No. ______

CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

75

GEOLOGICAL SURVEY OF CANADA
TOPICAL REPORT NO. 75

ROCK AND MINERAL ANALYSIS IN GREAT BRITAIN AND SCANDINAVIA

(Report on visits to organizations concerned – Autumn, 1962)

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BY
J. A. MAXWELL

OTTAWA
1963

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INTRODUCTION

In his report (GSC Topical Report No. 58) on visits to organizations in Great Britain and Northern Europe engaged in scientific work similar to that being done in the Petrological Sciences Division, Dr. S.C. Robinson makes frequent reference to the chemical and spectrographic methods and equipment used in these countries for the analysis of rocks and minerals. Many of the countries visited have long traditions in this specialized field of analysis and his comments were of particular interest to the writer, whose special field this is. Initial contacts made by Dr. Robinson were followed up by correspondence which led to an exchange of ideas and information on methods and equipment for rock and mineral analysis.

The Fifth Summer School in Analytical Chemistry, at the Manchester College of Science and Technology, 9-15 September, 1962, offered an opportunity for the writer to hear about and see at first-hand the methods, techniques and equipment that are in use in Great Britain. It was later decided to precede, and follow, this course by visits to pertinent laboratories in Scandinavia and Great Britain in order to meet those engaged in rock and mineral analysis. Shortly after the itinerary for this was arranged, an invitation was extended to the writer to attend a conference on analytical methods for meteorites to be held in London; this was accepted and the itinerary modified accordingly.

The objects of this extended trip were:

(1) To see the facilities and equipment, and to meet the personnel of organizations engaged in rock, mineral and related analysis;

(2) To learn about research studies, in progress or proposed, on the analysis of rocks and minerals, and of new equipment and techniques designed to facilitate this work;

(3) To discuss methods of operation and methods of analysis, both for routine and non-routine work.

Visits were made to a variety of institutions, including universities, geological surveys, research establishments, museums, instrument manufacturers and one mining company, in Norway, Sweden, Finland, England and Scotland. During the period 22 August to 19 October, involving 43 working days away from Ottawa, the writer visited 34 institutions and met and talked with over 90 scientists and others engaged in various aspects of rock and mineral analysis, and related work.

ITINERARY

Date	City	Institution and Address	Visited
22 Aug.-25 Aug.	Oslo, Norway	University of Oslo, Department of Chemistry Blindernveien.	Dr. F.J. Langmyhr Mr. P.R. Graff Mr. I. Dahl
		Mineralogisk-Geologisk Museum Sarsgatan 1 Oslo 45	Dr. R.H. Filby Dr. T.K. Ball Mr. S. Bergstol Dr. H. Neumann Mr. A. Brünfelt
26 Aug.-30 Aug.	Stockholm, Sweden	Swedish Cement and Concrete Research Institute Drottning Kristinasvag 26	Dr. R. Hedin
		Ingeniorsfirma Hugo Tillquist Sodra Langgatan 21 Solna	Dr. A. Danielsson[a] Mr. T. Adolphson
		Geological Survey of Sweden Stockholm 50	Dr. S. Landergren[b] Dr. F. Manheim[c] Mr. W. Muld Dr. B. Rünnholm Dr. G. Ostlund

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[a] now at Geological Survey of Sweden, Stockholm.

[b] now at Marine Laboratory, University of Miami, Miami, Florida.

[c] now at Department of Geology, Yale University, New Haven, Conn.

Date	City	Institution and Address	Visited
	Skelleftehamn	Central Laboratories Bolidens Gruvaktiebolag	Dr. A. Olafsson Mr. G. Sundkvist Mr. I. Nilsson
31 Aug.-3 Sept.	Helsinki, Finland	Geologinen Tutkimuslaitos Otaniemi	Prof. V. Marmo Dr. V. Veltheim Mr. A. Nurmi Dr. H.B. Wiik Mr. P. Ojanpera Mr. V. Hoffren Mr. A. Lofgren Mr. P.J. Vaananen Mr. Isola Dr. Hytanen
		Institute of Geology and Mineralogy University of Helsinki, Snellmaninkatu 5	Prof. K. Rankama Prof. Th.G. Sahama
4 Sept.-7 Sept.	London, England	British Museum (Natural History) Department of Mineralogy, Cromwell Road, London, S.W. 7	Conference on analytical methods for meteorites. Dr. M.H. Hey Dr. G.F. Claringbull Mr. D.I. Bothwell Mr. A.G. Hayward
8 Sept.-17 Sept.	Manchester	College of Science and Technology Whitworth St.	Summer School in Analytical Chemistry
		Department of Geology The University Manchester 13	Prof. E.A. Vincent Dr. R.A. Howie Dr. W.S. Mackenzie Dr. J. Zussman
		Department of Metallurgy The University Manchester 13	Dr. H.J. Axon
	Leeds	Department of Geology The University Leeds 2	Miss Joan M. Rooke Dr. P.G. Harris Miss J. Baldwin
18 Sept.-20 Sept.	Liverpool	Department of Oceanography The University Liverpool 3	Dr. J.P. Riley

Date	City	Institution and Address	Visited
	Stoke-on-Trent	British Ceramic Research Association Queens Road, Penkhull	Mr. H. Bennett Mrs. I. Thwaites Mr. R.P. Eardley
	St. Helens	Central Analytical Laboratories Pilkington Brothers Ltd. Eccleston Grange	Mr. F. Hartley Mr. W. Ward Mr. W.J.R. Merren Mr. J.P.L. Truesdale Mr. Blanchard Mr. Blackmore
21 Sept.-23 Sept.	Glasgow, Scotland	W.H. Herdsman Chemical and Metallurgical Laboratories 229 West Regent St., Glasgow C.2	Mr. B.G. Docherty
24 Sept.-26 Sept.	Edinburgh	Department of Geology The University West Mains Road	Dr. E.L.P. Mercy
		Department of Chemistry The University	Dr. C.C. Miller (retired)
27 Sept.-1 Oct.	Aberdeen	Department of Geology and Mineralogy Marischal College The University	Prof. T.C. Phemister Dr. W.E. Fraser Mr. G. Downie
		Department of Chemistry King's College, The University	Dr. R.A. Chalmers
		Macaulay Institute for Soil Research Craigiebuckler	Dr. A.B. Stewart Dr. R.L. Mitchell Dr. R.C. Mackenzie Dr. R.O. Scott Mr. D.J. David (D.S.I.R., Australia)
2 Oct.-4 Oct.	Oxford, England	Department of Geology and Mineralogy University Museum	Prof. L.R. Wager Dr. N.J. Snelling Dr. E. Hamilton
	Harwell	Atomic Energy Research Establishment Analytical Chemistry Branch Harwell, Didcot, Berks.	Mr. A.A. Smales Dr. G.W.C. Milner Mr. D. Mapper Dr. R.K. Webster

