OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

DISCLAIMER

This document has been produced without formal United Nations editing. The designations employed and the presentation of the material in this document do not imply the expression of any opinion whatsoever on the part of the Secretariat of the United Nations Industrial Development Organization (UNIDO) concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries, or its economic system or degree of development. Designations such as "developed", "industrialized" and "developing" are intended for statistical convenience and do not necessarily express a judgment about the stage reached by a particular country or area in the development process. Mention of firm names or commercial products does not constitute an endorsement by UNIDO.

FAIR USE POLICY

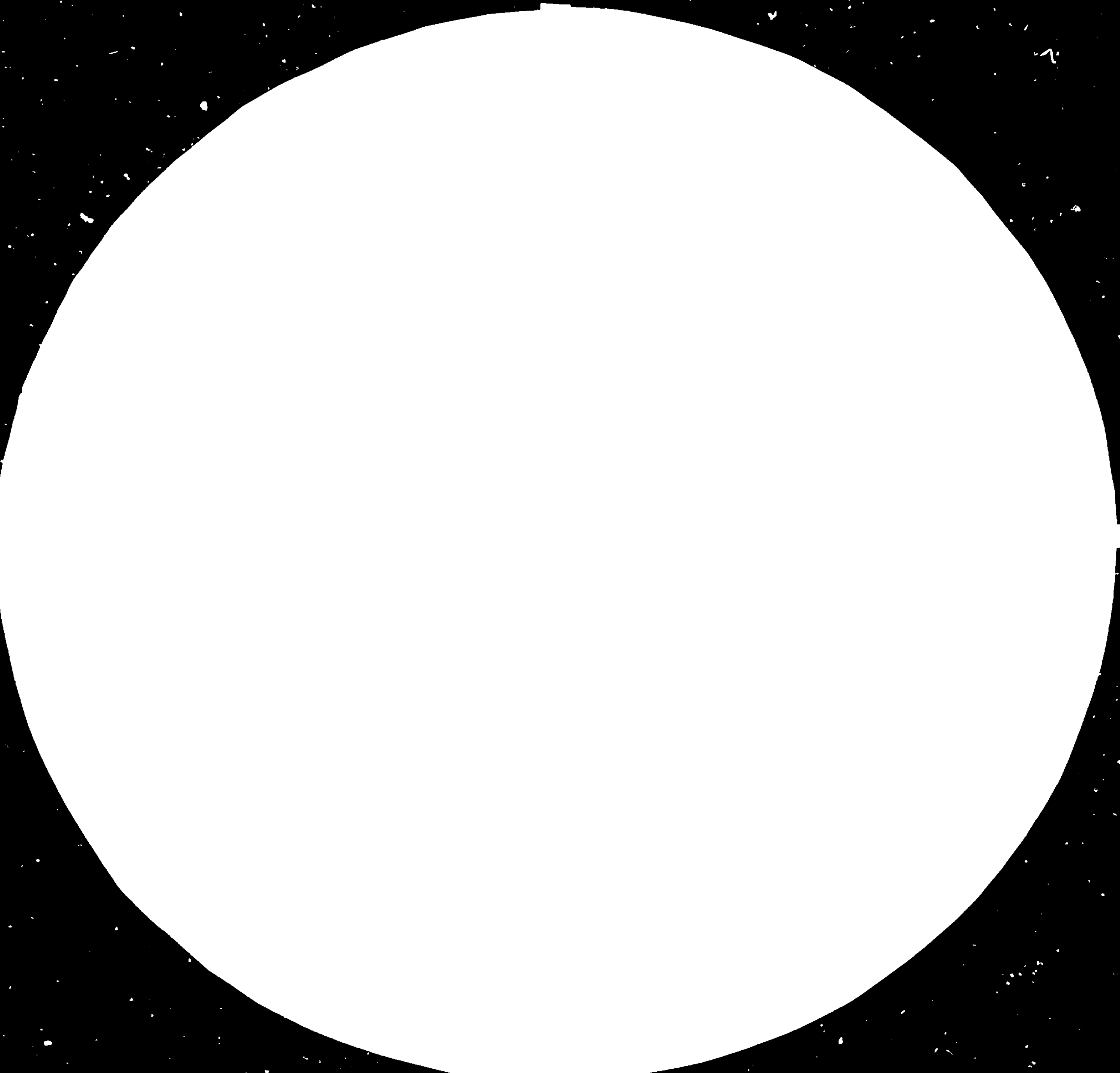
Any part of this publication may be quoted and referenced for educational and research purposes without additional permission from UNIDO. However, those who make use of quoting and referencing this publication are requested to follow the Fair Use Policy of giving due credit to UNIDO.

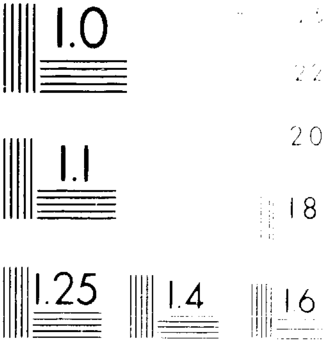
CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org




1.0
2.5
2.2
2.0
1.1
1.8
1.25
1.4
1.6

13262

5 December 1983
ENGLISH

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

Oman.

TRAINING ON POLAROGRAPHY, X-RAY FLUORESCENCE AND ATOMIC SPECTROSCOPY

DP/OMA/81/008
SULTANATE OF OMAN

Terminal report *

Prepared for the Sultanate of Oman
by the United Nations Industrial Development Organization,
executing agency for the United Nations Development Programme

Based on the work of Keith C. BURKE,
Expert in Fluorescence Spectrometry and Polarography

---

* This document has been reproduced without formal editing.

TABLE OF CONTENTS

REFERENCES:

LIST OF TABLES

APPENDIX I

APPENDIX II

APPENDIX II

NOTES

Abbreviations used in the text:

PPM = Parts Per Million, a microgram per gram, 0.0001%

PPB = Parts per Billion, a monogram per gram, 0.0000001%.

INTRODUCTION

This report describes the Chemical Section of the Central Laboratories on Wadi Shabb in the Sultanate of Oman. The Central Laboratories began in 1973 with temporary facilities located in the Ruwi business district. The first phase of the new laboratory was opened on 18 November 1980 and construction of the second phase began in June of 1983. The laboratories are divided into four technical sections plus administration. The four technical sections are for: Chemical Analysis, Physical Testing, Microbiology, and Assay of Precious Metals. A second gold and precious metals assay laboratory was dedicated 18 November 1983 in Salalah.

The Central Laboratories are part of the Directorate General of Specifications and Measurements (DGSM) under the direction of the Ministry of Commerce and Industry. Currently, 41 Omani specifications have been set up and are listed in Table I. The mission of the Central Laboratories is to make assessments on the quality of products being imported or manufactured in Oman as well as to monitor the environment for pollution.

To assist the DGSM the Ministry of Commerce and Industry requested an Analytical Chemist who is an expert in the fields of X-ray fluorescence spectroscopy, atomic absorption spectroscopy, and polarography from the United Nations Industrial Development Organization (UNIDO). The UNIDO expert arrived in Oman 10 June 1983 in order to strengthen and expand the Chemical Section of Laboratories in the fields of standard reference materials, quality control, as well as formal instructions in basic analytical chemistry and instrumental analysis. The project concluded after six months, as planned.

