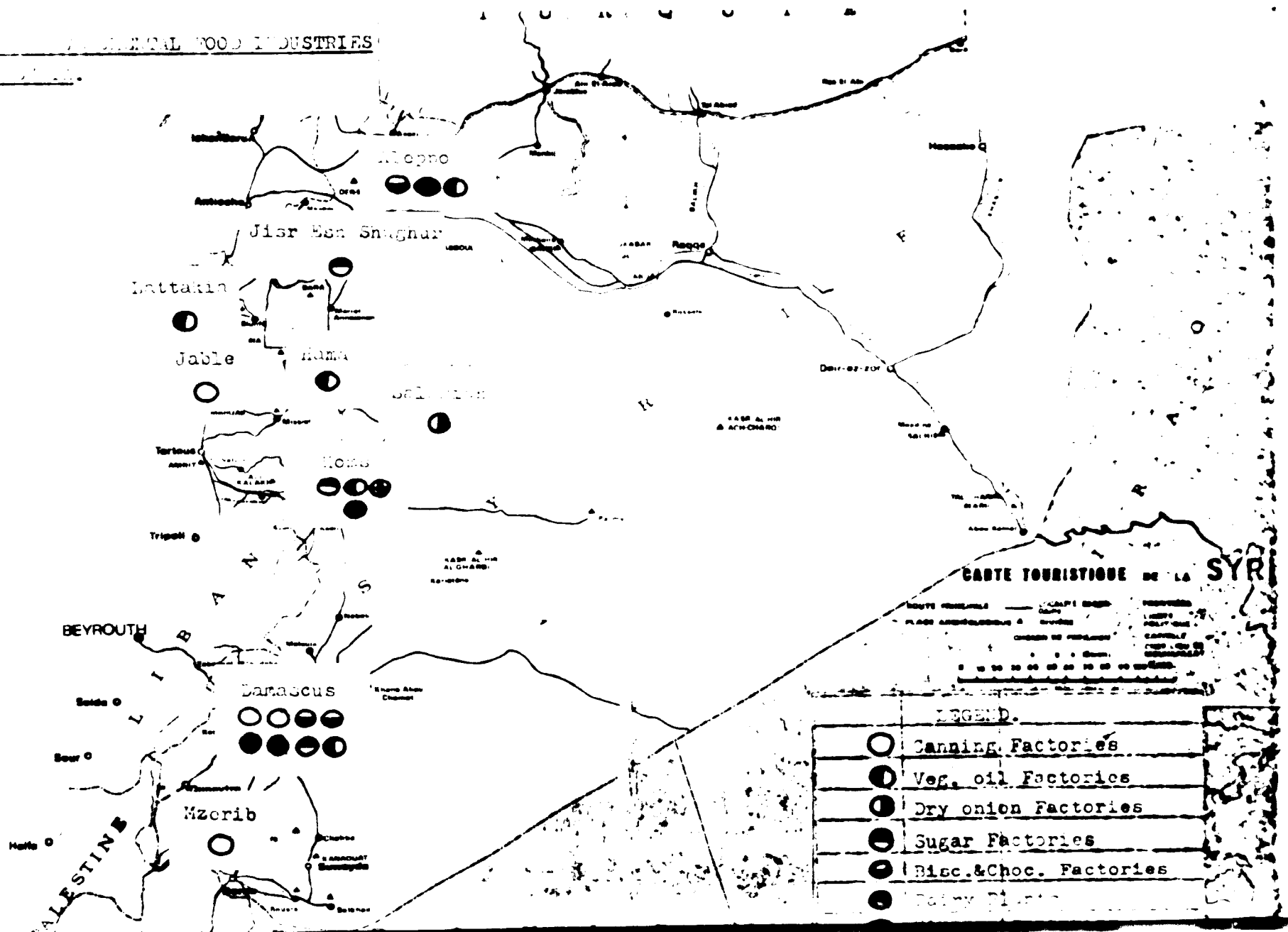
...ICAL FOOD INDUSTRIES



Summary of Technical Report

A Recommended Program of In Plant Quality Control for the Modern Conserves and Agricultural Industries, Damascus.

At the request of the Union of Food Industries a comprehensive program of quality control for the production of various canned food products has been prepared by the Expert. The program comprises recommendations for control of raw materials, production steps and final products and also guidelines for a proper control in the production of empty cans. The latter program includes technical specifications and quality control of tinplates, specifications and control of can laquers, specifications and control of can lid linings and a recommended system for routine control of can seams.

To perform this program, a special Manual of Laboratory Methods for Quality Control in the Syrian Canning industry has been prepared. The manual gives suitable methods for determination of drained weight, enzyme activity, pH, total acidity, soluble solids, total solids, moisture in dry products, salt content and SO_2. Recommendations are also given for incubation tests, vacuum measurements in cans, microscopic examination of can contents, sanitation control, organoleptic control of food products and for control of tinplates, can laquers, can lining compounds and can seams.

Recommended types of forms for reporting the control results are also illustrated.

Through Production Manager and General Director of the IRDC, the recommended program is presented to the Union of Food Industries and it is attached in extenso to the Mid Term Report from the Expert.

RECOMMENDATIONS FOR SUITABLE METHODS OF FOOD ANALYSIS AT THE INDUSTRIAL RESEARCH AND DEVELOPMENT CENTER

1. Introduction

The food industries in Syria are ameliorating a wide variety of raw materials, such as fruit and vegetables, meat, milk, sugar, oils, cereals, etc. Numerous methods of analysis have been proposed for the evaluation of quality in those raw materials, in its processing and in the final food products. Some of these methods have been adopted by national and international associations as official reference methods. Others may serve as convenient routine methods where the official methods are too time consuming, too complicated or too expensive to run.

In the following recommendations are given for a series of unofficial methods of food analysis suitable for use at the Industrial Research and Development Center. In each case reference is given to an official recommended method. In cases where numerous official and unofficial methods exist special surveys have been prepared to clarify the situation. These surveys are appended to this report.

Here recommendations are given for general methods of food analysis and for special methods for analysis of meat and meat products, fruit and vegetables foods and for the canning of food products. A bibliography is attached comprising references to control methods in the dairy industry, the oil and fat industry, the sugar industry and in the cereal industry.

2. General Methods

2.1 Microbiological methods

Generally, the methods and procedures listed in Recommended Methods for the Microbiological Examination of Foods (1) are recommended for use at the Center. Equipment is ordered to be able to perform most of the techniques mentioned in this book.

Attention should be paid to Survey of Methods for the Enumeration of Microorganisms (13) prepared by the Expert. Of these methods it is, at the present stage, recommended to apply only smear counts and dilution methods. If in future, however, the Center would deal with routine analysis of water, equipment should be ordered for membrane filter counts.

For sanitation control, a series of methods are developed (8), of which the Agar Sausage Method for Sanitation Control, listed in Manual of Laboratory Methods for Quality Control in the Syrian Canning Industries (11) is recommended at the present stage. For special purposes, however, many of the other methods mentioned in the survey should be considered.

2.2 Chemical methods

As a general rule, methods listed in references 2, 3, 5 and 15 should be chosen. Those are official recognized methods and should be used as reference in evaluating food standard and for other official use. However, for routine purposes, several rapid, inexpensive and convenient methods are available. Some of these are given a detailed description in references 11 and 12.

For moisture determination drying methods are recommended as methods of reference. For routine examination the Ultra-X determination should be chosen. Equipment for this method (Ultramat, Aust & Hackmann No. 429105) is ordered through UNIDO, and a detailed method description is given in reference (12).

The Harasta Institute has newly bought equipment for moisture determination according to the method of Karl Fischer (6). Efforts should be taken to get cooperative experience in using this equipment.

Determination of water activity in foods is of great importance. A survey of methods for such determination has been prepared by the Expert (9). For routine purposes the Filter Strip Hygroscopic Method (12) is recommended. For more accurate determinations the Dew Point Measuring Method mentioned in the survey (9) should be applied. At the moment, however, the Center should give such an investment a rather low priority.

Ash is most commonly determined according to muffle furnace heating methods. However, with the Ultramat the residue after moisture determination can be treated for routine ash determinations (12). Accessories for performing such tests are ordered through UNIDO.

Determination of protein is recommended as quoted by Pearson (15), an official method also known as Macro-Kjeldahl Method. Protein can also be determined by subtraction after measuring moisture, fat and ash by the Ultra-x method (12). This is a convenient and rapid method and should be applied where not so accurate results are required.

A special survey is made over methods for fat determinations in solid food products (10). Besides the common known and official recognized method of Soxhlet (2), many new and more convenient methods are now developed. With the Ultra-X equipment, a fat extraction may be done after drying to constant weight (12) and a special equipment is developed (FOSS-LET) for rapid determinations in solid products (10). This equipment seems very well suited for determining the oil content in oil seeds, and the Syrian oil processing industry should be given an opportunity to become acquainted with this equipment.

Determination of chloride(salt) should either be according to a mercuric nitrate titration method (16), a silver nitrate titration method (4) or to a potentiometric method officially recognized by A.O.A.C. (2). Attention should also be paid to a rapid method using a special patented titrator method, listed in the Manual of Supplementary Methods (12). At the moment, however, the vendor of the device (Ames Co.) Elkhart, Indiana, USA) is on the Arab boycott list.

The most common preservatives in food, sorbic acid and benzoic acid should be determined according to methods given in the Manual of Supplementary Methods (12). References for these methods are given by Welcher (17) and by A.O.A.C. (2).