Date	City	Institution and Address	Visited
5 Oct.-8 Oct.	Cambridge	Department of Mineralogy and Petrology The University Downing Place	Mr. J.H. Scoon Mr. S. Reed
		Unicam Instruments Ltd., York St.	Mr. N.H.E. Ahlers Mr. D. Griffiths Mr. Harrison
9 Oct.-19 Oct.	London	Royal School of Mines, Imperial College of Science and Technology Prince Consort Rd. London, S.W. 7	Prof. D. Williams Dr. J.R. Butler Dr. J.S. Tooms Dr. I. Carmichael Mr. R. Stanton Miss A.J. McDonald Mr. R. Berlin
		Chemistry and Technical Services Division General Electric Company Ltd., Wembley, Middlesex	Mr. R.C. Chirnside Dr. H.J. Cluley
		Overseas Geological Surveys 64-78 Gray's Inn Road, London, W.C. 1	Mr. T. Deans Mr. W.H. Bennett Mr. D.R. Curry
		Geological Survey and Museum Exhibition Road London, S.W. 7	Mr. G.A. Sergeant
		Laboratory of the Government Chemist Clement's Inn Passage, Strand London, W.C. 2	Mr. A.D. Wilson
		The Analyst 14 Belgrave Square London, S.W. 1	Mr. J.B. Attrill (Editor)
		Hilger and Watts, Ltd. 98, St. Pancras Way Camden Road London, N.W. 1	Mr. Moore
	Stevenage	Department of Scientific and Industrial Research, Warren Spring Laboratory Gunnels Wood Road Stevenage, Herts.	Mr. S.H. Clarke Dr. P.G. Jeffery

The itinerary was drawn up on the basis of contacts made by Dr. Robinson and on personal knowledge of places of analytical interest. Arrangements were made well in advance by means of personal letters to the various analysts or heads of the laboratories concerned. Very useful travel information was obtained from Survey colleagues, particularly with regard to reliable and convenient hotel accommodation. Travel and hotel reservations were made by an Ottawa travel agency who were supplied by the writer with a list of preferred hotels; it is a pleasure to report that all the arrangements so made proved to be very satisfactory.

In addition to a personal 35-millimetre camera the writer took a Polaroid Land Camera (10-second development) for photographing laboratory interiors, equipment and personnel. This proved to be very successful and many useful photographs were obtained; flashbulbs were not used but it is recommended that a supply be taken to improve the poor lighting conditions that are often encountered. The advantage of the Polaroid camera is in the instant availability of the picture; it also served as a means of ready conversation in laboratories where it was unknown. Less successful was a small dictating machine (Dictet) taken for the purpose of recording impressions, laboratory descriptions and non-technical details at the end of the day's work. It did not contribute much to the record of the trip.

A set of 35-millimetre colour slides depicting the laboratories, equipment and facilities of the Survey was taken along and proved very useful, as did a book of 8" x 10" photographs of Survey activities. Because of a general lack of knowledge of our work and facilities these were viewed with much interest and some surprise. A set of colour slides of Ottawa and the surrounding area was also taken and shown on request.

Generally one day was sufficient time for each visit and on only two occasions would more time have been useful.

CONCLUSIONS

The most obvious conclusion to be reached as a result of this laboratory tour is that none of the organizations visited are engaged in rock and mineral analysis of similar scope to that of the Geological Survey of Canada. Statements made about the number of analyses requested and supplied in the GSC were received with some amazement and only at the Macaulay Institute and at Pilkington Brothers Ltd. did the writer find a similar emphasis on the production of a large number of analyses, and these two organizations are not really comparable to the GSC in the type of work being done.

The facilities of the Geological Survey of Canada for rock and mineral analysis are fully equal to those available in the institutions visited and are better than those in many of the older organizations. Differences in the equipment used are minor, which came as something of a surprise, although it is often these small differences, based upon accumulated experience, that are of most interest and value to others. There is no lack of equipment in the laboratories visited, indeed there seemed to be an overabundance of it in most of them, and the writer was impressed by the quality of most of it. The British equipment manufacturers have much to offer and more effort should be made on our part to investigate their products before buying new equipment. The most outstanding piece of equipment in this respect is the small, inexpensive Evans Electroselenium (EEL) flame photometer, a diminutive instrument of amazing simplicity and reliability.

None of the laboratories are using the classical methods of rock analysis in their true sense. All have introduced instrumental techniques of one kind or another (it must be noted that in some instances this has been followed by a return to the essentially classical approach!). The rapid methods of Shapiro and Brannock, in use in the GSC since 1958, were designed for batch analysis and are thus highly empirical in nature, and are seldom used as intended by their originators; some of the methods are used as alternatives to the older procedures when conditions warrant their use. Much modification of the Shapiro and Brannock procedures has been done and the methods of J.P. Riley are generally more favoured. These latter methods, using solvent extraction and ion exchange methods to achieve separations before colorimetric measurement, are designed for semimicro-and microanalysis and it is in this one area of rock and mineral analysis that the GSC is deficient, chiefly because of the lack of opportunity, resulting from heavy work load and shortage of personnel, to set up the necessary equipment and methods.

No use is being made of X-ray fluorescence spectroscopy for rock analysis as it is now being used at the Survey, nor was any such use foreseen. A number of organizations are using this method, or plan to use it, but only for selected elements and usually without any preliminary treatment (i.e. fusion, pelletizing) of the sample. Wide use is being made of emission spectrographic methods, and several laboratories are exceedingly well-equipped for the purpose, but no single laboratory attempted to cover the same wide range of sample type and element concentration that is being covered in our Survey laboratory.

The only research that, to the writer's knowledge, is being done into really new methods of analysis was that of Langmyhr at the University of Oslo. He is developing a scheme of silicate analysis that will be both rapid and highly accurate; the scheme is based on solution of the sample in hydrofluoric acid and the determination of various constituents, including SiO_2, on weight aliquots of this solution. Riley (Liverpool) is now more concerned with methods for the analysis of sea-water, although the ion-exchange separations that he is developing have application to rock and mineral analysis as well.

Much was learned by the writer as a result of this trip, including the discovery that the Survey rock and mineral analysis laboratories are in the forefront of advances being made in this field of analysis, and many interesting and useful contacts were made. As a result of these visits the writer is now better able to evaluate the significance of publications emanating from these laboratories, and to maintain and develop these contacts on a much more personal basis than was formerly possible.

GENERAL FACILITIES AND ORGANIZATION

General

The facilities for rock and mineral analysis varied widely, being for the most part directly related to the age of the building in which they were housed. The newer buildings tended to favour the module system, with movable inner walls, but this is the only general observation that can be made. Nowhere did the writer encounter facilities and equipment for rock and mineral analysis superior to those of the Geological Survey of Canada.

The following section includes a brief description and explanation of the places visited, presented in the order given in the Itinerary, and with comment upon laboratory lay-out and organization where such is warranted.