I. PROJECT PERSONNEL

A list of those attending technical lectures or demonstrations is given in Table II. A star beside a name indicates an Omani national. Attendance at the formal lectures was not mandatory but strongly recommended by the Director General. The number attending varied from 23 to a minimum of two. The average number in attendance was eight. Low attendance was in part due to annual leave, sickness in the family or because of heavy work load. The maximum number of employees are on annual leave between July and October. No lectures were allowed during the month of Ramadan (June and July).

Out of the twenty three who attended the lectures sessions, 30,4% were not affiliated with the laboratories or were university students working at the laboratory during summer holidays. There were 34.8% non-Omanis in attendance. A total of 8.7% from administration and 26.1% of those in attendance were working full-time in the Chemical Section of the Laboratories.

II. THE CENTRAL LABORATORIES

The Central Laboratories are physically divided into six areas:

1. Reception, Metrology, and the chemical and general storage area,
2. Administration which includes offices for the specification staff, the library and rest rooms; the technical sections which are dedicated to:
3. Chemical Analysis,
4. Physical Testing of building materials,
5. Microbiological testing of food stuffs, and the
6. Laboratory dedicated to Assay of jewlery and precious stones.

The Chemical Analysis Section is about one fourth of the total space. It has a total floor space at ground level of 231 square meters. The Chemical Section is roughly divided into large work areas each with four fume exhaust hoods located in the center of each area. Between the two large work areas are four smaller work areas designed for glass washing, electric furnaces, balances and the atomic absorption, as well as special apparatus and two supervisors desks. In addition to the eight main hoods which are placed back to back there are exhaust hoods for the electric furnace room and over the atomic absorption. The Chemical Laboratory provides high purity demineralized water for the other technical sections of the laboratory and has the capacity to provide this high purity water to outside sources.

Food stuffs and water analysis each take out half of the two main laboratory work areas. They consist of modern apparatus for the analysis of protein, fat and carbohydrates as well as the requisite hot plates and grinding equipment. The remainder of the laboratory space is dedicated to instrumental analysis.

The total cost of the Central Laboratories is US$ 2.3 million (excluding personal emolument). Of this sum the total cost of equipment was US$ 717,500. The cost of equipment associated with this project was US$ 243,950 for the X-ray spectrograph including spare parts, US$ 18,000 for the polarography and the 1974 cost of the atomic absorption spectrometer was US$ 28,700. All of the costs were paid for by the Oman government. The yearly service for an electronic service maintenance contract is about 12% of the equipment cost.

Appendix 1 describes the laboratories in general lines.

III. LABORATORY OUTPUT

Tables III and IV indicate the number and types of samples being analyzed by the Chemical Section of the Central Laboratories. The total number of samples analyzed for each classification does not reflect the amount of work required for the analysis of each sample. For instance, each water sample is normally analyzed for about 24 consitutients. Water analysis are normally made for the following: conductivity, pH, turbidity, total dissolved solids, residual chlorine, BOD, COD, calcium, magnesium, sodium, potassium, iron, manganese, sulfate, chloride, trace elements, alkalinity, hardness, nitrate, fluoride, carbonate, and appearance.

Similarly each cement sample is analyzed for about a dozen components, such as: silica, alumina, calcium oxide, magnesium oxide, iron oxide, sulfur trioxide, manganese oxide, sodium oxide, potassium oxide, titanium oxide, phosphorus oxide, chromium oxide, strontium oxide, zinc oxide, and loss on ignition.

The number of samples analyzed by the Chemical Section will double within the next five years; if the yearly rate of increase continues to grow as indicated by the number of analyses made in 1979, 1982, and the first quarter of 1983. The growth should be considerably larger than this prediction after markets are designated for instrumental analysis of organic materials.

IV. EQUIPMENT AND INSTRUMENTATION

The following modern laboratory equipment was purchased in 1981.

	FUNCTION	SOURCE
1.	X-Ray Fluorescence Spectrograph	Diano XRF 8565/75A
2.	Polarograph	EG+G Model 384
3.	Atomic Absorption (purchased 1976)	Pye Unicam SP1900
4.	Total Organic Carbon Analyzer	Beckman Tocamaster
5.	Ion Chromatography	Dionex Aution 12
6.	Infrared Spectrometer	Pye Unicam PS 3-050-080
7.	High Performance Liquid Chromatograph	Tracor Model 900-78
8.	Gas Chromatograph	Pye Unicam Series 204

Each of these instruments is installed and equipped with computerized data handling facilities. The X-ray spectrograph has the Digital PDP 11/03 and the atomic absorption is interfaced with the HP-97 programmable calculator. The polarograph computer has a 8085 CPU integrated into the system.

A microcomputer by Epson, the QX-10, was acquired in September of 1983 and will be used to inventory electronic spare parts, standard reference material and to summarize quality control data from the proposed system.

V. STANDARD REFERENCE MATERIALS

Only a limited number of standard reference materials (SRM) were on hand at the beginning of the UNIDO project. SRM's must be acquired and maintained in order to standardize or confirm the accuracy of an analysis.

Gratis SRM's were obtained, through the UNIDO expert, from the United States Geological Survey as well as the French-based International Working Group. These 34 gratis standards supplement the seven certified cement SRM's from the National Bureau of Standards and the eight secondary cements available at the Oman Chemical Laboratory. The secondary reference materials are required for routine analysis and quality control once a method has been firmly established.

A cross index listing the SRM's has been prepared to allow sorting of the reference materials according to:

1. Type
2. Source
3. Desination
4. Element
5. Element Concentration
6. Matrix

A cross index can be conviently be inserted into a computer data base.

Additional SRM's have been recommended and their purchase approved and their delivery is expected by early 1984. Once these materials are received they will be used to validate analysis as well as to establish a quality control programme. All of the SRM's recommended for this project can be used for atomic absorption, X-ray fluorescence, polarographic analysis as well as for traditional analysis. A very large number of organic SRM's are also required before adequate applications can be made with the infrared spectrophotometer or the gas or liquid chromatographs. These special organic reference materials must be in Muscat before other UNIDO experts can effectively demonstrate applications.

The gratis SRM's which are now in the Central Laboratories are geochemical in nature. They are required because the future applications will require analysis of rocks and ores. These reference materials will allow the laboratory to perform quick instrumental analysis for a wide variety of materials. The requisite instruments must be operational.

In the latter half of 1983 a need developed for SRM's to be used for the determination of trace consitutients in biological materials. Several materials have been recommended which will allow the certification of methods for mercury in fish as well as for arsenic, lead, selenium, cadmium, zinc, etc, in water and various food stuffs. These reference matcrials are especially important because the chemical decomposition processes may cause considerable loss of elements such as mercury, arsenic, and selenium.

VI. PRACTICAL DEMONSTRATIONS

Several modifications had to be made in the original work plan due to the lack of supplies required to demonstrate preconcentration techniques or the lack of instrumental spare parts. Several examples illustrate the quality of the work being produced in the Chemical Section of the Central Laboratories.

Mercury in Fish

When the Oman National Fisheries (ONF) were required to produce certificates indicating the mercury content of fish being shipped from Oman to Europe they requested analysis at the Central Laboratories. The request was made in July of 1983. Several techniques are capable of determining mercury in fish flesh but none of the requisite equipment was available in Oman. The determination could have been completed polarographically, if a gold electrode was ordered, or by a specific mercury vapor detector. The flame atomic absorption method is capable of analyzing microgram (PPM) quantities of mercury; however, the requisite mercury hollow cathode was not working. ONF was able to truncate purchase procedures and supply a mercury hollow cathode as well as a mercury cold vapor cell and the associated glassware for the atomic absorption determination of nanogram (PPB) quantities of mercury within two weeks. Analysis for several 50 ton shipments have been made and the fish was certified for its mercury content.