2.3 Other methods

To complete and to assess the microbiological and chemical methods of analysis, a sensory evaluation of food quality is often applied. A sensory judgement - or organoleptic control - is a direct reflection of the consumers acceptance of a product and, therefore, of great importance to the food industry. A brief review of methods for organoleptic control of food products is given in Manual of Laboratory Methods for Quality Control in the Syrian Canning Industry (11). Of these methods the Triangle Test is recommended as a convenient and reliable test for evaluating differences in quality between food samples.

Food texture may be tested by sensory methods but also by using different instruments specially designed for giving objective reproducible indications of the consistency of a food sample. A survey of such methods is prepared by the Expert and appended to this report (14). If, in the future, the Center will be dealing with problems concerning food texture, the Instron 1140 is recommended purchased. Unfortunately, the Instron 1026 purchased for textile testing cannot be used for food testing.

3. Special methods for meat and meat products

3.1 Microbiological methods

American Public Health Association gives special recommendations for the microbiological examination of meat and meat products (1). These should be followed. Besides, attention should be paid to the chapter "Meat and Meat Products" in the Survey of Laboratory Methods for Detecting Spoilage in Food and Food Products (7) prepared by the Expert. Of these methods the Filter Strip Resazurin Method for Detecting Spoilage is recommended as a rapid method of estimating microbiological quality in meat and meat products. The method is described in the Manual of Supplementary Methods (12).

3.2 Chemical methods

Determination of nitrite in meat and meat products is described by A.O.A.C. (2). Besides a method using nitrin (2-aminobenzalphenylhydrazon) should be given consideration. The method is described in detail in the Manual of Supplementary Methods (12).

In chopped and ground meat product a fat determination according to a modified Gerber Method is recommended as a rapid and convenient technique. The method is discussed in the survey over methods for fat determinations (10) and is given in detail in the Manual (12). For in plant control of fat in meat mixtures, attention should also be given to a non destructive gravimetric method developed at the Danish Meat Research Institute mentioned in the survey (10). The amount of free fatty acids in meat fat gives an indication of its grade of rancidity. According to official methods, free fatty acids are determined by potassium or sodium hydroxide titration (2). The rancidity in animal fat can also be determined according to a rapid method described in the Manual of Supplementary Methods. (12). This is a convenient method when time is short and laboratory facilities are limited. It should supplement and not replace the official recognized methods.

4. Special methods for fruit and vegetable products

4.1 Microbiological methods

Here, the methods listed by American Publich Health Association (1) should, first of all, be supplemented by the **Howard Mould Test**. This is a method described and officially recognized by A.O.A.C. (2). Equipment for performing this test is ordered through UNIDO. As this method needs an experienced technician to give reliable results, measurements should be taken so that training in use of the Howard mould cell will be included in the fellowship programme of Mrs. Abeer Khaznadar.

For routine examinations of the microbiological quality of tomatoes for processing, the Fuchsin-SO_2 test described in the Manual of Supplementary Methods (12) is recommended.

4.2 Chemical methods

Determinations of SO_2 should be according to the principle of Monier Williams, recommended by A.O.A.C. (2). However, as this is a time-consuming and rather complicated method, a simpler technique is proposed for routine control purposes. The method is described in control in the [illegible] Manual of Laboratory Methods for Quality Control in the Syrian Canning Industries (11).

The enzyme activity in blanched vegetables should be checked by determining the amounts of catalase or peroxidase present. A rapid method for the detection of peroxidase activity is given in Manual of Laboratory Methods for Quality Control in the Syrian Canning Industries (11), while more accurate methods for both catalase and peroxidase are detailed by A.O.A.C. (2). The rapid method is recommended for industrial process control and the two other methods for determination of enzyme activity where a high grade of accuracy is required.

In addition to the methods already mentioned, the Manual of Supplementary Methods of Food Analysis (12) contains a few methods recommended in more special fields of fruit

and vegetable processing: Gravimetric determination of maturity (vegetables for freezing or canning), determination of lye solution strength (lye peeling of vegetables) and a tough string test for canned green or wax beans. Methods for determination of pectic substances are also given.

5. Special methods in food canning

In Manual of Laboratory Methods for Quality Control in the Syrian Canning Industries (11), various methods for process and product control in food canning are outlined. Recommended procedures are given for incubation tests, vacuum measurements in cans, microscopic examination of can contents, and control of tinplates, can lacquers, lid lining compounds and can seams.

For use at the Industrial Research and Development Center, equipment for calculation and control of the effect of sterilization (F_o-value) in canning has been ordered. (Ellab Automatic F_o-value computer with accessories). A detailed method for using this equipment is given in the Manual of Supplementary Methods (12). However, practical running of the instrument customarily requires some instructions, and it is recommended that Mrs. Abeer Khaznadar during her fellowship should be made familiar with the operating of the F_o-value computer.

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APPENDIX II

SUPPLEMENTARY LABORATORY METHODS OF FOOD ANALYSIS FOR USE AT THE INDUSTRIAL RESEARCH AND DEVELOPMENT CENTER, DAMASCUS

Content

- Ultra-X determination of moisture, fat, ash and protein.
- The filter strip hygroscopic method for determination of water activity in food
- Determination of NaCl using Quantab chloride filtrator
- Determination of sorbic acid in food.
  Determination of benzoic acid in food.
- The filter strip rezazurin method for detecting spoilage in meat and meat products.
- Determination of nitrite with "Nitrin"
- The Gerber method for fat determination in meat and meat products
- Determination of rancidity in animal fat.
- The Fuchsin-SO_2 test for detecting spoilage in fruits.
- Gravimetric determination of maturity in vegetables
- Determination of peeling lye solution strength
- Tough string test for canned green or wax beans
- Determination of pectic substances in food
- Methods of determining the effect of sterilization.

ULTRA-X DETERMINATION OF MOISTURE, FAT, ASH AND PROTEIN

Principle

A quick analysis instrument consisting of an infra-red lamp with built-in timer and a potentiometer is applied for determination of moisture. Upon extraction with carbon tetrachloride, the same instrument is used to estimate the content of fat. By use of a quick ashing equipment the amount of ash may be determined and in meat the content of protein is obtained by subtraction of moisture, fat and ash.

Equipment

Ultra-X analyzing scale with accessories
Quick Ashing apparatus with accessories
Sieve and Aluminium container for extraction of fat
Reagents: Carbon-tetrachloride
15% solution of magnesium acetate.

Procedure

A sample of approximately 100 g is taken and mixed thoroughly. 2.5 g are weighed on the scales so that the indicator arrives exactly at the 0 point. Necessary measuring temperature (voltage) and time are set and the infra-red lamp is switched on. The moisture content may be read off directly after drying to constant weight. This weight is referred to as the first reading. The weighing pan is removed from the scale and some carbon tetrachloride is poured over the dry sample. After an extraction of two minutes the liquid is sieved into the aluminium container. According to the character of the material, the extraction procedure should be repeated three to five times. The pan is then again placed under the lamp and subjected to heat until the pointer on the scale does not alter its position. The value now read is referred to as the second reading.
Ashing is performed by transferring the sample to the quartz shell of the Quick ashing apparatus, pouring 1 ml of the magnesium acetate solution over it and starting the apparatus. This process usually takes 5 to 10 minutes. The remaining sample is again weighed and this value is referred to as the third reading.

Interpretation of results

1st reading = % total water content+
2nd reading = % total water + fat content
3rd reading = % total water, fat and protein content

2nd reading - 1st reading = % fat content
3rd reading - 2nd reading = % protein
100% - 2nd reading = % fat free dry substance
100% - 3rd reading = % minerals (ash)

Water added to a meat product may be calculated as follows:
% protein x 4 = % given water content
% total water - % given water = % added water

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THE FILTER STRIP HYGROSCOPIC METHOD FOR DETERMINATION OF WATER ACTIVITY IN FOOD

1. Introduction

The water activity (Aw) represents one of the main factors in determining possibilities for microbial growth. To predict the stability of a food, it is, therefore, of great importance to be able to measure the Aw of that food. The following method was first published in 1963 (Kvaale, O. & Dalhoff, E.: 1963. Determination of the Equilibrium Relative Humidity in Food. Fd. Technol 17, 151) and has later undergone small modifications.

In principle, the method makes it possible to read the value of an equilibrium relative humidity by means of dry/wet changes in filter strips impregnated with different chemical salts.

2. Chemicals

$Pb(NO_3)_2$
KNO_3
$BaCl_2$
$ZnSO_4$
KCl
KBr
$(NH_4)_2 SO_4$
NaCl
$NaNO_2$

The salt should be of pro analysis quality and salts containing water of crystallisation should not be used. Such water can be removed by heating the chemical up to 300°C before use.

3. Production of filter strips

Each of the salts is transferred to a saturated solution, using distilled water, and strips of filter paper (Schencher & Schull No. 589, 2L Weissband or similary quality) are dipped in these solutions. After being exposed to the chemicals, the strips are allowed to dry in an atmosphere of 40% relative humidity or less. Strips not intended for immediate use, should be kept in a desiccator.

4. Procedure

The food sample is placed in a plastic petri dish. A series of test strips (one of each salt) of about 1 x 2 cm size are taped to the inner side of the lid of the petri dish,

The sample should be big enough to fill the dish almost completely. It is, however, very important that there is no contact between sample and test strips when the lid is placed on the dish. An unhomogenous sample should preferably be cut in small pieces or be minced before testing.

The lid and dish are sealed with 2-3 turns of plastic tape as to get an air tight container. The sample is then placed at 20°C and results are read after 20 hours of incubation.