University of Oslo

Dr. F.J. Langmyhr has laboratories in the Chemical Institute (A) at Blindernveien, a suburb of Oslo. He at one time used to train geology students in methods of chemical analysis but this is now done in the Geological Institute under Dr. Mladek. Dr. Langmyhr is now working on a new scheme of silicate analysis which will be rapid but highly accurate.

Mineralogisk-Geologisk Museum, Oslo

The surroundings and facilities are reminescent of those of our National Museum, and include laboratories for rock analysis (Mr. Bruun, Dr. Ball), radiochemistry (Dr. Filby), X-ray diffraction and X-ray Fluorescence (Dr. Newmann, Mr. Bergstol), and mineral separation. Mr. Bruun uses the classical methods of rock and mineral analysis; Dr. Ball is using the rapid methods, and hopes eventually to use neutron activation and isotope dilution methods for the major elements.

Swedish Cement and Concrete Research Institute, Stockholm

Dr. R. Hedin developed a colorimetric scheme of silicate analysis which is widely quoted, but he is no longer engaged in analytical work. The Institute is supported by the cement industry and all results are available to anyone interested.

Geological Survey of Sweden, Stockholm

The Geochemical Institute is housed in a three-story building adjacent to the main building. Chemical laboratories (Dr. Runnholm) are on the top floor, with the geochemical laboratories (Dr. Manheim) on the second floor. Dr. Landergren, in charge of the laboratories, has now retired and has been succeeded by Dr. A. Danielsson. The chemical work is done by both classical and rapid methods and it is hoped eventually to introduce X-ray Fluorescence methods as well. Five spectrographs are used to handle the geochemical work, which includes rock analysis, geochemical prospecting and the analyses of sulphide ores.

Bolidens Gruvoktiebolag, Skelleftehamn

The Bolidens Mining Company Ltd. is a leader in the Swedish mining, metal and chemical industries. It operates several mines, and the installations at Rönnskär include facilities for copper processing, lead smelting, and the processing of by-products. Its most important products are Cu, Pb, Au, Ag, Se, As, iron pyrites concentrates, zinc concentrates, lead oxides and sulphuric acid.

The facilities of the Central Analytical Laboratories (Dr. Olafsson) at the Rönnskärverken are housed in a rectangular three-story building in forested surroundings on the shore of the Baltic Sea and are most impressive. A central lobby divides each floor into two wings, each wing being traversed by a central corridor. Mineral dressing and physical methods laboratories are on the first floor, with the library, administrative and related offices on the second, and the chemical laboratories on the top floor. Routine chemical analysis laboratories are in one wing, the developmental and special analysis laboratories in the other. Each routine laboratory has its own function, e.g., the determination of S, of As, Sb and Bi, of Pb, and Zn, and fire assaying for Au and Ag. Maintenance, storage and sample preparation rooms are in the basement. The laboratories are all small in size, but clean and well-lighted. Only the Quantometer Laboratory is air-conditioned. These laboratories do all of the geochemical and smelter samples, and over 300,000 determinations were made in 1961. Mill analyses are done in smaller laboratories located at the individual mines.

The Central Analytical Laboratories are divided into the Analytical Department, the Chemical Processing and Metallurgical Development Department, and the Special Sampling Department. The Analytical Department is the largest of these, with a total staff of 76, of which 65 are laboratory technicians. The Chief of the Department is Dr. A. Olafsson, and the Department is divided into sections for routine chemical analysis (Nilsson), and special chemical analysis and physical methods (Sundkvist).

The Geological Survey of Finland, Otaniemi

The building housing the Finnish Geological Survey is located in a heavily forested area at Otaniemi, about 10 miles from Helsinki, and bears a distinct resemblance to the GSC building. The building is about five years old and has six floors and a basement; it is attractively designed, with a lobby on each floor complete with flowers, chairs and a coffee table. Sample preparation and storage rooms are in the basement, with a museum and offices on the first floor; administrative offices and library, geophysical, petrographic and cartography offices and laboratories, and ore prospecting laboratories occupy the second, third and fourth floors respectively. Laboratories and offices for surficial deposits studies are on the fifth floor, with the chemical and spectrographic, including X-ray Fluorescence laboratories, on the top floor. These laboratories are small, but well-lighted and in excellent condition. Two laboratories, separated by a smaller preparation room, are used for routine ore and silicate analyses; a general balance room and a separate instrument room are used by the whole staff. Each two chemists engaged in development work share an office and a half-module laboratory.

Professor E. Savolainen is Chief of the Chemistry Department, which has a staff of 6 chemists and 4 technicians. The chemists are divided as follows: silicate analysis (Wiik, Ojanpera); ore and silicate analysis (Heikinen), ore analysis (Vaananen), optical spectrography (Lőfgren), X-ray fluorescence (Hoffren). The laboratories do work for the approximately 30 Survey geologists and for the public (very heavy in summer period).

The C^{14} laboratory was formerly part of Chemistry but has now been moved to Surficial Deposits. Geological age measurement will start soon under Dr. Kouvo; Mr. Sakko, a physicist, will study mass spectrometry at the Geophysical Laboratory in Washington, D.C.

Dr. Veltheim, Chief of the Prospecting Department, described the way in which the public is encouraged to aid prospecting by submitting samples for study. Those samples that are of interest are plotted on a map. The man who receives these samples puts the details on a form which is then turned over, with the sample, to the appropriate geologist. The latter decides if the sample warrants further investigation, and if so, he puts the added information on the form. These forms are filed and the data can be consulted when other samples are received from the same area.

Senior officers of the Survey have a light code that is used to locate them when they are not in their office. A master control flashes the appropriate code to wall fixtures containing six numbered bulbs which are located in the corridors and offices; the individual being sought then telephones the central switchboard.

University of Helsinki, Finland

The Institute of Geology and Mineralogy is housed in an ornate building which has the distinction of having been the first apartment building in Helsinki. The building is to be taken over by the Foreign Office in 3-4 years and the Institute will move to newer quarters farther up Snellmaninkatu.

Professors K. Rankama and Th.G. Sahama are on the staff of the Institute; Dr. Rankama is chiefly concerned with writing, but Dr. Sahama is busy with a study of Belgian Congo rocks and minerals.

There is one small, ill-equipped laboratory for rock analysis but most of the minerals are sent to the Geological Survey of Finland for analysis by Dr. Wiik.

British Museum (Natural History)

The Department of Mineralogy has laboratories in the basement of the Cromwell Road building which are used for rock and mineral analysis by Mr. Bothwell, occasionally by Dr. Moss and Dr. Hey. The laboratories are old but have been remodelled, and are spacious and in good condition. There is a separate instrument room and balance room. The analytical work, using both classical and rapid methods, is done for Museum officers and occasionally for outside people as well.

University of Manchester

The Department of Geology is located in the Williamson Building. The main chemical laboratory is used for both classical and rapid methods of analysis, under the direction of Dr. R.A. Howie. An adjoining room is used as an instrument room and there is a smaller balance room and storeroom. There is also a spectrographic laboratory, under Dr. Nicholls who was absent at the time of this visit. The laboratories are used for the teaching of methods of rock analysis, and by graduate students who make their own analyses. The new Head of the Department, Professor E.A. Vincent, plans to introduce neutron activation methods as soon as possible.