The liver and kidney of fish accumulate trace elements up to 500 times the amount found in fish flesh. Fish can live with up to 1 PPM of mercury in their body without adverse effects. The normal mercury level in unpolluted sea water is about 0.14 microgram per liter. Typical analysis for mercury in fish flesh is less than 0.1 PPM by wet weight. Analysis for mercury in Oman cuttlefish and sea bream was found to be less than 0.1 PPM. The European specifications were for less than 0.7 PPM of mercury. Table V gives an indication of the sensitivity of the cold vapor atomic absorption method for mercury in kingfish liver and kidneys. Analysis are also given for several other trace metals by the polarographic method to illustrate what could be accomplished. No standard reference materials were available when these analytical techniques were developed.

Trace Metals in Water

The polarograph was also used to establish the quality of drinking water. The data in Table VI were obtained by the differential pulse stripping polarographic technique for the analysis of zinc, cadmium, lead, and coper in drinking water. The method is reliable in the range from 0.02 to 3 PPM.

Independent Study Programme

Due to the lack of requisite materials a simple spectrophotometric problem was devised to enforce the analytical thinking for the development of analytical procedures. The national staff members who are trained in chemistry have not had special training in analytical chemistry and benefit from this study. The iron complex with 1,10-phenanthroline was set up as a series of practical problems to demonstrate the effect of pH, reductant, interferences, effect of wavelength, and range of linearity. The method is established as a back-up for atomic absorption, X-ray fluorescence, or polarographic analysis.

XRF Analysis of Cements

Direct analysis of cements by X-ray fluorescence (XRF) spectroscopy is the most convient and reliable technique. The data in Table VII illustrates the analysis of three standard reference materials as samples for three components. The values under XRF were averages of ten analysis obtained at the Central Laboratories. The values indicated by ± S indicate the one sigma value or standard deviation.

VII. FORMAL SEMINAR AND LECTURE PROGRAMME

The plan of lectures given at the Central Laboratories is given in Table IIX. The lectures were designed to instruct the staff in three special areas of instrumental analysis as well as the requisite background and special application needs of the laboratory staff. In addition to the topics listed below two months were devoted to special lectures on elementary topics of analytical methodology such as calculations, methods of expressing results, statistical evaluation of data, basics of physics and chemistry, techniques of dissolution, and general inorganic chemistry. All of the notes used for the lectures have been transferred to the Director of the Laboratories along with special technical literature on instrumentation and procedures.

IIX. ANALYTICAL PROCEDURES

The requisite chemicals and glassware were not received during the lifetime of the UNIDO project. Consequently demonstrations of special procedures for trace X-ray fluorescence, atomic absorption, or polarographic analysis could not be made. The subjects were discussed and detailed procedures for several techniques are given in the appendix. Method modification may be required for the special types of samples analyzed in the Central Laboratories. Literature citations for the complete procedures are described under the Reference Section of this report.

Procedure I) uses a 5% solution of TOPO in MIBK to concentrate elements prior to their determination by non-aqueous atomic absorption spectroscopy (1). Rapid X-ray fluorescence methods for tellurium (2), arsenic (3), and selenium (4), are given in the appendix under sections II, II, and IV, respectively.

These three procedures require dissolution of the sample, prefiltration to remove any precipitate such as silica, separation of colloidal element of interest (Arsenic, Selenium, or Tellurium) and counting the dry micropore filter with a vacuum X-ray spectrograph. An atomic absorption procedure is described for the determination of copper and nickel in organic food stuffs (4), see Section V in the appendix for details.

IX. CONCLUSIONS AND RECOMMENDATIONS

The foundations of a sound chemical laboratory has been laid with the well equipped Central Laboratories in the Sultanate of Oman. The combined national and expatriate staff work harmonously and are able to analyze a wide variety of materials. It will be a minimum of five years before the national staff is able to provide all services, efficiently. For instance, the modern electronic equipment now in the laboratory requires the services of a full-time electronics technician. Currently, this service is provided through an outside contract. The administration of the laboratory is rather efficient; however, it can not act independently without the services of other government agencies. The Central Laboratories should be able to do the following:

1. Develop a complete administrative infrastructure which would allow the in-hous staff to do its own accounting and purchasing. None of the requisite chemicals or glassware, required for this UNIDO project were received, apparently due to the complex ministerial infrastructure.

2. Eliminate the need for senior staff personnel to act as junior administrators by adding one technical clerk to the Chemical Section and one administrative assistant to the office of the Director. The physical size of the current laboratory will perhaps not allow staff expansion until the completion of phase II.

3. Continue to purchase adequate standard reference materials for the development of new procedures and the control of the accuracy of existing methods, especially trace elements analysis in water and food stuffs.

4. Establish a quality control programme within the Chemical Section which should include the use of 'blind standard reference materials', analyst standards, collaborative analysis with other laboratories for check analysis and frequent reanalysis of analyzed materials.

ad 4) The quality control programme should increase the laboratory work load by about 10%.

5. Purchase an induction heating furnace with thermo-conductivity or infrared analyzer for the analysis of carbon dioxide and perhaps the readout might be for both carbon and sulfur if the analysis of metals and ores are required for the future development of the laboratory.

6. Allow qualified staff members to attend technical conferences and seminars once a year to keep the staff informed on new analytical techniques. The Royal Chemical Society hosts meetings on atomic spectroscopy every two years in the UK and the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy is held during the first week in March every year; both meetings are especially recommended. Staff members attending will be required to write reports on their findings and to present a verbal seminar on the topics of general scientific interest for the other members of the staff.

7. Subscribe to abstracting journals such as Chemical Abstracts (Analytical Section) and/or Analytical Abstracts. Proper reading of these journals will decrease the need to subscribe to special journals dealing with electrochemistry, chromatography, X-ray fluorescence, etc.

8. Complete sets of the Royal Chemical Society's (UK) books titled the Annual Reports on Analytical Atomic Spectroscopy should be ordered as well as the Perkin-Elmer Corporation's (USA) journal titled Atomic Spectroscopy. The total shelf space would be just less than one meter for both journals. The cost of the former is US$ 75 per year and the latter is US$ 20.

9. Set up an Oman general training programme for laboratory technicians as operators for analytical instruments. Such a technician training programme could follow completion of secondary school and begin with an in-service programme for the students in the various industrial, hospital, and governmental laboratories. The programme could function through the university now being formed and perhaps be assisted by UNIDO.

10. Current Central Laboratory nationals could broaden their technical background by working for a few months in other laboratories such as the Sohar copper refinery laboratory, the X-ray laboratory for the cement plan near Risail and perhaps in a clinical laboratory. A formal study/work programme would be required and perhaps could be designed through UNIDO.

11. Scholarships for university training in analytical chemistry as well as in business administration are required at the B. S., M. S., and PhD levels. Summer holidays should be spent in laboratories outside of Oman.

12. Install safety showers with floor drains and requisite safety equipment in the Phase II section of the Central Laboratories for those areas of the laboratory using acids or dangerous chemicals. No access to the laboratory should be allowed unless safety glasses are worn, so extra glasses should be provided for visitors.