5. Results

The results may easily be read without opening the air-tight Petri dish. Usually a visual examination is sufficient to judge whether the test strips are wet or dry. By knowing the Aw by which each paper changes from dry to wet, one gets a direct indication of the Aw of the sample. According to the procedure prescribed, the different salts will have the following values:

	Aw if wet
$NaNO_2$	> 0.75
$NaCl$	> 0.79
$(NH_4)_2SO_4$	> 0.82
KBr	> 0.85
KCl	> 0.87
$ZnSO_4$	> 0.90
$BaCl_2$	> 0.92
KNO_3	> 0.95
$Pb(NO_3)_2$	> 0.98

6. References

Kvaale, O. and Dalhoff E.: 1963. Determination of the Equilibrium Relative Humidity in Food. Fd. Technol. 17, 151

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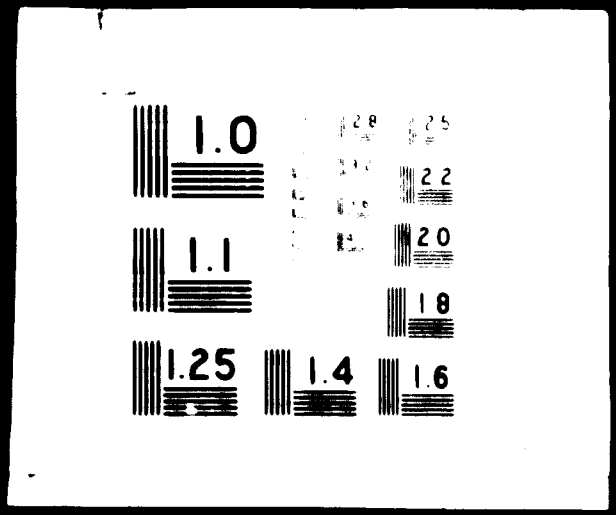

DETERMINATION OF NACL USING QUANTAB CHLORIDE TITRATOR

Principle

Quantab chloride titrator is a patented device for measuring salt directly in aquous solutions or in diluted aquous extracts of solids in the range 0.03% - 20.0% NaCl.
The filtrator consists of a thin, chemically inert plastic strip. Laminated within the strip is a capillary column impregnated with silver dichromate; a scale is superimposed on the capillary column. When the device is placed in an aqueous salt solution, capillary action causes the fluid to rise through the column of silver dichromate showing as a colour change.

Equipment

Quantab 1176 chloride titrator from Ames Co., Elkhart, Indianna, U S A

Procedure

A representative weighed or measured sample (10 g or 10 ml) is obtained by mixing, grinding or blending solid or semi solid products thoroughly before performing dilution and extraction. Aquous samples known to contain between 0.03% - 2% NaCl may be measured directly. For dilution and extraction the sample is mixed with 90 ml boiling water, stirred vigorously for 30 seconds and, after 1 minute, stirring for another 30 seconds to obtain a good extraction of salt from the sample into the solution. The mixture is then filtered and is ready for measuring.

The Quantab titrator is placed in the solution to be tested and the solution is allowed to saturate the capillary column completely. With a hot solution this usually takes about 10 minutes or less. Results should be read 30 seconds or more after a complete saturation of column.

Interpretation of results

The readings should be recorded to the nearest one-half division on the numbered scale at the tip of the white colour change, and is converted to per cent salt by using a calibration table given together with the Quantab titrator (NB! Each production lot has a special calibration table). If diluted, the value should be multiplied with the dilution factor (ten) to obtain the salt content of the sample.

DETERMINATION OF SORBIC ACID IN FOOD

Principle

The sorbic acid is distilled after being liberated by adding of sulphuric acid and magnesium sulphate. The distillate is measured against hydrochloric acid in an absorbance spectrophotometer at 263 µm

Reagents

1 M hydrochloric acid

0,01 M hydrochloric acid

$MgSO_4 \times 7\ H_2O$

0,05 M sulphuric acid solution

Sorbic acid standard solution prepared by dissolving 50 mg sorbic acid in a 100 ml volumetric flask containing 25 ml ethanol and dilute to volume with distilled water. Each ml contains 0,5 mg of sorbic acid.

Equipment

Beckman DB-GT UV-Spectrophotometer with Quartz cells and hydrogen discharge lamp.

Procedure

In a macro Kjeldahl flask 200 g $MgSO_4 \times 7H_2O$ and 200 ml of 0,05 M sulphuric acid are added to a sample containing between 0,50 to 2,00 mg of sorbic acid. The mixture is distilled on a Kjeldahl distillation rack until about 95 ml of distillate have been collected in a 100 ml volumetric flask.

The distillate is acidified with 1.00 ml of 1 M hydrochloric acid and diluted to volume with distilled water. The absorbance of the distillate is determined against 0,01 M hydrochloric acid at 263 nm. The absorbance of a comparable blank sample without added sorbic acid is determined and then substracted from absorbance of the sample. The micrograms of sorbic acid are determined by reference to standard curve. Standard curve is prepared as follows: Into a series of 100 ml flasks containing 1.00 ml of 1M hydrochloric acid, 0-1.0-2.0-3.0-4.0 and 5,0 ml of sorbic acid standard solution are pipetted, and it is diluted to volume with distilled water. Absorbance is determined and plotted against micrograms of sorbic acid on ordinary graph paper.

References: Welcher, F.J. (1966) Standard Methods of Chemical Analysis, Sixth ed., Vol. 3, Van Nostrand Reinhold Co., New York, p.111

DETERMINATION OF BENZOIC ACID OR SODIUM BONZOATE IN FOOD

Principle

There are two official recognized methods for the determination of benzoic acid, a titrimetric method and a spectrophotometric method.

The titrimetric method involves extraction of the benzoic acid from the food, conversion to benzoic acid, extraction of the latter with chloroform, evaporation of the solvent and titration of the acid with a base. Presence of vanillin interferes with this determination.

In the spectrophotometric method prepared solutions of benzoic acid is determined in a Beckman Model DU Spectrophotometer or equivalent, between 265 and 280 m . (the Beckman DB-GT at the Center is excellent for this purpose) the spectrophotometric method cannot be used on solid products and is limited to catchup and other tomato products, jams, jellies and other liquid products.

1. **Titrimetric method**

Reagents

0,05 N NaOH
10% NaOH-solution
$CHCl_3$ (Chloroform)
NaCl
Sat. solution of NaCl
HCl (1 + 3)
Ethanol (neutral to phenolphthalain)
Milk of lime (1 part of powder recently slaked $Ca(OH)_2$ suspended in 3 parts H_2O)

Procedure

1. 150 g of mixed or ground sample is transferred to a 500 ml volume flask, NaCl is added to saturation and the mixture is made alkaline (to alk. reaction on litmus paper) with 10% NaOH or with milk of lime. The sample is then diluted to mark with saturated NaCl-solution.
2. After frequent shaking during a 2 hrs standing period, the sample is filtered, a 100-or 200-ml aliquot of the filtrate is transferred into a separator, neutralized with HCl (1 + 3) and 5 ml of this acid are added in excess.
3. The acidified solution is extracted successively with 70, 50, 40 and 30 ml of $CHCl_3$. To avoid emulsions, rotary motion is advised when shaking during extraction.
4. The combined $CHCl_3$ extractions are transferred to a porcelain dish and evaporated at room temperature under a current of dry air.
5. The residue of benzoic acid is dried overnight in a H_2SO_4 desiccator and dissolved in 30-50 ml ethanol neutral to phenolphthalein.
6. 10 ml of H_2O are added to the solution and titration with 0,05 N NaOH follows.

Interpretation of results

1 ml 0,05 N NaOH = 0,0072 g anhydrous Nabenzoate.

Reference

A.O.A.C. (1965): Official Methods of Analysis, p. 450

2. Spectrophotometric Method

Reagents

Sat. solution of NaCl

Ether

HCl (1 + 1000)

0.1N NH_4OH

Procedure

1. 10 g of a mixed or ground sample are transferred to a separator and diluted to 200 ml with saturated solution of NaCl. Solution is made definitely acid to litmus with HCl and well mixed.
2. The prepared solution is extracted with successive 70, 50, 40 and 30 ml portions of ether during intense shaking to ensure complete extraction.
3. Mixture is drained and aqueous phase discarded, whereafter the combined ether-extractions are washed with 50, 40 and 30 ml portions of HCl (1 + 1000) and HCl washings discarded. If no purification is required, this step is omitted.
4. The ether solution is extracted with 50, 40, 30 and 20 ml portions 0.1% NH_4OH and the ether is discarded. The combined NH_4OH-extractions are neutralized with HCl and 1 ml acid is added in excess.
5. The acidified solution is extracted with 70, 50, 40 and 30 ml ether and combined ether-extractions are diluted to 200 ml with ether.
6. Absorbance is determined on this solution in well stopped cuvette in a Beckman UV spectrophotometer at predetermined wave lengths and with predetermined standard curves. Conc. of benzoic acid is determined from standard curve after corrections for dilutions. Acid x 1.18 = Na-benzoate.

References A.O.A.C. (1965) : Official Methods of Analysis, p. 450

THE FILTER STRIP RESAZURIN METHOD FOR DETECTING SPOILAGE IN MEAT AND MEAT PRODUCTS

1. Principle

In principle, the method is similar to the methylene blue reduction method for determining the microbiological and hygienic quality of milk. Here the blue coloured resazurin is used to measure the amount of reducing enzymes of microbiological origin in a given sample. Resazurin is reduced to the red coloured resorufin and further to the colourless dehydroresorufin. The speed of this reaction is a measurement of the amount of bacteria present. A rapid change in colour indicates a high bacterial load and a bad quality, while a slow change proves a good quality.