The Department of Metallurgy occupies the former Dental College; the building is old but in good shape and there is an abundance of equipment. Dr. H.J. Axon is making a study of the metallurgy of meteorites, attempting to reproduce with Armco iron the conditions that produced the metal phases in iron meteorites. He is to get a Metropolitan-Vickers (A.E.I.) electron microprobe to aid in these studies. Dr. Axon was much interested in the Abee meteorite that was studied by the GSC.

University of Leeds

The Department of Geology, which includes the Institute of African Geology, is housed in portions of three buildings. The Institute is located in an old vicarage, lecturing is done in a second building and geochemical laboratories are situated in a third. A new building, to have about 75000 sq. ft. of space for the Department, is anticipated by 1964. Professor W.Q. Kennedy, Head of the Department, is also Director of the Institute. The Institute received a 7-year grant, which was renewed in 1962, of about $300,000 from the Anglo-American Mining Company in Africa to make studies of African geology. Ph.D. candidates do their field work in Africa and laboratory work at Leeds.

Dr. O. von Knorring formerly lectured in geochemistry and directed the work of the chemical laboratory; he resigned and was succeeded by Dr. P.G. Harris but has since returned and there are now two lecturers in geochemistry. Dr. Harris has set up a chemical laboratory which will be used for teaching methods of rock analysis, and by graduate students who need rock analyses. The chemical laboratory under Dr. von Knorring provides a service for the Institute staff; it consists of a single laboratory with an adjoining balance and instrument room, located on the second floor of the vicarage. Modified classical methods are used (Mrs. M. Kerr, Miss J.M. Baldwin).

The Institute has a very active program of spectrographic analysis in operation under Miss Joan M. Rooke. Miss Rooke is a Ph.D. candidate as well and her thesis is a comparison of the geochemistry of older (orogenic) and younger (non-orogenic) African granites; she is attempting to delineate geochemical provinces and uses a computer to calculate the significance of her data.

The spectrographic laboratory (not air-conditioned) is in the basement (moved there one day before the ceiling of the second-floor former laboratory fell!), with sample preparation and plate-reading being done on the second floor.

There is also a very well-equipped machine shop in the Institute.

University of Liverpool

Dr. J.P. Riley is a lecturer in geology and geochemistry in the Department of Oceanography of the University (total enrollment about 4000). The University is in the process of constructing a number of new, very modern buildings in a large area in the heart of Liverpool, razing many old houses and factories for this purpose. Dr. Riley is also a leader in research on rapid methods of analysis and on sea-water studies, particularly methods for the analysis of sea-water (to extend the work of Dittmar).

The Department, which is relatively small (Professor Bowden, 5 lecturers), shares a new building with the Department of Mathematics (50 lecturers!). Dr. Riley has 5 graduate students.

The chemical and spectrographic laboratories were designed by Dr. Riley and are very well laid out. There are 2 chemical laboratories, 1 spectrographic and radiochemistry laboratory, and a balance and instrument room.

British Ceramic Research Association Laboratories

The Analysis Department (Mr. H. Bennett, Head) occupies most of the third floor of a 3-4 story building which, together with two converted old homes on either side, comprise the BCRA Laboratories. A new building is being planned. The chemical and spectrographic laboratories originally supplied analyses only to other sections of the BCRA laboratories but in 1960 they were reorganized to provide service work for member organizations. The staff swelled from 5 to 13 and is expected to reach 50 in the new building. The total work done for outside people was approximately 300 analyses of all types in 1960, but was 1500 in 1961. The increase in volume is due to the introduction of rapid methods of analysis but instead of reducing the total work load, as was hoped, the considerable jump in the number of samples submitted has increased the load to the point where research and development work is slowed.

There is a large laboratory, approximately 20' x 40', with a smaller one (15' x 15') used as an instrument room, and a separate balance room. The laboratories are old in style but are well organized and kept in excellent condition. The spectrographic laboratory was formerly adjacent to the chemical laboratories but has been moved to air-conditioned quarters in the basement and the former area is to become a laboratory for research on methods of chemical analysis.

The staff is made up of two professional chemists with the balance being high school graduates. A requirement is that new staff members take Extension Courses at the nearby Technical College; the Analysis Department staff take chemistry and physics, leading to a certificate in 6 years and Associateship in the Royal Institute of Chemistry in 7 or 8 years. One day off per week is granted to attend these courses for the first 6 years, after which further time must be made up.

Pilkington Brothers Limited, St. Helens and Lathom

All of the analytical work required by the seventeen companies of Pilkington Brothers throughout the world is done in the Central Analytical Laboratories at St. Helens, as well as the analyses made for the Central Research Laboratories at Lathom. This amounts to about 30,000 samples per year, or 70-90 per day, with about 300 samples being in progress at one time. The samples are mainly glasses, but include clays, sand, magnesite, limestone, silica bricks and refractories, and few 'complete' analyses are done. Priority is given to analyses for production.

The analytical laboratories are housed in the old Pilkington home, Eccleston Grange, which has been converted into an L-shaped, 3-story laboratory building. The stables are used for special purposes, such as the manufacture of hydrofluoric acid which they use in large quantity.

The chemical laboratories are old in style and of an industrial nature, and reflect the disadvantages imposed by the original intent of the building. The spectrographic laboratories (Mr. F. Ward) are an outstanding example of organization and are very impressive. He has 5 spectrographs including a direct-reading instrument, and a staff of 27.

The Central Research Laboratories at Lathom are new and are the most modern, the most attractively and the most expensively designed buildings of any seen on this trip. They are set in a 100-acre area formerly belonging to the Stanley family; the buildings occupy 30-40 acres and the rest is let out as farms. The cost of the buildings was just over three million dollars. They are designed to accommodate a staff of 500, of which 150 will be scientists, the rest supporting staff. The lecture theatre was particularly outstanding and contained every conceivable piece of equipment, including a blackboard which is raised or lowered to desired height behind the rostrum, at the touch of a button on the speaker's desk. There is a large computer and statistics section, and laboratories for X-ray fluorescence, electron microprobe studies, infrared studies, mass spectrometry and differential thermal analysis, to mention only a few. The Director of Research is Mr. W.J.R. Merren.

The Head Analyst is Mr. F. Hartley and there have been Hartleys with the firm since shortly after it was founded (1826). Pilkington Brothers Ltd. is strictly a family affair and one is immediately aware of the friendly atmosphere that pervades the analytical laboratories. The staff numbers 120, chiefly analysts who have risen through the ranks and have taken extramural studies. There are a number who are F.R.I.C., and some 43 with the High National Certificate. Staff members are encouraged to take extramural study and some are financed to four years of full-time university study.