13. Set up a safety committee for routine inspection of the laboratories for good laboratory practices to insure that the fire exits are not blocked, and that every staff member has had practical experience with the use of a fire extinguisher.

14. Continue the monthly seminar presentation by each staff member on current topics of analytical chemistry as well as group discussions of current laboratory problems.

15. Install at least one exhaust hood especially for oxidations with perchloric acid (such as Alberdene Stone) or purchase the requisite fume control glassware so this important analytical acid can be safely used. Refer to G. F. Smith Company in the USA.

16. Do not purchase additional accessories for the current atomic absorption spectrometer, such as the hydride attachment or the electrode discharge lamps. Such applications, especially those requiring ultraviolet wavelengths should be attempted on a new instrument or perhaps a combination ICP (inductively coupled plasma) and atomic absorption unit.

17. Obtain and apply the gold electrode for the polarographic determination of mercury in fish as well as an auxillary electrode stand for this electrode and the glassy carbon electrode which is now in the laboratory.

18. Set up a preventive maintenance schedule to clean and check all instruments routinely. Each operator must keep records on instrument performance. It is generally a good practice to keep instruments always turned on if they might be sensitive to humidity.

19. Set up a formal programme to make use of employees spare time when the sample work load is low. The supplies, reagents, and SRM's recommended by the UNIDO expert will allow the following activities to be developed:

    a. Trace analysis of selenium, arsenic, and tellurium by X-ray fluorescence analysis. The preconcentration techniques could also be applied to polarographic analysis when sub-PPB analysis are required.

    b. Preconcentration of lead, etc, by solvent extraction for either polarographic or atomic absorption analysis.

    c. Direct X-ray fluorescence analysis of food stuffs for major metals.

d. Major and minor elements in cements and geological materials by atomic absorption as a back-up for X-ray fluorescence.

e. Direct analysis of geological materials by X-ray fluorescence, as well as application of the fusion method to prepare glass beads. Neither the requisite apparatus or crucibles are on order to prepare the glass beads.

f. Polarographic analysis coupled with atomic absorption analysis for trace metals in Oman sea water, ocean sediment, and atmospheric dust will produce base line data for future pollution studies.

g. Analyze the two International Working Group reference materials for major, minor, and trace consitutients and report the data to IWG by the end of the first quarter of 1984.

h. Analyze the International Atomic Energy Authority's new water reference material for trace consitutients by polarographic and atomic absorption methods and report the data to IAEA by the end of the second quarter of 1984.

i. Analyze building aggregates for sulfate and chloride by X-ray fluorescence.

j. Analyze Oman fish flesh for lead, cadmium, arsenic, zinc, copper, manganese, zinc, and selenium.

20. When new equipment is purchased the manufacturer normally installs the equipment, gives brief operating instructions and supplies a free short course on special techniques of operation and numerous applications. These application courses are three to five days in length and should be made available to the <u>operator</u> within four to six months of installation. The operator(s) selected for this study programme must be fluent in English because the programme will either be in the UK or USA. Ideally the manufacturers programme would be coupled with a short course in analytical instrumentation plus a few weeks working in a government laboratory or industrial laboratory. The instrument manufacturer ideally would suggest a laboratory to demonstrate their equipment which is being used routinely.

21. Restructure the Chemical Stores area of the Central Laboratory to separate the dry chemicals from the flammable organics and the corrosive acids. Perhaps this will be easy after completion of Phase II.

22. Reports from the laboratory should indicate the type of sample as well as the number of consitutients being analyzed. Each staff member should prepare a weekly or mothly report to indicate work activity, problems, and future activities. The supervisor will then be able to summarize the work activities for the periodic administrative reports.

23. An adequate data storage system (Hard Disk) is required for the laboratory microcomputer.

24. Reduce the exhaust flow for the atomic absorption hood system in order to eleminate flame flicker. Clean the optical system and replace corroded mirrors in the atomic absorption system now in the laboratory.

25. Begin analysis for poisonous pesticide residues which are found in water, fish and horticultural crops. The pesticide peril causes 370,000 poisonings a year in the third world countries.

26. Obtain pheno-fabric (or any material which does not give a signal in the radiation region of interest) to make correct size mask for the sample support for the XRF.

27. Obtain a shatter box or proper mixing mill to blend and homogenize samples for the XRF.

28. Add a CRT (cathode ray tube readout) to the Dyano XRF to improve the speed and readability. The CRT would function as a print out device when the printer is out of service and allow the XRF to be programmed and data obtained.

29. Activate the Dyano XRF "Sample Spin" mechanism.

30. Obtain "Air Pollution Abstracts" and "Selected Water Resources Abstracts".

31. Obtain a wrist action shaker.

32. Improve the exhaust velocity of the hood for the muffle furnace room.

33. Test plastic bottles used to store "Tanuf" drinking water for trace metals that might be stripped into the water from the plastic, especially: lead, arsenic, cadmium, chromium, tin, and mercury where the limit must not exceed 0.05 mg/liter.

34. Make use of safety straps for all gas cylinders.

35. Keep a daily log of the variation of temperature and humidity within the laboratory.

REFERENCES

1. Keith E. Burke, Analyst 97, 19-28 (1972).
2. Keith E. Burke, M. M. Yanak, C. H. Albright, Anal. Chem. 39 14-18 (1967).
3. C. H. Albright, Keith E. Burke, M. M. Yanak, Talanta 16, 309-313 (1969).
4. Keith E. Burke and M.M. Yanak, Anal. Chem. 41 (7) 963-965 (1969).
5. Keith E. Burke and C.H. Albright, Journal of the Association of Official Analytical Chemists 54 (3), 658-662 (1971).

TABLE I

OMANI SPECIFICATIONS

Designation	Title
1/1977	Precast concrete blocks
2/1982	Aggregates from natural sources for concrete
3/1977	Cement flooring titles
4/1978	Building sands from natural sources
5/1978	Wheat flour
6/1978	Methods of testing wheat flour
7/1979	Ordinary Portland cement
8/1978	Drinking water
9/1979	Non-alcoholic carbonated beverages
10/1980	Non-alcoholic carbonated beverages-Sampling
11/1981	Mineral aggregates, sands fillers-Sampling
12/1982	Mineral aggregates, sands and fillers - Determination of grading by sieve analysis
13/1981	Concrete-Determination of compressive strength of test cubes
14/1979	Drinking water-methods of sampling
15/1979	Drinking water-Determination of lead, selenium, and arsenic contents
16/1979	Drinking water-Determination of cadmium and cyanide contents
17/1979	Drinking water-Determination of luoride and nitrate contents
18/1979	Drinking water-Determination of copper and iron contents
19/1979	Drinking water-Determination of total hardness calcium and magnesium contents
20/1980	Drinking water-Determination of Manganese and zinc contents
21/1980	Drinking water-Determination of Chloride and Sulfate contents
22/1980	Drinking water-Determination of phenolic compounds (as phenol) content
23/1980	Drinking water-Determination of total dissolved solids and residual chlorine contents and pH range
24/1981	Drinking water-Methods of vacteriological examination
25/1979	Methods of testing cement-Chemical Tests
26/1981	Methods of testing cement-Physical Tests
27/1980	Non-alcoholic carbonated beverages-Preliminary examination and determination of total acidity and sodium carbonate contents
28/1980	Non-alcoholic carbonated beverages-Determination of carbon dioxide content
29/1980	Non-alcoholic carbonated beverages-Determination of sulfur dioxide content
30/1980	Non-alcoholic carbonated beverages-Determination of arsenic and lead contents