2. Chemicals and equipment

Tablets of resazurin (British Drug House)
Plastic bags (polythene)
Filter paper (Schleicher & Schull No. 589, Blau Band, or a similar quality)

3. Preparing test papers

4 tablets of resazurin are dissolved in 100 ml distilled water. The filter paper is dipped in this solution and then allowed to dry in a dark room. When dry, the paper is cut into test-strips of about 1 x 2 cm and packed in light-tight aluminium foil wrappings. Handling of the papers should be carried out with absolute clean fingers or with pincers, otherwise red finger prints are most likely to appear.

4. Procedure

By checking fresh meat a sample of about 1 kilo should be drawn, taken as to represent a good average of the lot to be examined. The whole sample is placed in a plastic bag and the bag is manipulated as to mix its content as well as possible. After thoroughly mixing for about one minute, the distribution of bacteria on the meat is anticipated to

be even, and the test may be carried out.

3 resazurin test-strips are dipped in distilled water and placed inside the bag in close contact with the meat. The strips should be handled with pincers and not with fingers. After an exposure to the meat for exactly one minute, the strips are placed between two sheets of plastic foils, air is squeezed away and the strips are placed in a dark place at about 20 - 22°C. The meat sample is not adulterated and can go back to the lot it was picked from.

The test strips are examined for change in colour by start and after 10, 30 and 60 minutes. The colour change should be given following interpretation.

Immediate change - not acceptable meat
Change in less than 10 minutes - bad quality
Change in 10-30 minutes - substandard quality
Change in 30-60 minutes - normal good quality
Change after more than 60 minutes - very good quality

Vacuum packed meat and meat products should be examined by opening the package, placing the test-strips on the surface of the product and expose the strips to the sample for exactly one minute. Interpretation of results should be as for fresh meat.

5. Discussion

The method gives very rapid but somewhat rough results. It should supplement and not replace the usual classical methods for counting bacteria in meat and meat products as set forth in Recommended Methods for the Microbiological Examination of Food. These methods are, however, rather time consuming and give results after two days only. For practical purposes where a rapid answer is of great importance, the resazurin method is very convenient.

The method suffers from the same inaccuracy and have the same limitations for use as the methylene blue reduction test for milk examination. Different species of bacteria have different reducing capacity and their environmental condition will also influence the results.

Other reducing substances present in meat will interfere with the results. If ascorbic acid or other antioxidants are added to the meat, the interpretations of results will have to be changed.

A 0.2% aquous solution of resazurin is stable when kept cool and in a dark place. The test-strips, however, are quite unstable and very sensitive to light.

Results from this method show better correlation with total bacterial count as measured by microscopic counting (live and dead bacteria) than with total bacterial count as measured by outgrowth on suitable media (live bacteria only). When interpreting the results, this should be kept in mind.

6. Reference

Simonsen, J.L.: 1965. Some quick methods for the quality control of meats. Proceedings 11th Meeting European Meat Research Workers, Belgrade.

DETERMINATION OF NITRITE WITH "NITRIN"

Principle

With nitrites, Nitrin (2-aminobenzalphenylhydrazon) forms a violet colour which quickly changes into yellow and dark yellow shades.

Equipment

Beakers
Glass cylinders
Volumetric flask
Warm water bath (60°-70°C and steam bath)
Balance
Reagents:

Absolute alcohol
10% hydrochloric acid
25% sulphuric acid

Reagent solution:
2.0 g of Nitrin (Merck reagent grade) is mixed with 4 ml of 10% hydrochloric acid and 100 ml of absolute alcohol is added. The mixture is heated in a warm water bath (60-70°C) to dissolve the Nitrin. During cooling, a minute amount of Nitrin becomes crystallized from the super-saturated solution. The remaining clear solution is used as a reagent. The reagent has to be kept in a dark bottle.

Procedure

Extraction of samples is done as described by A.O.A.C. (1):
5 g of finely comminuted and thoroughly mixed sample is weighed into a 50 ml beaker. Approximately 40 ml nitrite-free H_2O heated to 80°C is added and mixed thoroughly with a glass rod to break up all lumps and transferred to a 500 ml volumetric flask. The beaker and rod are washed thoroughly with successive portions of the hot H_2O, adding all washings to the flask. Enough water is added to bring the volume to about 300 ml, the flask is then transferred to a steam bath for 2 hours and occasionally shaken. 5 ml of saturated $HgCl_2$ solution is added and the mixture cooled to room temperature, filled up to the mark with nitrite-free H_2O and mixed again. The whole mixture is then filtered.

50 ml of the liquid sample, 30 ml of 25% sulphuric acid and 20 ml of absolute alcohol are measured into a suitable flask or cylinder. The contents are well shaken and 1 ml of Nitrin reagent solution is added to the mixture. In the presence of nitrite a more or less intensely coloured violet ring forms when the reagent is added. After subsequent shaking the liquid assumes an intense violet-red colour, which within a few minutes changes from red-brown and brown to yellow and sometimes dark yellow.

Interpretation of results

If the nitrite concentration is high, the violet colour appears only in the upper layer, since by shaking, the reaction mixture becomes yellow immediately. Extracts having high nitrite concentrations are suitable for colorimetric analysis when diluted 10 or 20 times. In this case, 50 ml of the diluted extract is used for colorimetric analysis at 520 µm

References

1. Assoc. of Official Agricultural Chemists: 1965. Official Methods of Analysis. 10th Ed., Washington, D.C., p. 347
2. Pfeiffer, H.: 1948. O-aminobenzalphenylhydrazon, ein spezifisches Reagenz auf salpetrige Säure und deren Salze. Z. Lebensmittelunters. U.-Forsch, 88, 287.

Rapid Method using Nitrin

A piece of sample of about 5 cm thickness is placed on a sheet of white paper. A filter paper wetted in distilled water is then pressed to the sample surface with a spatula. After 1 minute exposure 2 drops of 10% sulphuric acid and 2 drops of Nitrin reagent solution are placed on the filter paper. A violet colour shows presence of nitrite in the sample. The colour intensity gives an indication of the quantity of nitrite present. A point violet colour would indicate about 15 p.p.m. nitrite.

THE GERBER METHOD FOR FAT DETERMINATION IN MEAT AND MEAT PRODUCTS

Principle

The sample is digested with sulphuric acid in a butyrometer, the fat is separated and volumetrically measured. For all kinds of dairy products this technique is recognized as standard methods and special butyrometers are developed for cheese, cream and milk. In principle, all types of butyrometers can be used for fat determination in meat and meat products. The procedure and reading of results will, however, vary accordingly.

Equipment

Gerber centrifuge
Butyrometers
Water bath
Analytical balance
Gerber amyl alcohol
Gerber sulphuric acid

Procedure

1. With milk butyrometers

   2 g of sample are weighed into the butyrometer whereupon 10 ml Gerber sulphuric acid (s.g. 1.820 - 1.825) and 1 ml Gerber amyl alcohol are added. Hot water (70-80°C) is added to bring the level up to a point just below the rim. The butyrometer is shaken vigorously and placed in a water bath at 68°C for about 5-7 minutes, whereupon it is centrifuged in a balanced Gerber centrifuge at full speed for 3-4 minutes. The tubes is returned to the water bath for about 2 minutes and the fat volume is then read.

2. With cheese butyrometers

   3 g of sample are weighed into the beaker of a van Gullik butyrometer and 15 ml Gerber sulphuric acid are added through the thin end of the tube. This mixture is placed in a water bath at about 65°C for about 50 minutes until all particles of meat are dissolved. 1 ml of amyl alcohol is then added and then sufficient sulphuric acid to raise the level of the liquid to the mark 35 on the tube. The butyrometer is then

inverted about 10 times and centrifuged for 6 minutes at full speed, whereupon it is placed in a water bath at 65°C for five minutes.

Interpretation of results

Using milk butyrometers the fat content is calculated as follows:

$$\frac{\text{Butyrometer reading}}{\text{Sample weight in grammes}} \times 11.2 = \text{percentage of fat.}$$

Using cheese butyrometers the percentage of fat is read directly from the scale on the tube.

Reference

Talbot, A.: 1949. Rapid Estimation of fat in sausages and sausage meats. Analyst, 74, 462.

DETERMINATION OF RANCIDITY IN ANIMAL FAT

Principle

In the presence of ferrous chloride, oxidized or rancid fat reacts with ammonium thiocyanate to form a red colour, the intensity of which is proportional to the amount of rancidity. In this method, fat is primarily dissolved in chloroform

Many methods exist for determining the changes in fat commonly referred to as rancidity. In this method the oxidizing processes are measured only. The method is convenient and rapid. It should, however, supplement and not replace other laboratory methods.

Equipment

Filter paper (Whatman No. 1 or similar quality)

Reagents:

Chloroform, kept in a dark bottle, well closed.

Ammonium thiocyanate solution prepared by dissolving 8 g ammonium thiocyanate in 100 ml absolute alcohol

Ferrous chloride solution prepared by dissolving 0.8 g barium chloride ($BaCl_2$, $2H_2O$) in 50 ml distilled water to mix with a solution of 1 g ferrous sulphate ($FeSO_4$, $7\ H_2O$) dissolved in 50 ml distilled water. To the mixture 2 ml of concentrated hydrochloric acid are added, whereafter the solution is cleared by filtering. The solution is kept in a dark bottle, preferably refrigerated.