Mr. Hartley (with Messrs. Ward, Blanchard, Blackmore and Truesdale) has evolved a philosophy for the functioning of a chemical laboratory that seeks to retain the best of the older methods and at the same time to make use of new methods and techniques. He insists that one method should not be replaced by another until the other has been shown over a year to be as accurate and reliable, and to offer a definite advantage over the old one. He feels that the switch to new methods may actually result in a loss of knowledge which may prove embarrassing if an instrument fails to function properly. To circumvent this he has planned a course in the older methods for his senior analysts. In general the laboratories are seeking new rapid methods but methods which are also of high accuracy and precision.

W.H. Herdsman Laboratories, Glasgow

This firm, founded in 1908 to provide a commercial chemical and metallurgical analysis service, is now at West Regent St., after some 30 years at the Bath St. quarters. The work is directed by Mr. B.G. Docherty, who joined the firm in 1946. Mr. Docherty does silicate analysis and 2 assistants handle the metallurgical analysis. The charge for an analysis of 14 constituents is about $60.

The laboratories are on the second floor of an old reconditioned building and consist of a general laboratory, a laboratory for rock analysis and a balance room.

University of Edinburgh

The Department of Geology (Professor Stewart) is housed in the Grant Institute of Geology on West Mains Road. The building is relatively new (1931) and attractively landscaped.

The chemical and spectrographic laboratories are under the direction of Dr. E.L.P. Mercy, who has lectured in geochemistry since 1958, and are used to provide analyses for himself and other staff members, as well as for teaching purposes. Dr. Mercy has been building up geochemical studies since his arrival and he hopes in time that it will become a Sub-Department. Courses in geochemistry are given in the third and fourth years, and one on applied geochemistry may soon be offered in the second year. He now has his first Ph.D. candidate in geochemistry.

The chemical laboratory is modern and well-designed; an instrument room and a balance room, both air-conditioned, open off the main chemical laboratory. The spectrographic laboratory contains equipment for X-ray, X-ray fluorescence and optical emission spectroscopy, is air-conditioned, and has just started operation. Dr. Mercy has a full-time chemist (Mr. Saunders) and an assistant for the laboratories. Dr. Mercy uses both classical and rapid methods, the latter of the Riley type having a high degree of accuracy (mainly instrumental).

While at the Department the writer attended a meeting (extraordinary) of the Mineralogical Society (the first to be held in Edinburgh) with Professor L.R. Wager in the chair.

University of Aberdeen

The University has two colleges. The Department of Geology is in Marischal College, the Department of Chemistry some distance away at King's College.

The visit to the Department of Geology (Professor T.C. Phemister) was primarily to discuss a spectrographic method for the analysis of feldspars with Dr. W.E. Fraser.

Dr. R.A. Chalmers, Department of Chemistry, has done much work on the microanalysis of silicates, chiefly in conjunction with Dr. Christina C. Miller, formerly with the Department of Chemistry at Edinburgh, and now retired. The writer had the pleasure of a meeting with Dr. Miller while in Edinburgh. Dr. Chalmers is concerned now with fundamental studies in inorganic chemistry and has discontinued his silicate work.

Macaulay Institute for Soil Research, Aberdeen

The new laboratory building of the Institute (Dr. A.B. Stewart, Director) at Craigiebuckler, although occupied in 1961, was formally opened on 21 September 1962, by Mr. D.L. Macaulay of Montreal, son of the founder. It has a total floor area of 60,000 sq. ft., and consists of two floors at the front and three at the back. It is a very modern, well-designed building and gives adequate space for the soil studies for which the Institute is justly famous. The Institute is maintained financially by the Department of Agriculture for Scotland, and also receives support from interested associations. The staff number 180, of whom 80 are Scientific and Experimental Officers.

The Department of Spectrochemistry, which occupies about half of the top floor, is headed by Dr. R.L. Mitchell (who is also Deputy-Director) and has a staff of 23, with additional postgraduate visiting scientists. The chemical laboratories, of which there are 3, are devoted to preparative work on soils, rocks and similar materials received from various Agricultural Colleges who are engaged in soil surveys, prior to their analysis by spectrochemical methods. The laboratories are bright and well-equipped, with many built-in features, and are separated by smaller service and general preparation rooms. There are two balance rooms, and 4 additional rooms used for such work as sample mixing, electrode loading, and calculations; two rooms are used for flame photometry and atomic absorption spectroscopy. The spectrographic laboratories occupy 8 rooms, all of which are air-conditioned and 3 of which have the humidity controlled to less than 40%. A feature of the laboratories is that the major DC power supply (rectifiers and resistances) is centralized in a separate small room which minimizes the effect of the heat dissipation from the resistance on the temperature control. Fine adjustments are made at each spectrograph.

Approximately 20,000 samples per year are handled by the staff, including about 150 samples (mainly soil extracts) for other Departments of the Institute. Research studies and method development are carried on by Dr. R.O. Scott. Separate areas for soil sampling and plant sampling are in the basement.

Besides being the only institution visited where routine use is made of atomic absorption spectroscopy (Mr. D.J. David, from Australia and one of the leading proponents of this technique, was a visiting scientist at the time of my visit), it is also the only place visited where infrared spectroscopy is being used for mineralogical studies, to supplement X-ray, TGA and DTA data. Dr. V.C. Farmer is using this technique for qualitative and semiquantitative mineralogical analysis of clay, usually when other techniques have failed.

The Institute has a central storeroom, presided over by a full-time storekeeper. The Department of Spectrochemistry has an auxiliary storeroom for common equipment, apparatus and supplies; an Experimental Officer is in charge of its maintenance on a part-time basis.

There is a small laboratory for rock analysis in the Department of Pedology (Dr. R.C. Mackenzie, Head) in which a mixture of classical and rapid methods are used, with the emphasis on semimicro methods.

University of Oxford

The building housing the Department of Geology and Mineralogy (Professor L.R. Wager) is being enlarged and the present laboratories are crowded and temporary in nature.

A small chemical laboratory is used by students who need analyses; the methods used are those of E.A. Vincent and J.P. Riley. The spectrographic laboratory does trace element analysis only, and uses the methods of L.H. Ahrens. There is also equipment for X-ray Fluorescence analysis.

The chief interest at the Department was the work of the Overseas Geological Surveys Dating Group, particularly that of Drs. N.J. Snelling and E. Hamilton. Dr. Hamilton's laboratory for the extraction of Pb from rocks and minerals and for Rb-Sr separations, is being remodelled and he had many ideas to offer about the way in which contamination may be kept at a minimum.

Atomic Energy Research Establishment, Harwell

The purpose of the visit was to see the laboratories of the Analytical Chemistry Branch, in particular those devoted to neutron activation and isotopic dilution methods (especially as applied to meteorite analysis), and to actinide analysis. The structure of the Division of Chemistry and the Analytical Chemistry Branch is as follows:

Division of Chemistry

Anal. Chem.
Process Chem.
Radiation Chem.
Basic Chem.