Continuation - TABLE I

Designation	Title
31/1981	Non-alcoholic carbonated beverages-Determination of sugar and sacchaine contents
32/1980	Non-alcoholic carbonated beverages-Determination of benzoic acid and its salts contents
33/1981	Non-alcoholic carbonated beverages-Determination Determination of phosphoric acid content
34/1981	Non-alcoholic carbonated beverages-Determination Detection of saponine, dolsine and mineral acids
35/1981	Non-alcoholic carbonated beverages-Methods of bacteriological examination
36/1982	Mineral aggregates and sands-Determination of clay, silt and dust
37/1982	Mineral aggregates and sands-Determination of soundness
38/1982	Mineral aggregates and sands-Determination of Chloride and Sulfate
39/1982	Mineral aggregates-Determination of crushing value, 10% fines value and abrasive value
40/1983	Calcium silicate bricks
41/1983	Methods of testing calcium silicate bricks

TABLE II

PROJECT PERSONNEL ATTENDING UNIDO SEMINARS

No.	Title (location other then CL*)	Name (nationality)
1.	Director General	Maher Al-Alawi *
2.	Laboratory Dirctor	Aida Riyami *
3.	Supervisor/Counterpart	Shirin Khorchid (Egypt)
4.	Chemist	C.V. Mariamma (India)
5.	Chemist	K.K. Thomas (India)
6.	Chemist	Babla Pradip (India)
7.	Chemical Engineer	Mohamed I. Beleiti (Eqypt)
8.	Chemist	M.R. Mostafu (Egypt)
9.	Geochemist (Petroleum Ministry)	Hamed Mohammed Al-Nuaim *
10.	Chemist	Shameem Mohd Salem *
11.	Asst. Chemist (Ministry Health)	Haseena Anwar Basha (India)
12.	Chemist (Ministry Health)	Mohamad Ibrahim Aird *
13.	Chemist (Ministry Health)	Self Al-Bamry *
14.	Microbiologist	Sadiq Taqi Abdulla *
15.	Student	MurtadhaTaki Abdullah *
16.	Zoologist	Abdullah Mohamed Hasan *
17.	Chemist	Jahla Habead Abdulla *
18.	Analyst	Ahmed Nasser *
19.	Analyst	Suleiman S. Al-Medhry *
20.	Analyst	Suleiman Salim Sharji *
21.	Analyst	Batool Moosa Jaffar *
22.	Analyst	Ahmed Zaher Salim *
23.	Student	Fatma O. Al-Harthy *
24.	UNIDO Expert	Keith E. Burke (U.S.A.)

* CL represents Central Laboratory
indicates Oman Nationals

TABLE III

NUMBER AND TYPE OF SAMPLES ANALYZED BY THE CHEMICAL LABORATORY

Year	Water	Cements, Rocks,etc.	Food Stuffs	Miscel.	Yearly Total
1976	402	338	...	...	740
1979	160	178	...	32	370
1980	124	132	...	6	262
1981	215	114	33	6	368
1982	727	498	176	30	1431
First Quarter 1983	260	163	120	26	2276*
Six Year *	2668	1912	689	178	5447

* Estimate

TABLE IV

AVERAGE DISTRIBUTION OF WORK ACCORDING TO THE TYPE OF SAMPLE FOR THE YEARS 1976 TO 1983

Type Sample	Percent
Water	49.0
Cements, etc.	35.1
Food Stuffs	12.6
Miscellaneous	3.3

TABLE V

ANALYSIS OF OMANI KING FISH LIVER AND KIDNEY FOR TRACE METALS

Methods:

DPS ..... Differential Pulse Stripping Polarography

AAS ..... Cold Vapor Atomic Absorption Spectrometry

Element, level	Method	Concentration Found	Mean ± S	%2S
Hg, PPM	AAS	0.067, 0.034, 0.047, 0.056	0.051±0.014	27
Zn, PPM	DPS	57, 52, 53, 50, 51, 57, 53	52.6±2.3	4
Cd, PPM	DPS	2.0, 1.8, 2.2, 2.4, 2.1, 3.8, 2.3	2.4±2.3	28
Pb, PPB	DPS	547, 411, 591, 490, 434, 510, 582, 384	494±78	16
Cu, PPM	DPS	4.t, 4.8, 4.2, 3.4, 4.7. 3.8, 4.6	4.3±0.5	12

TABLE VI

POLAROGRAPHIC RECOVERY OF PARTS PER MILLION QUANTITIES OF TRACE METALS IN OMANI DRINKING WATER

Zinc		Cadmium		Lead		Copper	
Added	Found	Added	Found	Added	Found	Added	Found
o.020	0.029	0.020	0.024	0.020	0.015	0.020	0.025
0.200	0.181	0.200	0.183	0.200	0.179	0.200	0.184
0.99	0.951	0.99	0.927	0.99	0.97	0.99	0.934
1.97	1.93	1.96	1.93	1.96	1.94	1.96	1.93
2.90	2.86	2.90	2.84	2.90	2.91	2.90	2.84

TABLE VII

X-RAY FLUORESCENCE ANALYSIS OF CEMENT

	% Al_2O_3		% SiO_2		% MgO	
No.	Certified	XRF ± S	Certified	XRF ± S	Certified	XRF±S
633	3.78	3.70±0.017	21.88	21.61±0.12	1.04	0.97±0.036
638	4.45	4.39±0.027	21.48	21.51±0.036	3.84	3.81±0.04
639	4.28	4.34±0.19	21.78	21.78±0.031	1.26	1.28±0.04

TABLE IIX

LECTURE PLAN FOR CENTRAL LABORATORIES OF OMAN

Beginning date of 1983 Lecture	Topics
June - July	No programme during Ramadan
23 July	An Overview of the UNIDO Programme
27 July	Principles of Instrumental Analysis
28 July	Computer Terminology
31 July	Basic Theory of X-Rays
1 August	Basic Steps in X-Ray Fluorescence Analysis
3 August	X-Ray Physics
6 August	X-Ray Sources and their Detection
10 August	Wavelenth Dispersive Spectrometer for XRF
13 August	Qualitative X-Ray Fluorescence (XRF)
17 August	XRF Errors and Methods of Sample Preparation
20 August	Trace Analysis by XRF
24 August	Standard Reference Materials for Analysis
27 August	Theory of Atomic Absorption Spectroscopy (AAS)
1 September	Preconcentration for AAS
6 September	Refractory Elements Analyzed by AAS Flames
9 September	Practical Applications of AAS
11 September	Electroanalytical Techniques
15 September	Classical Polarographic Methods
18 September	Modern Polarographic Techniques
25 September	Errors in Polarographic Analysis
2 October	Practical Application of Anode Stripping
9 October	Voltametry and Potentiometric Methods
7 November	Chemical Abstracting Literature
14 November	Quality Control in a Chemical Laboratory
21 November	Preventive Maintenance Records
28 November	Management of Analytical Data and Reporting

APPENDIX I

The following appeared in the Oman Daily Observer on page 5 on 27 November 1983. The account by P. Ravindranath is exactly as written, except that Oman Rials are converted to U.S. dollars.