Procedure

A sample of fat is slowly temperated to 15° - 20°C. One piece of filter paper (1.5 x 4.5 cm size) is dipped in the chloroform and placed on the sample. When the chloroform is evaporated, a few drops of the thiocyanate solution are added to the paper, and, when evaporated, a few drops of the ferrous chloride solution. A blank is made by adding similar quantities of the solutions onto a chlorofrm wetted filter paper placed on a clean glass slide.

Interpretation of results

Rancidity is indicated by a red-brown colour clearly different from the colour of the blank. The final check of the two paper should be taken after 10 minutes.

Reference

Norsk Institutt for Næringsmiddelforskning: 1974. Laboratorie - metoder, Kjøtt og kjøttvarer, NINF, Aas Norway.

THE FUCHSIN - SO_2 TEST FOR DETECTING SPOILAGE IN FRUITS

Principle

BY microbial spoilage of fruits, acetylmethylcarbinol is formed. In this method acetylmethylcarbinol (and other aliphatic aldehydes) are determined by distillation through a filter paper impregnated with a modified Schiff reagent (SO_2 - decolourized leucofuchsin). In the presence of aldehydes, the red colour of fuchsin is restored.

Equipment

Conical flasks (100 ml)
Filter paper (Whatman No. 1 or equivalent)
Pipette (1 ml)
Reagents:

Basic fuchsin
Ethyl alcohol
Charcoal
Na_2SO_3 , 7 H_2O (or anhydrous)

0.2 g basic fuchsin are dissolved in 150 ml of 70% ethyl alcohol and neutralized with 4 g of Na_2SO_3, 7 H_2O (or 2 g anhydrous Na_2SO_3) dissolved in 100 ml distilled water. A little charcoal is added and the solution is filtered. The solution should now be almost colourless. If not, a few drops of the SO_2 solution are added. (N.B. surplus of SO_2 will weaken the reagent solution). When kept cool and in a dark bottle, this solution will keep for about one week.

Procedure

About 5 g (or 5 ml) of sample are mixed with 25 ml distilled water in a 100 ml conical flask, the mouth of which is covered with a filter paper fastened with a rubber string around the neck of the flask.

With a pipette one drop of reagent solution is placed in the

center of the filter paper and the sample is heated to light boiling. If the sample is spoiled, a bright red colour develops on the filter paper. Reading of results should be done after three minutes of boiling, and a blank (distilled water) should always be run as a comparison.

Interpretation of results

In raw tomatoes there seems to be a good correlation between organoleptical quality and colour change. Correlations with microbiological, chemical or biochemical methods of detecting spoilage have not been established.

The method may be made more sensitive by adding the sample to pepton water and incubating at 37°C for 4-6 hours before performing the test.

References

The method is developed by the Expert and has not yet been published. As to principle and background the following literature may be quoted:

1. Association of Official Agricultural Chemists: 1965. Official Methods of Analysis, 10th Ed., A O A C, Washington D.C., p. 297.

2. Fields, M.L.: 1964. Acetylmethylcarbinol and Diacetyl as Chemical Indicators of microbiological quality of apple juice. Fd. Technol. 18, (8), 114.

3. Pearson, D.: 1962. The Analysis of Foods, 5th Ed, J. and A. Churchill, Ltd., London, 262.

[illegible] 10 [illegible] DETERMINATION OF [illegible] IN VEGETABLES

Principle

In most cases the specific gravity of vegetables increases during the maturing process, and in a particular vegetable the specific gravity gives an expression of the maturity stage. Specific gravity may be measured by weighing a sample first in air and then in a liquid of known specific gravity.

Equipment

Balance supported on a stand or shelf
Basket made of wire netting
Glass beaker

Procedure

Approximately 100 g of sample are placed in the basket and weighed, first in air and then in the beaker filled with water of specific gravity = 1,00 (if necessary adjusted by adding a little salt)

Interpretation of results

$$\text{Specific gravity} = \frac{\text{weight in air} \times \text{specific gravity of liquid}}{\text{weight in air} - \text{weight in liquid}}$$

Based on such determinations, tentative standards for maturity grades of different vegetables may be stipulated.

References

Tressler, E.K. et al: 1968. The Freezing Preservation of Foods, Vol. 2, 3rd, 4th Ed., The Avi Publishing Company Inc., Westport, Connecticut.

DETERMINATION OF PEELING LYE SOLUTION STRENGTH

Principle

The lye solution is diluted and then titrated with sulphuric acid using phenolphthalein as an indicator.

Equipment

100 ml [illegible] with [illegible].

1[illegible] ml [illegible]

50 ml burette

10 ml pipette with bulb attachment

400 ml wide mouth glass bottle [illegible] handles

Reagents

1 N sulphuric acid

Phenolphthalein indicator solution

Procedure

A sample is taken from the peeling lye solution using a 400 ml wide glass bottle and the sample is cooled down to room temperature. With the bulbed pipette 10 ml aliquot is transferred to a 100 ml volumetric flask which is then filled with distilled water exactly to volume. The flask is stoppered and shaken thoroughly to mix.

10 ml of diluted sample is transferred to a 400 ml Erlenmeyer flask, 90 ml distilled water is added and then three drops phenolphthalein indicator. In lye solutions expected to contain less than 20% by weight of caustic soda, 50 ml of diluted sample is removed and 60 ml distilled water is added together with 5 drops of indicator. The solutions are then titrated from burette with 1N sulphuric acid until the indicator is colourless.

Interpretation of results

For lye solutions expected to contain more than 20% by weight of caustic soda (second dilution 1:9) the concentration is calculated according to following formula:

ml of acid x 40 = g/liter NaOH

Reference

Anonymous: 19[illegible]. Lye Peeling of Fruit and Root Crops, Diamond / Alcali Company, Cleveland, Ohio

[illegible] TOUGH STRING TEST FOR CANNED GREEN BEANS (Type I)

Principle

A tough string, is a string which will support the weight of 250 g for 5 seconds or longer when tested in accordance with the procedure described below. Strings are removed from individual pods, fastened through a clamp assembly weighing 250 g, and hung so that the string supports the entire weight. If the string supports the weight for five seconds or more, it is considered a tough string.

Apparatus

A [illegible] clamp (with teeth filed of or turned back) or spring operated clothes pin, with weight attached so that the entire assembly of weight and clamp weighs 250 g. A plastic bag containing lead pellets is also convenient as a weight.

Procedure

A representative sample of not less than 285 g is selected from the drained product. Individual bean units are broken and those that show evidence of tough strings are set aside. Strings are removed from pods and pod material is retained for weighing. The clamp assembly is fastened to one end of the spring while the other end is grasped by the fingers and lifted gently. If the strings break in less than five seconds, the broken parts that are longer than 13 mm are re-tested to determine if such portions are tough. The bean units which contain tough strings are weighed and per cent beans with tough strings is determined as related to the test sample.

Interpretation of results

Per cent by weight of pods containing tough strings equals weight of pods containing tough strings divided by weight of test sample multiplied by hundred.

References

Joint FAO/WHO Codex Alimentarius Commission: 1968.
Report on the 4th session of the Codex Committee on Methods of Analysis and Sampling, Berlin, Alinorm 69/23, para. 51.

DETERMINATION OF PECTIC SUBSTANCES IN FOOD

GRAVIMETRIC METHOD

Principle

The pectin is saponified to sodium pectate and the material precipitated with calcium, dried and the precipitate weighed.

Reagents

1 N sodium hydroxide
1 N acetic acid
1 N calcium chloride
Diluted solution of silver nitrate (0.1N)

Procedure

A sample of 50 g is homogenized, transferred to a 600 ml beaker and boiled in 1 hour. Water is added to compensate loose during boiling. The content is transferred to a 500 ml vol. flask and water (20°C) is added to the mark. The mixture is filtered through a rapid filter (e.g. Whatman No. 4) and 100 ml filtrate is transferred to a beaker. 100 ml water and 10 ml 1N NaoH are added and the mixture is allowed to stand overnight.

50 ml 1N acetic acid is added and the mixture is allowed to stand for 5 minutes. 25 ml 1N calcium chloride is then slowly added and the mixture stirred. The precipitate is allowed to stabilize by standing for 1 hour.

A filter paper (Whatman No. 4) is dried in an exicator overnight and then weighed.

The precipitated solution is heated to boiling and filtered through the weighed filter.

The filter paper is thoroughly washed in warm water until traces of chloride cannot be detected in the filtrate by adding of one drop silver nitrate.

The filter paper + content is dried for 3 hours at 105°C, cooled in an exicator and weighed. The drying is repeated for 1/2 hour to check if there has been any further loss of weight.

Interpretation of Results

The weight of substance on the filter paper is recorded as content of pectin in the sample. This method is recommended by Carré and Haynes (1922). It is claimed that it gives high results because of the pressure of nonuronide colloidial materials (M.A. Joslyn 1970). However, Newbold and Joslyn (1952) found the method to be reliable.

References

Carré, M.H. and Haynes D (1922): Biochem J., 16, 60

Newbold R.P. and Joslyn M.A. (1952): J. Assoc. Offic. Agr. Chemists, 35 872 and 892

M.A. Joslyn (1970) : Methods in Food Analysis, 2 Ed. Academic Press, London, P. 579.

OPTICAL ROTATION METHOD

Principle

The rotation of polarised light by pectin solutions is a characteristic property which can be used to determine the concentration of a pectin solution provided that the specific rotation of the pectin in the product to be analyzed is known.