Analytical Chemistry Branch (150)

Anal. Chem. (20)
Actinide Anal. (20)
Service Anal. (75-100)
Mass Spectrometry (10-15)

Mr. A.A. Smales is a Deputy Chief Scientist and Head of the Anal. Chem. Branch. He also is in charge of the Anal. Chem. Group (20) which carries out a variety of research studies and includes method development (Dr. Loveridge), gas chromatography and electrochemistry (Dr. Wilson), solid source mass spectrometry, isotope dilution analysis, nuclear and electron spin resonance studies (Dr. Webster) and neutron activation analysis (Mr. Mapper). There are several laboratories devoted to this work, all of them very well equipped.

Dr. G.W.C. Milner, an authority on polarographic analysis, is now head of the Actinide Analysis Group. He is concerned chiefly with plutonium chemistry in material received from other Groups, particularly those working on metallurgical problems. His work is divided between non-irradiated material which can be handled in glove-boxes and with light shielding, to irradiated material which require extreme precautions in handling. His staff is made up of 3 Scientific Officers, 12 Experimental Officers and 5 Scientific Assistants.

University of Cambridge

The chemical laboratories of the Department of Mineralogy and Petrology, at Downing Place, are on the fourth floor of a new wing, and are spacious, bright and about 2 years old. One large laboratory, with adjoining balance room, is used by students. Mr. John H. Scoon has a separate laboratory and a combination office, balance and instrument room. Mr. Scoon teaches rock analysis and does analyses for staff members. He prefers classical methods but makes some limited use of rapid methods.

There is a separate spectrographic laboratory (Dr. Nockolds), and one for X-ray Diffraction and Fluorescence (Dr. Bown). There is also an electron microprobe that is being used to study iron meteorites; a new one is in process of construction.

Unicam Instruments, Ltd., Cambridge

This firm, a member of the Pye Group, manufactures a wide range of spectrophotometers and crystallographic equipment which is widely used in the laboratories visited. Particular interest was shown in the two infrared spectrophotometers made by them. The writer was able to see all their line of instruments in process of manufacture and under demonstration.

Royal School of Mines, London

The Department of Geochemistry (Professor J.W. Webb), on the fourth floor of the Royal School of Mines building (part of Imperial College of Science and Technology), has most of the laboratories of interest to the writer. There are two sections within the Department: 1. Pure Geochemistry (Dr. Butler, Dr. Carmichael), with laboratories for neutron activation (Mr. Thompson), optical emission spectrography (Mr. Berlin), X-ray fluorescence, and chemical analysis; 2. Applied Geochemistry (Dr. Webb, Mr. Stanton), with laboratories for chemical analysis (Miss McDonald) and optical emission spectrography (Miss Lewis). There is one large chemical laboratory which is shared, and some sharing of the other facilities is done also. The laboratories are used for training students and also for doing the analytical work for the staff projects, particularly those of Dr. Webb. Dr. Butler gives a course in optical spectrography to graduate students, Dr. Carmichael a 3-week course on rock analysis, and Mr. Stanton one on methods of applied geochemistry. Students work chiefly on their own samples.

There is also an electron microprobe that is used by metallurgists and physicists as well as mineralogists.

General Electric Company, Ltd., London

The Hirst Research Centre, at Wembley, was formed a few years ago to replace a number of separate research groups which were formed as the need for them developed. The Analytical and Technical Services Division, headed by Mr. R.C. Chirnside, one of the leading analytical chemists in Great Britain, serves the Centre staff, serves outside affiliated organizations that do not have facilities for research, and does special development and investigative work. There is no clearly defined analytical organization but rather a loose complex of various disciplines, the outline of which varies according to current needs. Mr. Chirnside has a definite policy of centralization of analytical services.

There are many laboratories, all very well equipped, spacious and with an industrial air about them, including those for chemical analysis, optical emission spectrography, infrared spectroscopy (organic and inorganic), X-ray diffraction, flame photometry, radiochemistry, atomic absorption spectroscopy, polarography, gas chromatography, electron-microscopy and polarimetry.

Overseas Geological Surveys, London

This organization is housed in a new building and the chemical laboratories, belonging to the Mineral Resources Division and located on the second floor, are among the finest laboratories seen by the writer. The laboratories (Mr. H. Bennett, Chief Chemist) do work for overseas geological surveys and for other organizations in London, including the Geological Survey of Great Britain.

Again there is much similarity between these laboratories and those of the GSC. The module is 20' x 20', with single and double module laboratories. Peninsular laboratory benches are used in the latter but the smaller laboratories usually have the centre left open; island benches, carrying electricity only, can easily be installed. They are kept in excellent condition, are bright, with a light vinyl tile floor. Wall cupboards were used but it was found too difficult to reach them across the benches. There is a separate laboratory for optical emission spectrography but X-ray Diffraction and Fluorescence is done in the mineralogy laboratories (3rd floor) under Mr. T. Deans. The balance room is also used as an auxiliary library, containing bound volumes of analytical chemistry journals.

The sample preparation rooms are in the basement and are very well organized and kept in very clean condition. There is also an adjoining room for assaying, and another for the preparation and testing of clay and concrete.

The samples received are varied and the work done ranges from complete chemical analysis (by classical and rapid methods) to geochemical prospecting work; it also includes assaying and the testing of clays for potential uses. There are 19 on the staff of the chemical and spectrographic laboratories, consisting of 1 Senior Principal Scientific Officer (Mr. Bennett), 4 Scientific Officers, 1 Chief Experimental Officer, 1 Senior Experimental Officer, 3 Experimental Officers and 9 Assistant Experimental Officers, Scientific Assistants, and laboratory attendants.

Mr. Bennett and Mr. Deans collaborate closely on the work done on samples. Mr. Deans examines the sample and advises on the method of preparation; a thin section is made of each sample which often provides additional information for the submitter. Unnecessary or wasted work is thus avoided by insuring that the sample is suitable for analysis.

Geological Survey and Museum, London

The chemical laboratories are small and located in old-fashioned quarters on the 3rd floor of the Geological Museum. There is one large laboratory, 3 smaller ones (including sample preparation room), and a balance room, and a separate spectrographic laboratory. Mr. A.G. Sergeant is Chief Chemist and there is a staff of 6.

Laboratory of the Government Chemist, London

A visit was paid to Mr. A.D. Wilson, formerly with the Geological Survey of Great Britain but now seconded to the staff of the Government Chemist. Mr. Wilson has done much work on methods of silicate analysis. He is occupying very unsatisfactory cramped quarters for the time being but is scheduled to move to new laboratories in the near future.