KEEPING A KEEN EYE ON THE QUALITY OF GOODS

A STEADY and continuous rise in industrial output is indispensable for economic development. Every developing country in the world, fully aware of this, now gives top priority to expanding its industrial base.

The Sultanate realised this about a decade ago and went all out to encourage the country's nascent manufacturing sector.

The result has been spectacular. Industrial production has forged ahead over the last ten years and its contribution to the Gross National Product has been growing at a sustained rate. But, the authorities also knew only too well that quantity was just one part of the story. Equally, or at times more, important was the other part - quality and specifications.

"Quality is vital. This is especially so when what is produced locally has to withstand stiff competition form imports from the well established firms of the more developed countries," says the Director-General for Specifications and Measurements at the Ministry of Commerce and Industry, Maher Salim al Alawi.

Established in 1974 as a department, the Directorate-General was upgraded to its present status in 1976, and is at present one of the busiest organizations in the Sultanate trying its best to cope with the steadily rising demand for standards put on it by the ever-expanding industrial sector.

Forty one Omani standards have so far been set by the Directorate-General through its two departments in Wadi Kabir - Specifications and Quality Control, and the Government Central Laboratory.

The specifications wing of the former lays down the Omani standards of industrial products. The standards, in turn, are formulated by technical committies comprising experts from the Directorate-General and representatives from the various Ministries and manufacturers and users of the product. This is done after detailed research studies. Teams of specialists are sent to visit factories to collect all the relevant data about the product in question. Wherever the standards are to be enforced compulsorily, the Ministry of Commerce and Industry issues a Ministerial order.

The quality control section carries out the inspection part of the job which includes surprise checks at factories to make sure that the fixed standards are adhered to. Samples are collected fro comprehensive testts at the central laboratory. If the product does not conform to the standard prescribed, studies are carried out to find out the causes. The findings and recommendations are forwarded to the company concerned.

A Royal Decree issued in 1978 authorising the Commerce and Industry Ministry to issue Ministerial decisions regarding specifications and measurements has laid down the actions that can be taken against erring manufacturers. The penalty ranges from US$ 579 to US$ 2895 and imprisonment of between a week and one month. A repetition of the violation can attract a double penalty and closure of that section of the factory responsible for it. So far, however, most of the companies have been conforming to the prescribed standards and are co-operative, says Mr. A. Alawi.

Apart from locally manufactured products, the Directorate-General has also set the specification standard for imported cement.

Normally, the Director-General adds, the organization's sphere of operations is confined to the Capital Area. However, its jurisdiction covers the entire Sultanate. Checks are sometimes carried out in factories in the Interior on the basis of specific complaints against the quality of the products.

The laboratory has been designed to analyse a wide range of products. The complex at present consists of four laboratories: chemical, physical, microbiology and predious metals assay. Two more laboratories for metrology and industrial research will go on stream shortly under the second phase of expansion of the Directorate-General.

Apart from the laboratories, the project consists of training facilities for the staff, stores, a big library and a lecture hall with a capacity to seat 100 people.

The project is expected to cost US$ 2,669.190.

Work on the second phase began in September and will be ready by April 1984.

Meanwhile, a branch in Salalah is under construction and is planned to be opened next month. To begin with, it will have a laboratory for testing precious metals. There are plans to set up more laboratories later on.

The Director-General, Mr. Al Alawi adds, that he lays great emphasis on training Omani personnel. Out of the total staff strength of 56, the number of Omanis is 35 of whom 22 are scientific and technical staff.

Staff members are sent abroad for advanced training. Arrangements have also been made with international organizations for sending experts to train staff at the Directorate-Genera. At present a specialist from UNIDO is here giving training in analytical instrumentation. Two more experts from the organization are expected to arrive shortly. The training will last about six months. The Director-General also often takes part in international seminars, workshops and conferences on measurements and specification. And in view of the emphasis the Sultanate has put on industrialisation, Mr. A. Alawi feels, the Directorate-General will have an increasingly vital role to play in the future.

APPENDIX II

ANALYTICAL PROCEDURES FOR TRACE ANALYSIS

1. Determination of Microgram Amounts of Antimony, Bismuth, Lead and Tin by Non-Aqueous Atomic-absorption Spectroscopy.

Reagents

Standard stock solution, 1000 ug/ml.

Prepare a stock solution from high purity metals. The final acid concentration should be 6 M in hydrochloric acid. Make dilute solutions according to the sensitivity of the instrument. The following Table is only a guide.

Element	Wavelen-length, nm	Slit mm	Lamp mA	Working Range ug/ml	Gas Flow, L/min C2H2	Air	N20
Antimony	217.6	0.2	35	1 to 20	2	24	...
Bismuth	233.1	0.2	30	1 to 10	2	24	...
Lead	217.0	0.7	30	1 to 7	2	24	...
Tin	286.3	0.7	30	1 to 50	2	..	12

The C2H2 is at 8 psig and air at 30 psig with N20 at 30 psig.

IOPO, 5% in MIBK.

Disclve 12.5 g of tri-n-octylphosphine oxide (TOPO) in 250 ml of MIBK, (methyl iso-butylketone).

Iodine Reagent.

Prepare daily a solution containing 30 percent (w/v) postassium iodide and 10 percent (w/v) hydrochloric acid. All chemicals used were of analytical-reagent graed. Grade A calibrated glassware was used. All glassware was acid washed.

PROCEDURE:

Weigh accurately into beakers samples that contain a sufficient amount of antimony, bismuth, lead, and tin to give a satisfactory signal. Instrumental response can be varied by adjusting the sample weight and volume of extractant as shown in the follwing example for the 10-PPM level:

Sample weight, gram	1	1	5	5
Final volume, ml	10	5	5	2
Element, ug/ml	1	5	10	25

Prepare a reagent blank. Dissolve the sample and remove any oxidant used in the sample preparation. Rinse the sides of the beaker with about 5 ml of water and add 4 g of ascorbic acid to those samples that contain iron (III). The disappearance of the dark yellow color caused by iron (III) chloride is used to judge the completeness of the reduction to iron (II). Add 15 ml of the iodine reagent and transfer the sample to a 150-ml separating funnel, rinsing the beaker with water, and adjust the funal volume to about 5o ml. With a pipette, introduce 10 ml of 5% TOPO-MIBK reagent into the separating funnel, equilibrate the solution for 30 seconds, and allow the phases to separate. Drain off the lower aqueous layer and discard it. Transfer the organic phase into a 15-ml stoppered vial. Occasionally an emulsion forms, which can be broken by biefly centrifuging the organic phase. Do not use phase-separation paper to break the emulsion unless it has been found to be free from a tin compound that is soluble in MIBK.

Optimize the burner height, aspiration rate and gas flow-rates; the conditions given in the table above can be used as guide-lines. Prepare calibration solutions by diluting the standard stock solution with MIBK. Begining with the solution that has the lowest metal content aspirate each solution and record its absorbance. Aspirate the sample solutions and blank and record their absorbance. Correct the absorbance reading of the samples for any blank. Convert the absorbance of the test solution into micrograms per milliliter by using the appropriate calibration graph and calculate the levels of antimony, bismuth, lead, and tin.

2. Determination of Parts-Per Million Quantities of Tellurium by X-Ray Spectrometry.

Reagents

Standard Tellurium Solutions, 500 and 5 ug per ml.

Dissolve 0.5 gram of high purity tellurium (Spex Industry, Metuchen, NJ) 99.999% in 10 ml of aqua regia and dilute to 1 liter with water. This solution contains 500 ug of tellurium per ml.