Reagents

Copper sulphate solution made up of the following:

9.4 g $CuSO_4 \cdot 5\ H_2O$

27.29 $CH_3\ CH_2$ - $COONa \cdot 3H_2O$

12 ml glacial acetic acid

H_2O to one liter solution

Procedure

100 ml of a solution containing up to about 0,5% pectin is filtered through a rapid filter. The first 25 ml of filtrate is discarded and the optical rotation of the filtrate is then measured in a polarimeter in a 1-dm tube.

To 25 ml of this solution is added 25 ml of copper sulphate solution. The precipitated copper pectinate is filtered and the rotation of the filtrate is measured in a 2-dm tube. The difference between this and the rotation of the pectin extract is the rotation due to the pectin and is used in the calculation.

Interpretation of results

$$\text{Pectin \%} = \frac{\text{net rotation} \times 100}{\text{specific rotation of the pectin}}$$

Orange peel has a specific rotation = + 230.

References

M.A. Joslyn (1970) : Methods in Food Analysis, 2 Ed. Academic Press, London, p. 582.

METHODS OF DETERMINING THE EFFECT OF STERILIZATION

Principle

In the determination of a safe heat process for canned food, two methods may be used: experimental pack procedures and calculation methods. The experimental pack procedure involves the inoculation of the canned food with bacteria of known heat resistance, processing at different levels of time and/or temperatures, and determining the degree of spoilage by incubation or sub-culturing. Today this method is in very little practical use.

The calculation method involves knowledge of heat penetration data for the process, knowledge of thermal death data for bacterial spores, and of methods of correlation of these data. This principle for determination of heat processes is widely used in the food canning industries.

Heat penetration is measured by placing thermocouples inside cans during processing, their tip located at the point in the can having the slowest temperature rise. Impulses from these thermocouples are transferred to automatic or semi-automatic temperature recorders, and heat penetration curves are thus produced.

The thermal death time for a bacterial spore is defined as the time required to kill all the spores in a given substance at a stated temperature. This value may be determined by experiments heating given amounts of spores at a given temperature for different periods of time and looking for subsequent outgrowth of live bacteria. When thermal death times for different temperatures have been established, a thermal death-time curve for the particular organism may be plotted.

If such values are plotted on a semilogarithmic paper (using the log scale for the time and the linear scale for the temperature) the thermal death time curve will present itself as a straight line. The curve can thus be drawn if two points are known, or if one point and the slope of the curve are known.

References to thermal death time curves are given for most spore forming bacteria. Usually the curves are defined by

$F_{121^{\circ}C}$ which is the thermal death time of 121°C (250°F) and by

z which is the slope of the curve, the number of degrees C (or F) required for the curve to transverse one logarithmic cycle.

The correlation of the heat penetration data and the thermal death time data can be achieved according to three different methods:

1. The Graphical Method, Bigelow (1920)
2. The Formula Method, Ball (1923, 1928)
3. The Nomogram Method, Olson and Stevens (1939)

The graphical method is founded on the fact that each point on the heat penetration curve of a can of food represents a lethal value for the organism studied and involves the construction of a lethality curve. This lethal rate is the reciprocal of the number of minutes required to kill a given organism at that temperature, as read from its thermal death time curve. Using the heat penetration data a lethality curve can be drawn. When the area beneath the lethality curve is equal to unity, the process is considered to be adequate with respect to the organism studied. This area will have to be determined by a planimeter or by other methods of area integration.

When lethal rates are calculated from a thermal death-time curve with $F_{121^{\circ}C}$ = 1 minute and with a slope (z) having an intercept difference of 10°C, the sum of lethal rates is usually called the F_o- value. The F_o-value of a process is then the number of minutes required to destroy a specified number of spores at 121°C (250°F) when z = 10°C (18°F).

The formula method developed by Ball makes it possible to apply any given heat penetration data and thermal death time to any can size or retort temperature provided the thermal death times and the heat penetration rates approximate straight lines when plotted on semi-logarithmic papers.

Olson and Stevens have simplified the application of this method by nomograms enabling the calculation to be carried out graphically. The references give complete details of these principles.

Practical application of the graphical calculation method.

Equipment

Electrical temperature recorder with accessories (thermo-electric couples, cables, pressure tight junctions, etc.)
For the Center, an LLLAB automatic temperature recorder with 6 measuring applicators for stationary sterilization is ordered. This instrument is also supplied with an automatic F_0-value computer.

Procedure

The thermocuples may be mounted in the sides as well as in the ends of the test cans. These couples are nonprojecting so they can be mounted in the can end before the can is filled with food, and the sealing of the lid may be done in the ordinary way. The instruction for penetration of cans and fitting of the couples should be carefully observed.

The retorts for heat penetration tests should be equipped according to the guidelines laid down in"Recommended Syrian Regulations for Good Manufacturing Practice in the Processing of Low Acid Foods in Hermetically Sealed Containers" prepared by the Expert. For details concerning fitting thermocouple leads through the retort shell, see "Laboratory Manual for Food Canners and Processors", Vol. I, p. 205.

By the equipment ordered, up to five cans may be tested simultaneously and the test cans should be placed at different sites in the retort (front, rear, top, middle, bottom). One thermocouple should always be placed in the retort water, outside the cans.

The ILLAB automatic temperature recorder registers temperature values for each test can once a minute. If using a semi-automatic recorder, the temperature of each channel should also be measured manually once every minute.

Interpretation of results

The ILLAB equipment ordered for the Center has an inbuilt automatic F_o-value recorder, where the sterilization effect is shown continuously during the process. When F_o-values have to be calculated by the operator, the following rapid method may be applied:

The lethal rates of temperatures below 100°C are insignificant and may be omitted. The lethal rates for each 1/2 degree centigrade above 100°C are given in the table below:

°C	L	°C	L	°C	L	°C	L	°C	L
100	0.0080	105	0.0245	110	0.0770	115	0.245	120	0.776
100.5	0.0088	105.5	0.0277	110.5	0.0880	115.5	0.277	120.5	0.880
101	0.0097	106	0.0309	111	0.0977	116	0.309	121	0.977
101.5	0.0110	106.5	0.0349	111.5	0.1103	116.5	0.349	121.1	1.000
102	0.0123	107	0.0389	112	0.1230	117	0.389	121.5	1.103
102.5	0.0139	107.5	0.0439	112.5	0.1390	117.5	0.439	122	1.230
103	0.0155	108	0.0490	113	0.1550	118	0.490	122.5	1.390
103.5	0.0175	108.5	0.0553	113.5	0.1750	118.5	0.553	123	1.550
104	0.0195	109	0.0616	114	0.1950	119	0.616	124	1.950
104.5	0.0220	109.5	0.0696	114.5	0.220	119.5	0.696	125	2.450

The lethal values for each minute on the heat penetration curve are then simply added. This calculation method gives only an approximate estimation of the actual sterilization effect. For practical purposes, however, the method is very convenient. Calculations may be performed during the sterilization and the retorting may thus be regulated to the F_o-value required by the particular process.

The following table gives an example of such a calculation, leading to a F_o-value of 10.6:

TIME (MINUTES)	TEMP. oC OUTSIDE CAN	TEM. oC INSIDE CAN	LETHAL RATES	F_o VALUE
0	30	50		
1	35	50		
2	41	50		
3	50	50		
4	60	51		
5	72	57		
6	86	66		
7	100	82		
8	119	100	0.008	
9	122	113	0.155	
10	120	119	0.616	
11	120	120	0.776	
12	120	120	0.776	
13	120	120	0.776	
14	120	120	0.776	
15	120	120	0.776	
16	120	120	0.776	
17	120	120	0.776	
18	120	120	0.776	
19	120	120	0.776	
20	120	120	0.776	
21	118	120	0.776	
22	105	119	0.616	
23	95	117	0.389	
24	86	114	0.195	
25	78	110	0.077	
26	72	102	0.014	10.606
27	66	94		
28	60	87		
29	56	80		
30	62	75		
31	48	70		
32	44	66		
33	41	62		
34	38	59		
35	36	56		

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ANNEX 12

RECOMMENDED SYRIAN REGULATIONS FOR GOOD MANUFACTURING PRACTICE IN THE PROCESSING OF LOW ACID FOODS IN HERMETICALLY SEALED CONTAINERS

A. Definitions

"Aseptic processing and packaging" means the filling of a commercially sterilized cooled product into presterilized containers, followed by aseptic hermetical sealing, with a presterilized closure in an atmosphere free of microorganisms.

"Bleeders" means openings used to remove air, that enters with steam from retorts and steam chambers and to promote circulation of steam in such retorts and steam chambers. Bleeders may serve as a means of removing condensate.

"Coming-up-time" means the time which elapses between the introduction of steam into the closed retort and the time when the retort reaches the required processing temperature.

"Commercial sterility" of food means the condition achieved by application of heat which renders such food free of viable forms of microorganisms having public health significance, as well as any microorganism of non health significance capable of reproducing in the food under normal nonrefrigerated conditions of storage and distribution.

"Headspace" of a container is the vertical distance between the level of the production (generally the liquid surface) in the upright container and the inner surface of the lid.

"Hermetically sealed containers" means a container which is designed and intended to be secure against the entry of microorganisms and to maintain the commercial sterility of its contents after processing.

"Incubation" means the holding of a sample at a specified temperature for a specified period of time before examination.

"Initial temperature" means the average temperature of the contents of the coldest container to be processed at the time the sterilizing cycle begins, as determined after thorough stirring or shaking of the filled and sealed container.