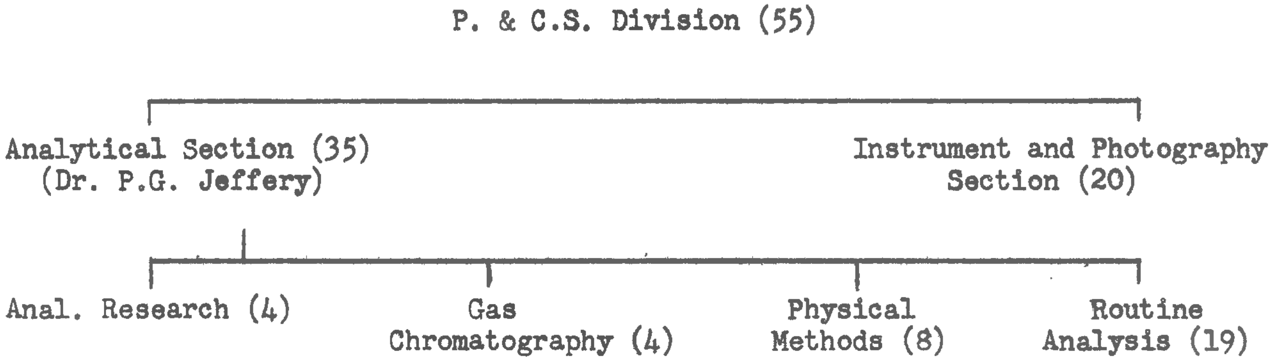
Hilger and Watts Ltd., London

A brief visit was made to these manufacturers of spectrographic and spectrophotometric equipment, particularly to see their latest equipment for atomic absorption spectroscopy.

Warren Spring Laboratory, D.S.I.R., Stevenage

This is the newest of the D.S.I.R. laboratories (Director, Mr. S.H. Clarke) and is located at the new satellite town of Stevenage, about one hour by train from London. The five Divisions of the Laboratory are Chemical Engineering and Process Development, Mineral Processing, Air Pollution, Human Sciences and Physical and Chemical Services. The terms of reference are wide and the Laboratory "is free to carry out any type of investigation which appears to be in the national interest or to the benefit of industry, provided only that the work could not be carried out more appropriately elsewhere". The writer was concerned chiefly with the work of the Physical and Chemical Services

Division which has the following organization:



The staff of the Analytical Section includes 8 Experimental Officers, 12 Assistant Experimental Officers and 15 Scientific Assistants. The Analytical Research Group provides the analytical methods needed by the others, and also does special analytical work for other Divisions of the Laboratory. Dr. Jeffery said that efforts have been made to break up the P. & C.S. Division and to distribute it among the other Divisions. The function of the Instrument and Photography Section is to advise on the purchase of new instruments, to check out such instruments on arrival and, if necessary, to develop and build special instruments. The staff of this Section includes a glass-blower.

The Physical and Chemical Services laboratories are on the third (top) floor of the long east-west main building. The building is divided by a corridor situated in the south half of the building which makes for smaller modules (12' x 12') on the south side than on the north side; the smaller modules get too warm in summer and the larger ones too cold in winter as a result of this imbalance. The arrangement of the laboratories, which are very modern, clean and bright, is very similar to that in the GSC; the main chemical laboratories form a continuous row down one side, broken only by the glass-panelled laboratory used for the beryllium analysis. Other rooms are used for optical emission spectrography, X-ray fluorescence and infrared spectroscopy (organic only). Equipment for atomic absorption spectroscopy is on order.

SAMPLE PREPARATION

General

Various methods of sample preparation, ranging from simple to elaborate, are in use in the laboratories visited. Preparation by hand to avoid loss of sample by dusting is favoured in some; the use of an automatic mortar grinder is also considered (Pilkington Brothers Ltd., Mercy) to take more time and to result in undue grinding and contamination of the sample. Hand preparation also favours the use of individual mortars and thus localizes the possibility of sample contamination. The type of preparation is governed usually by the size and quantity of the sample, and by the type of analysis to be done, e.g. whether or not trace elements are to be determined. The availability of crushing and grinding equipment plays a minor role.

Coarse Crushing

Initial breaking of the specimens is done by rock trimmers in some laboratories (Manchester, British Survey, Overseas Surveys, Mercy), by steel plate and muller, or jaw and roller crusher in others. At the Macaulay Institute, rocks are wrapped in filter paper and broken with a hammer on a granite slab; more often they are heated to a high temperature in a furnace and quenched in distilled water, thus rendering them more amenable to crushing in an agate mortar. This latter technique is not one that can be recommended. Mercy breaks samples into half-inch cubes in a rock trimmer and rejects the powder formed; the material is then shaken in a bag and a 250-gram sample is taken.

Splitting of the sample is done before further crushing at the Geological Survey of Great Britain, using automatic splitters (cone-shaped, rotary-type manufactured by Pascall Engineering Co., Ltd., and a home-made vibratory rotating splitter). A Jones splitter is also used for this purpose in some laboratories.

Grinding

Further crushing is done by hand with steel plate and pestle faced with tungsten carbide (Mercy, Royal School of Mines), or by plate, disc and cone grinders. At Manchester the knife-edges of the rock trimmer have been replaced by flat surfaces and the sample is crushed rather than broken, and then ground in a special roller-crusher designed in the Department of Geology, a description of which is given in Methods of Geochemistry (Smales and Wager, Interscience Publishers, 1960). A left-hand set of rollers reduces the coarse material to 80-mesh and this sand is then crushed to less than 200-mesh in the right-hand set of rollers. The Schwingmühle (Bleuler Mill, Shatterbox, Swingmill), with a tungsten carbide head, is used at Bolidens and at the Finnish Geological Survey (for geochemical samples). Final grinding to between 100 and 200 mesh is done largely in agate, with some use being made of the Schwingmühle, Mixer Mill and disc-grinder. An attractive automatic grinder, utilizing agate or mullite mortars, is the Retschmühle (Germany), available from Shandon Scientific Co., London. It features a chromium-plated cover with a plastic liner which fits over the mouth of the mortar; a weighted (large ball) pestle passes through the cover and the sample is returned to the centre of the mortar by a hard rubber scraper. A 20-year old Morrice automatic agate mortar and pestle, similar to the one in the GSC, is used at the Macaulay Institute; it is fitted with a variable speed motor and is operated beneath a bell-jar.

The use of brass sieves is generally avoided in favour of silk bolting cloth stretched over plastic rings, and frequent sieving of the sample is done. The Geological Survey of Great Britain uses a home-made automatic siever, 6" in diameter, which is vibrated rapidly and erratically. At Manchester those samples destined for spectrographic analysis are prepared in a roller-crusher similar to that described above but containing a built-in siever to effect continuous removal of the fines.

Sampling

At the Overseas Geological Surveys the crushing and grinding machines are fitted with dust extractors. Mixing and sampling of the ground material are done on a table equipped with a canopy and a vent for removal of dust. At the Macaulay Institute soils are sampled on a wall-table fitted with an exhaust vent at the rear; a current of air is forced through a narrow, angled slit at the front of the table and thus creates a sloping current of air moving from the front to the rear. Dust from the sample is carried away from the operator, but the sample remains below the moving current of air.