Tin (II) Chloride, 1 gram per ml.

Weigh 250 grams of stannous chloride, $SnCl_2 \cdot 2H_2O$, into a 400-ml beaker, add 100 ml of concentrated hydrochloric acid, and place on a warm hot plate until the solution becomes clear. Stir occasionally. Transfer the solution to a 250-ml bottle and dilute to 250 ml with concentrated hydrochloric acid. Prepare this solution daily, as tin (II) is readily air-oxidized.

Use reagent grade chemicals and class A volumetric glassware for the preparation of all solutions. All glassware is acid washed.

PROCEDURE - Dissolution:

Accurately weigh the sample into a 100-ml beaker. Select a sample weight so that 5 to loo ug of tellurium will be present. Dissolve the sample in 35 ml of concentrated hydrochloric acid with dropwise addition of nitric acid. If it is necessary to add more than 2 to 3 ml of nitric acid, either take the smaple to near-dryness and remove the nitrate by repeated evaporations with hydrochloric acid or fume with 5 ml of perchloric acid. Add 5 ml of sulfuric acid when the sample is known to contain loo mg or more of molybdenum, or to volatilize selenium partially when it is present as a major constituent. Adjust the volume to about 15 ml if selenium is known to be absent and proceed with the prefiltration.

Prefiltration:

Use a microfiltration apparatus (XX-1002500, Millipore Filter Corp., Bedford, Mass, USA) fitted with glass fiber filters (934 AH, Reeve Angel, Clifton, NJ, USA) for rapid filtration of any silica, graphite, etc, which might be present.

For samples containing less than 50 mg of selenium, make the concentration of hydrochloric acid 6 to 9 N, add 8 grams of hydroxylamine hydrochloride, and boil the solution for 5 minutes to precipitate elemental selenium. Separate the selenium along with any other precipitate on an 8-micronmicropore filter. Repeat this step if the selenium level is greater than 50 mg. Rince the filtrate back into the original beaker and dilute to 40 ml. If the volume is too large at this point, transfer the sample from the filtration flask to a 150-ml beaker and reduce the volume by evaporation. Adjust the hydrochloric acid concentration to at least 3 N for th tellurium precipitation.

Tellurium Precipitation:

Add a miniumum of 10 ml of the tin (II) chloride solution to the sample. Add 10 ml more of reductant for a 2-gram sample, or a total of 30 ml for a 3-gram sample. Stir the solution and allow it to stand for 5 to 10 minutes before filtration onto a 2.5-cm, 8-micron micropore filter. Wash the precipitate with 50 ml of 3 N hydrochloric acid followed by 50 ml of water to remove any trace of the reductant, any other metal ion, or acid. Increase the air flow through the filter for about a half minute. Place the filter on a spot plate and dry for 10 minutes in an oven set at 90 C. Mount the dry filters with double back adhesive tape on squares or circles of 1/8 inch thick phenol fabric. Any material may be used which does not have detectable X-ray lines in the spectral region of interest.

Measurement:

Place the dry filter containing the precipitate of tellurium in the vacuum X-ray spectrometer equipped with a chromium target, lithium fluoride crystal, 0.01-X 4-ince collimator, gas flow proportional counter and phenol fabric masks with openeings 1-inch in diameter. Measure the tellurium L alpha radiation for 40 seconds at a two theta of 109.54 degrees at 50 mA and 65 kV. Focus the crystal with pure tellurium.

Determine the background by reference to a blank. Calculate the concentration of tellurium by comparison with standards after substracting the background correction. Count a few calibration points along with the samples.

Calibration Curve:

Place alliquots containing 0, 5, 10, 20, 50, and 100 ug of tellurium in 100-ml beakers with 8.5 ml of concentrated hydrochloric acid, dilute to 40 ml, and add 10 ml of tin (II0 solution. Filter the precipitates on 8-micron filters. Wash, dry, and mount the precipitates. Measure the tellurium L alpha radiation, subtract the background, and draw a calibration curve. The standards may be reused for subsequent analyses.

3. A Chemical Concentration X-Ray Determination of Selenium

Reagents

Standard Selenium Solution.

Dissolve 0.5000 gram of 99.999% pure selenium in 10 ml of concentrated nitric acid and dilute to 1 liter with water. Prepare a selenium solution containing 10 ug/ml by dilution of 10 ml of this stock solution to 500 ml with water.

Other reagents should be analytical grade.

Apparatus

Microfiltration apparatus:

A Millipore (Bedford, Mass) filtration assembly no XX100 2500, containing a suction flask, micro funnel, sintered glass disc and clamps. Micropore filters (GS WPO 2500) of 0.22-pore size and 25 mm idameter. Glass fiber filters (No. 934AH, Reeve Angel, Clifton, NJ).

X-Ray Spectrograph:

Diano X-ray spectrograph was used with EZ-75 platinum target tube, lithium fluoride crystal, and 0.25 X 100 mmm collimator. The tube was operated at a power of 50 mA and 50 kV. A combination scinillation (SPG-4) and flow (SPG-7) counter was used with a mixture of argon and methane (P-10) gas. The sample drawer mask and mounts for the sample filters were made of 3-mm thick phenol fabric.

PROCEDURE

- Dissolution:

Transfer an accurately weighed representative sample to a 150ml beaker and treat it with 5 ml of perchloric acid and 25 ml of nitric. (This acid mixture is designed for sample weights of 1 gram or less.)

Cover the beaker with a watch-glass and digest on a hot-plate at moderate heat until the reaction is complete. Continue heating until the nitric acid has been removed by volatilization. Cool to room temperature, then add 75 ml of 6 M hydrochloric acid. (Digest at a moderate heat to dissolve the salts.) Filter the solution through a glass fiber filter attached to the microfiltration apparatus. Wash the filter with water. Air-dry the filter for about 1 hour and then mount it on a phenol fibric disc with double-backed adhesive.

- X-ray Analysis:

Position the mounted smaples in the sample drawer of the spectrograph and measure the K alpha radiation for 10 seconds at the two theta value of 31.89 degrees. Obtain a background correction by making a blank filter disk. Calculate the concentration of selenium by using a factor obtained by analyzing a series of standards containing 5 to 100 ug of selenium.

4. Separation and X-Ray Spectrographic Determination of Microgram Quantities of Arsenic.

Apparatus

An X-ray spectrograph capable of measuring the arsenic K alpha doublet, a platinum target X-ray tube, lithium fluoride crystal, 0.01 X 4 inch collimator, gas-flow proportional and scintilation counters with power at 50 kV and 50 mA.

Reagents

Standard Arsenic Solutions, 500 and 5 ug per ml.

Dissolve 0.5000 gram of 99.999% arsenic (Spex Industries, Metuchen, NJ) in 10 ml of nitric acid, and dilute to one liter with water. This solution contains 500 ug of arsenic per ml. Also prepare the 5 ug per ml solution.

Standard Tellurium Solution, 50 ug per ml.

Dissolve 0.5 gram of high purity tellurium (Spex Industries) in 10 ml of aqua regia and dilute to 1 liter with water. Dilute 10 ml of this solution to 100-ml.

Tin (II) Chloride, 1 gram per ml.

Weigh 250 grams of stannous chloride into a 400-ml beaker, add 100 ml of concentrated hydrochloric acid, and place on a warm hot plate util the solution becomes clear. Stir occassionally. Transfer the solution to a 250-ml bottle and dilute to 250 ml with concentrated hydrochloric acid.