"Lot" means the product produced during a period of time indicated by a specific code.

"Low-acid foods" means any foods, other than alcoholic beverages, with a finished equilibrium pH value greater than 4.6 and a water activity greater than 0.85 and also includes any normally low-acid fruits, vegetables or vegetable products in which for the purpose of thermal processing the pH value is reduced by acidification. Tomatoes, pears and pineapples, or the juices thereof, having a pH of less than 4.7 and figs having a pH of 4.9 or below shall not be classed as low-acid foods.

"Minimum thermal process" means the application of heat to food, either before or after sealing in a hermetically sealed container, for a period of time and at a temperature scientifically determined to be adequate to ensure destruction of microorganisms of public health significance.

"Retort" means any closed vessel or other equipment used for the thermal processing of foods.

"Scheduled process" means the process selected by the processor as adequate under the conditions of manufacture for a given product to achieve commercial sterility. This process may be in excess of that necessary to ensure destruction of microorganisms of public health significance.

"Shall" and "should". As used in this connection, "shall" refers to mandatory requirements and "should" refers to recommended or advisory procedures or equipment.

"Vents" means openings controlled by gate, plug cock, or other adequate valves used for the elimination of air during the venting period.

B. Product Preparations

1. Incoming raw materials, ingredients and packaging components should be inspected upon receipt to ensure that they are suitable for processing. Raw materials should be received in an area separate from the processing areas. Prior to being placed in the inventory, ingredients susceptible to microbiological contamination should either be examined for microbiological contamination or should be received under a supplier's guarantee that they are of a microbiological condition suitable for use in processing of low acid food. The examination should either be performed at a factory laboratory or at the Industrial Research and Development Center. Products should be held prior to processing in such a manner as to minimise growth of microorganisms.

2. Blanching by heat, when required in the preparation of food for canning, should be effected by heating the food to the required temperature, holding it as this temperature for the required time, and then either rapidly cooling the food or passing it to subsequent processing without delay. The control of blanching recommended in: "A Recommended Programme of In Plant Quality Control" prepared by the Expert should be applied. Where the blanched food is washed prior to filling, potable water should be used.$

3. The filling of containers, either mechanically or by hand, shall be controlled so as to ensure that the filling requirements specified in the scheduled process are met. The control of filling weights and of filling temperatures as recommended in: "A Recommended Programme of in Plant Quality Control" prepared by the Expert should be applied

4. The exhausting of containers for the removal of air shall be controlled so as to meet the conditions for which the process was designed. This may be done by heat exhausting, mechanical exhausting, hot brining, or steam injection.

5. When normally low-acid fruits, vegetables or vegetable products require sufficient acidification to permit safe processing at low temperatures, such as in boiling water, there shall be careful supervision to ensure that the equilibrium pH of the finished product meets that of the scheduled process.

C. Establishing scheduled processes

Scheduled processes for low-acid foods shall be established by qualified persons having expert knowledge of thermal processing requirements for low-acid foods in hermetically sealed containers and having adequate facilities for making such determinations. Upon request from the processors, the Industrial Research and Development Center shall be able to establish heat sterilisation data based on scientific methods including process calculations based on product heat penetration data and data for microbial thermal death time.

The type, range and combination of variations encountered in a commercial production shall be adequately provided for in establishing the scheduled process. Critical factors which may affect the scheduled process (e.g. minimum headspace, consistency, maximum drained weight, etc.), shall be specified in the scheduled process.

D. Operations in the thermal processing room

1. Retort room

There shall be suitable and sufficient area around each retort so that baskets containing processed and non-processed cans may be placed separately without any risk of mixing. Moreover, all baskets containing non-processed cans shall be plainly and conspicuously marked so that they may be easily distinguished from baskets containing processed cans.

Schedules of the thermal processin[illegible] for the various products in question shall be readily available to the retort operator and to officially appointed inspectors.

Suitable time recording equipment shall be available (a pocket or wrist watch is not considered satisfactory).

2. Background for the use of pressure retorts

The internal pressure from entrapped gas is sufficient to distort some types of glass or aluminium containers or damage their seams. To avoid this, products in aluminium or glass containers is heat processed in special retorts using compressed water under sufficient pressure to balance the pressure inside the container. At the end of the heating process the product is cooled sufficiently to reduce the internal pressure before the pressure on the retort is released.

3.. Thermometers

Each retort shall be equipped with at least one mercury - inglass thermometer with a minimum scale length of 150 mm and with scale divisions for each 1°C.

The thermometer should be fitted directly into the shell of the retort so that the whole of the bulb is inserted into the retort. If this is not possible, the thermometer may be installed in a well attached to the shell in such a way that the bulb protrudes at least 50 mm from the inside surface of the retort. The external well must be at least 100 mm wide and be equipped with a bleeder to ensure uniform circulation of the retort water or steam around the bulb.

A satisfactory position for the thermometer is half way up from the bottom of the retort. In the case of water filled retorts, the thermometer must be fitted so that the bulb is always under water during processing.

The thermometer shall be installed where it can be accurately and easily read. Its accuracy must be tested against a known standard accurate thermometer upon installation and at least once a year thereafter or more frequently as may be necessary to ensure its accuracy. A thermometer which deviates

more than 0.5°C from the standard or has a divided mercury column, must be replaced.

4. Temperature Recording Device (Thermograph)

Each retort shall be equipped with a temperature recording device adjusted to agree within 0.5°C of the known accurate mercury-in-glass thermometer. The chart graduations shall not exceed 1°C within the range 60°C to 130°C. The chart shall have a scale of 1°C per mm. The temperature recorder bulb shall be installed near to and in the same manner as the bulb of the mercury thermometer. A means of preventing unauthorised changes in adjustment of the recorder shall be provided.

The temperature recorder may be combined with an automatic temperature controller to comprise a so called recording temperature controller.

5. Temperature Controller

Each retort shall be equipped with an automatic temperature controller. This may be combined with a temperature recorder. If this is the case, it must be ensured that the bulb is so placed as to give correct temperature recording as well as satisfactory temperature controlling.

The servo-valve of the temperature controller should close with failing servo pressure. A fairly large shut-off valve should be fitted in shunt with the servo-valve in order to ensure a short coming up time, as well as to enable manual control of retort temperature if the temperature controller should fail during processing.

6. Pressure gauge (Manometer)

Each retort shall be equipped with a pressure gauge graduated in divisions of 2 pounds per square inch or less and which shows at least 60% above the certified pressure of the retort. The diameter of the pressure gauge shall not be less than 100 mm. It must be connected to the retort via a goose-neck and shall not be more than 100 mm above this. A test cock shall be fitted between the pressure gauge and the gooseneck.

7. Steam Inlet

This shall be large enough to give a short coming up time and to provide sufficient steam for proper operation of the retort. The steam shall be led in at the bottom of the retort in such a way as to give effective heat distribution. The spreaders shall therefore extend along the bottom for the entire length of the retort and have evenly spaced perforations along their top surface.

8. Pressure Controller

Pressure retorts shall be fitted with equipment that ensures satisfactory pressure control. In retorts where pressure is achieved by means of compressed air, an automatic pressure controller shall be fitted. A check valve preventing water from entering into the compressed air system shall be provided between the retort and the servo-valve of the pressure controller.

9. Safety Valve

Pressure retorts shall be fitted with a safety valve large enough to prevent any build-up of pressure in the retort, when the steam valve is turned on full for a period of 15 minutes.

10. Water Level Indicator

There shall be a means of determining the water level in the retort. Suitable equipment may consist of a gauge water or petcocks.

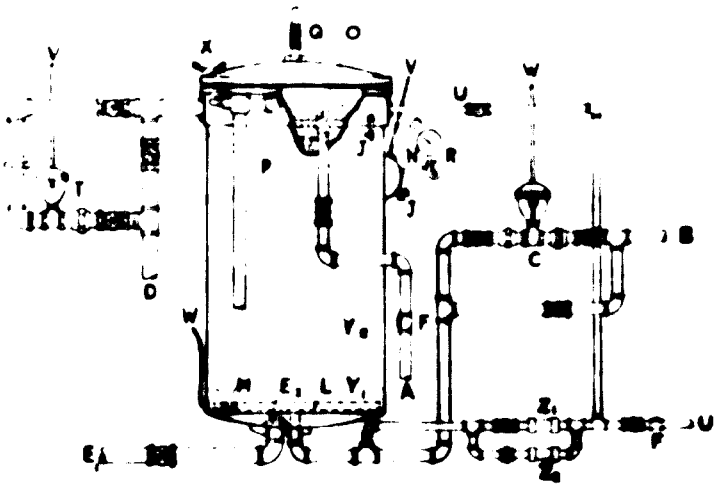
11. Cooling water inlet, cooling, etc.

Cooling water pressure and inlet must be such as to ensure correct and rapid cooling. In pressure retorts, pipe dimensions must be such that cooling to 60°-70°C can take place within 10 minutes. If the water pressure is not sufficient, pressure pumps must be installed. Cooling water shall be led into the retort through perforated pipes or other suitable arrangements, such that cooling is uniform.

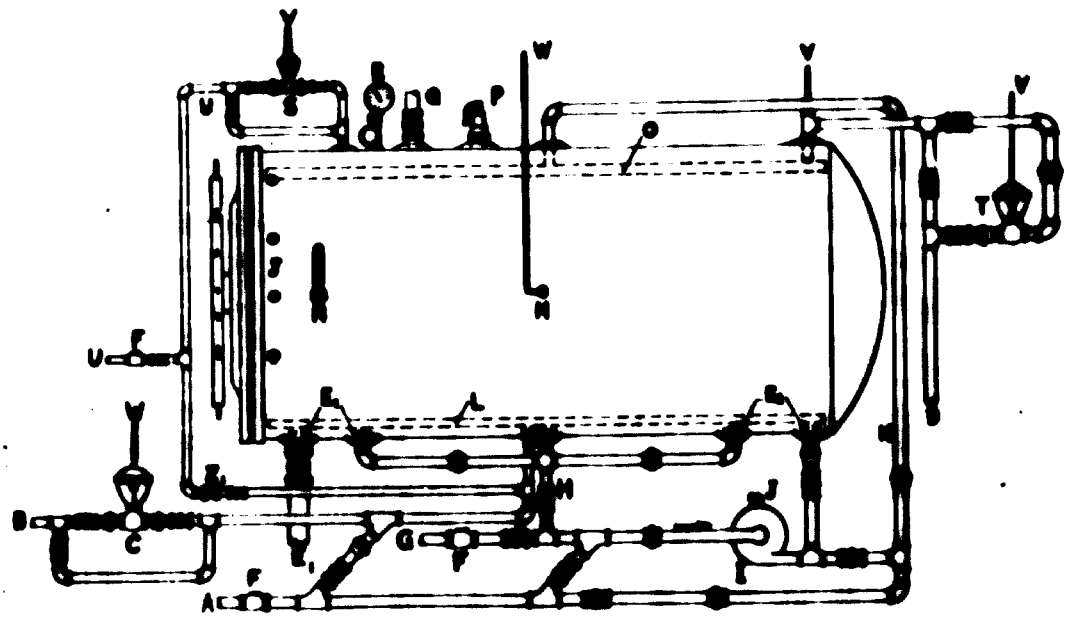
Vertical Retorts

B
R
P

I
J
K
L
M Temperature
N Thermometer
O Water sprinkler
P Safety valve
Q
R Pressure gauge
S
T Pressure control
U Air line
V To pressure control instrument
W To temperature control instrument
X Wing nuts—8 required
Y_1 Crate support
Y_2 Crate guides
Z_1 Constant flow orifice valve used during come-up
Z_2 Constant flow orifice valve used during cook



Horizontal Retorts



12. Retort Baskets

These shall be constructed of wire mesh, perforated sheet metal or other material which does not prevent satisfactory heat distribution in the retort. When perforated sheet metal is used, the holes should be at least 25 mm on 50 mm centers. Any dividing plates in the basket should be perforated as above.

Retort baskets in vertical retorts shall not stand on a "false" bottom, but be supported so that circulation around the baskets is satisfactory. The retort should have an arrangement for centering the baskets. There should be a space of about 60 mm between the baskets and the wall of the retort.

13. Inspection and Control

At all times it must be ensured that all valves, gaskets and other associated equipment in the retort plant are in full working order so that leakages do not occur.

Every retort plant and its equipment shall be inspected at least twice a year by officially appointed inspectors.

E. Containers

1. Closures. Regular observations shall be maintained during production runs for closure defects. Any such defects shall be recorded and corrective action shall be taken and recorded. The principle given in "Routine Control of Can Seams" prepared by the Expert, should be applied.

For closures other than double seams, appropriate detailed inspections and test shall be conducted by qualified personnel at intervals of sufficient frequency to ensure proper closing machine performance and consistently reliable hermetic seal production. Records of such tests shall be maintained.

2. Cooling

Container cooling water should be chlorinated as necessary by the processor so that there is a measurable free chlorine residual at the water discharge point of the container cooler. Other safe chemical or physical treatment which is equivalent to chlorination in its bactericidal effect may be used. Where pressure cooling is utilized, adequate pressure should be maintained for a time sufficient to prevent permanent distortion of the container.

3. Coding

Each hermetically sealed container of low-acid processed food shall be marked with an identifying code which shall be permanently visible to the naked eye. Where the container does not permit the code to be embossed or inked, the label may be legibly perforated or otherwise marked, provided that the label is securely affixed to the product container. The required identification shall identify in code the establishment where packed, the product contained therein, and the year and the day packed. If necessary to identify smaller lots, the code should be changed with sufficient frequency during one days production.

4. Postprocess handling.

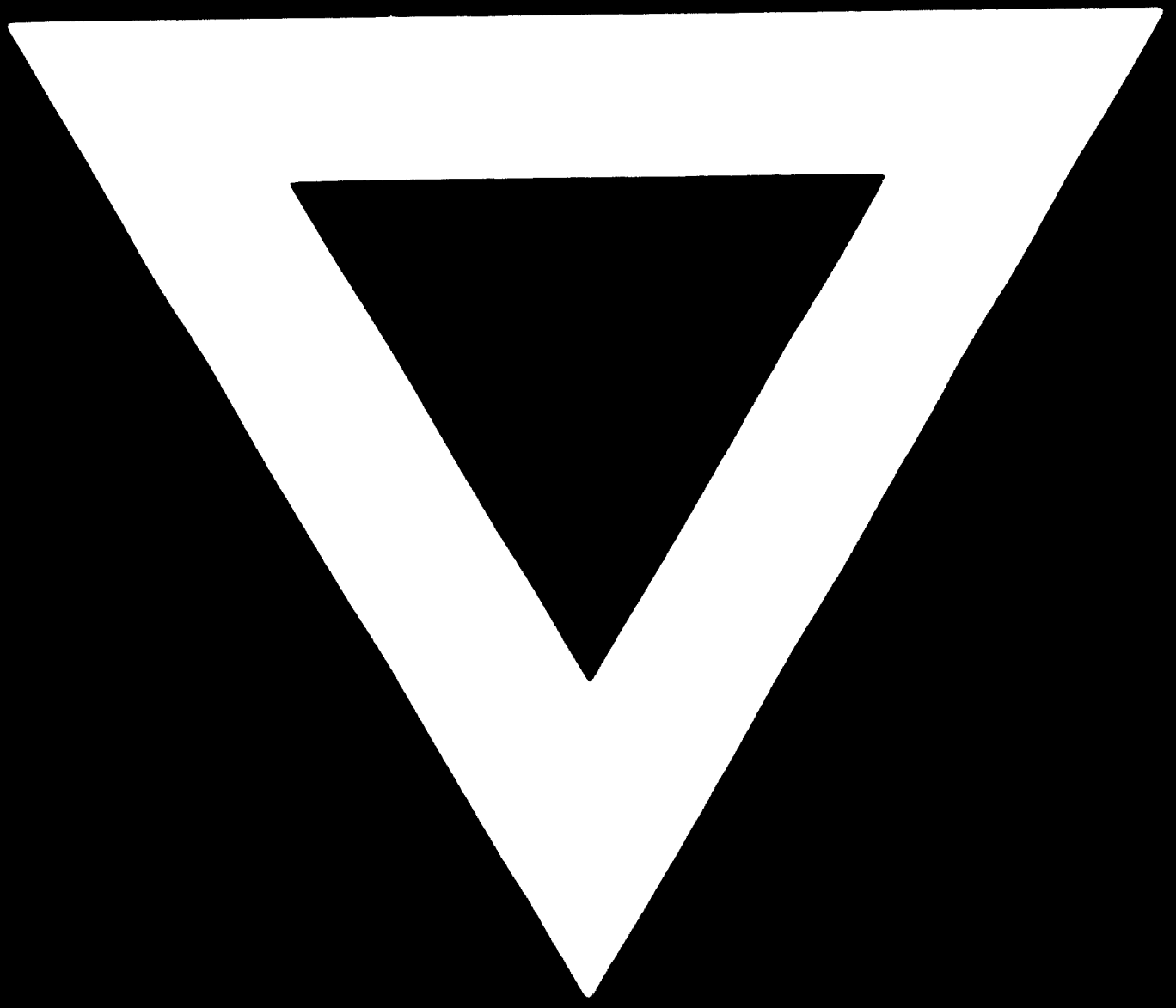
Where cans are handled on belt conveyors, such conveyors should be such constructed as to minimise contact by the belt with the double seam, i.e. cans should not be rolled on the double seam. All fracts and belts which come into contact with the can seams should be thoroughly scrubbed and sanitized at intervals of sufficient frequency to avoid contamination. Automatic equipment used in handling filled containers should be so designed and operated in such a manner as to preserve the can seam or other container closure integrity.

F. Deviations in processing

Whenever any process is less than the scheduled process for any low-acid food or container as disclosed from records, by processors check, or otherwise, the processor of such food shall either fully reprocess that portion of the production involved, keeping full records of the reprocessing conditions or, alternatively, shall set aside that portion of the production involved for further evaluation as to any potential public health significance. Such evaluations shall be made by the Industrial Research and Development Center and shall be in accordance with procedures recognised by international authorities as being adequate to detect any potential hazard to public health. Unless such evaluation demonstrates that the product had been given a thermal process that rendered it free of microorganisms of public health significance, the product set aside either shall be fully reprocessed to render it commercial sterile or it shall be destroyed. Either upon completion of full reprocessing and the attainment of commercial sterility or after the determination that no significant potential for public health hazard exists, that portion of the production involved may be shipped in normal distribution

G. Personnel

All operators of seaming machines and retorts shall be under supervision of a person who has had special theoretical and practical instruction in retort operation, processing systems operations, packaging systems operations and container closure inspections, and has been identified by the responsible for that instruction as having satisfactorily completed the prescribed course of instruction.



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