Copper, 4 grams per 100 ml.

Dissolve 4 grams of high purity copper in nitric acid and fume with 20 ml of perchloric acid, dilute tc 100 ml with water.

PROCEDURE

Calibration Curve:

Place aliquots containing 0, 5, 10, 20, 50, and 100 ug of arsenic into 100-ml beakers, add 2 ml of copper solution, 2 grams of sodium hypophosphite, and make the hydrochloric acid concentration 6 N. Heat the solution to near boiling, add 100 ug of tellurium and 5 ml of tin (II) chloride solution. Boil the solutions for 5 minutes. Filter the precipates on 0.22-micron filters (apparatus XX-1002500, Millpore Filter Corporation; Bedford, Mass) wash, dry, and mount. Measure the intensity of the arsenic radiation and plot counts per second versus micrograms of arsenic. Copper is added to all systems analyzed for arsenic because it is present with any arsenic or arsenic-tellurium precipitated in the presence of copper. A calibration curve prepared in the absence of copper will produce high results for the analysis of a copper-base system.

Dissolution:

Select a sample weight so that 5 to 100 ug of arsenic will be present. Accurately weigh the sample into a 150-ml beaker and add 2 ml of copper solution. Dissolve the sample with nitric acid, add hydrochloric acid if necessary, after dissolution add 5 ml of perchloric acid (10 ml for 2-gram sample) and fume. Cool and add 30 ml of water and 20 ml of hydrochloric acid.

Prefiltration:

Add 10 ml of tin (II) solution and filter off any silicon, graphite, etc. Use an 8-micron micropore filter for separation. Rinse the precipitate with 6 N hydrochloric acid. The addition of tin (II) may be eliminated if less than 20 ug of selenium and tellurium are present in the sample.

Arsenic Precipitation:

Add 2 grams of sodium hypophosphite, $NaH2P02.H20$, heat to near the boiling point, add 100 ug of tellurium and 10 ml of tin (II) solution. Boil the sample for about 5 minutes so that the precipitate coagulates. Filter the solution through an 0.22-micron micropore filter. Wash with 6 N hydrochloric acid, water, and finally a 50% ethyl alcohol solution to wash off any precipitate which might cling to the funnel. Dry, mount, and measure the intensity of the arsenic K alpha radiation using an X-ray spectrometer. Calculate the arsenic content after using the calibration curve.

5. Atomic Absorption Spectroscopy for Determination of PPM Quantities of Copper and Nickel in Organic Food Stuffs

Principle

Samples are wet ashed and after dilution are aspirated into air-accetylene flame. Radiation at 324.7 nm from a copper hollow cathode lamp and at 232.0 nm from a nickel hollow cathode lamp is passed through the flame. Attenuation is measured in a spectrophotometer calibrated with known concentrations ov copper and nickel in the presence of matrix similar to that of samples, which avoids interference from elements such as sodium and potassium. Concentration range is 5 to 100 PPM, depending upon the sensitivity of the instrument. Working range is 0.2 to 10 ug/ml. The recommended upper limit is that which gives about 0.4 absorption.

Apparatus

Atomic absorption spectrophotometer consists of light sources, atomizer, burner assembly, monochromator, and detector. Light sources are single element hollow cathode lamps with stabilized but adjustable current. Radiation is modulated with detector system tuned to the same frequency. The monochromator must resolve 324.7 and 232.0 nm lines; half-band pass of 0.5 nm is adequate. Burner head must safely burn fuel-rich flame. The detector must respond with adquate sensitivity to lamp radiation after passing through the flame and monochromator. It must be capable of responding to a 0.2% change in intensity of incident radiation. The complete apparatus must be capable of measuring the content or change of 0.05 ug of nickel or copper in a ml of aqueous solution.

Preparation of Standard Solutions:

- Copper Standard Solution

Dissolve 1.000 gram of 99.99% copper in 20 ml of nitric acid, cool, and dilute to 1 liter with water.

- Nickel Standard Solution

Dissolve 1.000 gram of 99.999% nickel in 20 ml of nitric acid, cool, and dilute to 1 liter with water.

- Matrix Standard Solutions

Prepare solutions containing 0, 0.2, 0.4, 0.8, 1.6, 2.0, 4.0, 8.0, and 10 ug/ml of nickel and copper and the major metal matrix components. For a 3 gram sample also add to contain 180 ug calcium, 100 ug magnesium, and 40 ug perchloric acid. For a 6 gram sample prepare the matrix to contain 7000 ug potassium, 70 ug sodium, 700 ug magnesium, and 130 ug calcium per ml.

Preparation of Calibration Curve:

With the copper hollow cathode tube in position, energized and stabilized, locate the wavelength setting that gives maximum response to the radiation at 324.7 nm. Wash the combustion chamber and burner head with nitric acid (1+1). Light the burner, let it reach thermal equilibrium, and zero the instrument while aspirating water. Aspirate 10 ug/ml copper standard and adjust the burner heights, air and fuel pressures and flow rates, aspiration rate of the solution and position of the capillary to obtain maximum responsed adjust slit setting and gain to obtain optimum signal-tonoise ratio. Repeat the procedure with a nickel hollow cathode, using the wavelenght at 232.0 nm. The absorbance should be about 0.32 for 10 ug/ml of nickel. Scale expansion is required to obtain appreciable reading for copper and nickel concentrations which are less than 2 ug/ml. Three-fold expansion is normally required for the determination of nickel.

Aspirate 10 ug/ml standard solution enough times to establish that the absorbance reading is not drifting. Record six readings and calculate standard deviation, S= (x-y) x 0.40, where x and y as maximum and minimum readings, respectively, and 0.40 is a factor to convert range of six values into S or sigma.

Beginning with the solution containing 0 copper, aspirate each matrix standard solution and record the absorbance. If the value for 10 ug/ml solution differs from the average of six values used to calculate S by more than 0.01 x (the average of the 6 values), repeat measurements. If these determinations indicate drift, determine the cause (e.g. deposits in the burner or clogged capillary), correct it, and repeat calibration. Repeat for nickel solutions. Plot the absorbance against the metal concentration in ug/ml.

Wet Ashing - Accurately weigh the sample into a 400-ml beaker. add 100 ml nitric acid, and swirl. Cover and let react 10 minutes; then place on the hot plate. Evaporate it to near dryness and cool. Add 50 ml of nitric acid and 10 ml of perchloric acid. Continue to evaporate until a clear solution is obtained.

Transfer to a 50-ml volumetric flask and dilute to volume with water (Insoluble potassium perchlorate, which settles to the bottom of the flask does not interfere).

Prepare a reagent blank containing the same amounts of acids taken from the same lots of acids and evaporate as indicated above.

Photometry - Aspirate sample and blank solutions and record their absorbance. Measure absorbance of the matrix standard solutions containing 10 ug/ml. If this value differs from the value of the average of six values used to calculate S by more than 2S, repeat measurements. If these values indicate drift, determine the cause, correct it, and repeat calibration and sample and blank readings.

Calculations - Correct readings of sample solutions for the blank. Convert corrected absorbance readings to ug/ml from the calibration curve. Calculate PPM Ni (or Cu) = (C x V)/W, where C= ug metal per ml from the curve, V= final volume of sample solution, normally 50 ml, and W = sample weight in grams, normally 3 or 6 grams.