Over 30,000 samples per year are handled by the Central Analytical Laboratories of Pilkington Brothers Ltd. All sampling is the responsibility of the individual works chemist; sampling is done by trained men who periodically attend a special plant course on the techniques of sampling and their significance. The coarse sample is given to the analyst who prepares his own sample by hand. Because the samples are largely glasses, this is not as inconvenient as it may sound.

Mr. Scoon, at Cambridge, prefers to take a selected slice of a hand specimen, rather than to crush the whole sample.

Samples for rock analysis in the Geological Survey of Finland are pulverized by hand in Plattner-type mortars, and then ground in an automatic agate mortar. Geochemical samples, on the other hand, are prepared in automatic crushers and grinders. Soil samples are merely dried, placed in a paper envelope, crushed by blows of a mallet, and sieved through silk bolting cloth. At the Macaulay Institute separate areas are used for soil and plant sampling.

At D.S.I.R. (Jeffery) the sample, whose weight is known, is put through various sizes of Jones splitters to yield a homogeneous sample of about the size needed for the chemical determinations; this also gives a duplicate sample for use if needed. The British Ceramic Research Association Laboratories usually prepare two samples of particularly resistant material; the coarser sample is used for the determination of ferrous iron.

SPECIAL FEATURES, EQUIPMENT, APPARATUS

I. General

An important objective of this tour of laboratories was to see British and European counterparts of the type of equipment used in the GSC laboratories, the majority of which is designed and made in the United States. In addition, attention was paid to items designed locally for specific purposes, a knowledge of which is gained only by personal inspection. The following section lists a number of such items of interest; others having to do with sample preparation have been discussed previously.

II. Special Features

(a) Balance Tables

No commercial balance tables were seen and those of interest made use of several layers of insulating material to minimize vibrations, instead of the weighted floating pedestal used in the GSC laboratories. They varied from a simple cement shelf set into the wall of the room, about 3" above the surface of table underneath (Langmyhr), to a multilayered arrangement at the Swedish Cement Research Institute consisting of metal wall braces which support, in ascending order, layers of rubber, concrete, marble, plastic sponge, four pyramid-shaped steel corner plates and an upper steel plate; this latter is used to support the microbalance, while ordinary balances are supported only by the rubber, concrete and marble. The room adjacent to the balance room is used for tensile strength testing but no vibration problem exists. Working tables are usually built around these wall slabs. At the Geological Survey of Finland a concrete, reverse L-shaped footing is inset into the side walls of the balance room (5th floor), 6-10" out from the rear wall; the foot of the L supports a series of solid rubber balls which in turn support a concrete slab, and a work-table is built around, but not touching, the balance support. At the British Museum (Bothwell) the balances are all enclosed in glass-walled cabinets with double doors (they have a dust problem) which rest on special floating pedestals, in turn resting on tables set on concrete pillars set into the ground (laboratories are in the basement). In England the use of brick pillars is generally favoured; at Manchester the balances rest on 1" thick slate underlain by felt and a layer of lead bricks, whereas at Liverpool the brick pillars have rubber at the top and bottom, and support a slab of concrete finished off with layers of wood and Formica. A microbalance and an ultra-microbalance, in cupboards, are mounted on this table and are free from vibration.

(b) Titration Tables

At the Swedish Cement Research Institute a titration table is used which consists of a large box open at the front and sides, with the rear wall and floor painted white; a fluorescent light, with reflector, is suspended from the roof which is in turn supported by three poles suitable for holding burette clamps. At the Geological Survey of Finland the light source is mounted vertically at the rear of the white-painted titration bench (which also has a canopy) and can be moved horizontally to a desired position; a green fluorescent light is mounted in the canopy for EDTA titrations.

(c) Fume Hoods

At the Swedish Cement Research Institute, the fume hoods have a false glass roof below the light fixture and air-duct opening which collects dirt; the electric outlets in the hood are plastic to prevent corrosion and are explosion-proof, and there is a pattern of threaded holes in the rear wall into which support rods can be screwed, thus freeing the floor of the hood. The hood can also be entered from either side as well. At Manchester the fume hoods are tiled with resistant plastic tiles. The fume hoods at Pilkington Brothers Ltd. (Hartley) are located in the centre of the laboratory and are accessible from both sides; if both doors are open at the same time, however, the fumes blow into the room. At the Hirst Research Centre (Chirnside) the fume hoods are made to a standard pattern worked out by a Works Committee; they are large, doorless, more like a ventilated work-bench than a fune hood, and a gentle but steady ventilation is achieved over the whole rear area through a network of small spaces left between alternating layers of bricks. The hoods used for work with HF are of wood covered with Formica, with PVC fittings and perspex (lucite) windows. In the laboratories of the Overseas Geological Surveys a similar use is made of a vented work bench, in this case using lucite sheets to enclose the area; the fume hoods have secondary exhaust ducts over the door as well as at the base of the rear wall but this has not been much of an improvement.

(d) Miscellaneous

At BCRA Laboratories (Bennett) the bottles of hydrofluoric acid are kept in Pb-lined cupboards.

Dr. Neumann, at the Mineralogical Museum in Oslo, has designed vertical viewing screens for X-ray powder patterns. The doors of these wall cabinets are replaced by panels on which are mounted tracings of X-ray powder patterns, in a definite order; fluorescent lights are mounted behind translucent panels. It is only necessary to hold the unknown spectrum against the mounted tracings to achieve rapid identification.

At the Warren Spring Laboratory, Dr. Jeffery has two interesting peg-boards on display for the benefit of visitors. One is a Periodic Table in which the elements appear as removable pegs; those elements determined in the laboratories are shown in red, those not yet determined are white. The other is a large board which gives at a glance the present status of the laboratory work load and staff; elements are coded by colour, analysts by name, and the board is divided horizontally into a number of holes corresponding to numbers of samples. Vertically the board is divided into projects and by a combination of pegs one can show the elements to be determined, the analyst and the determinations in progress, and the number of samples yet to be done.

At the Overseas Geological Survey, Mr. Curry has a display made up of coloured films of characteristic spectra mounted in a light box that has proved very popular with visitors.

III. Equipment

(a) Balances, all types

In the Scandinavian laboratories the Bunge and Sartorius balances are easily the most popular, with several of the Mettler single-pan balances in use as well. The Sartorius microbalance (MPR 5) is well-liked in several laboratories (Filby, Manheim, Bolidens); the Cahn Electrobalance is used for assay work and in the spectrographic laboratory at the Finnish Geological Survey, and the Mettler microbalance is also receiving favourable attention.

In England and Scotland the Oertling balances are the most popular type, followed by the Stanton balances. There are a number of semimicro Mettler balances in use also; all thermobalances seen were Stanton models.

(b) Spectrophotometers and Colorimeters, all types

Thirteen different types of spectrophotometers and colorimeters are in use in the laboratories visited. In England and Scotland the Unicam instruments are very popular, particularly the SP 500 (UV and visible) and SP 600 (visible) types. Two laboratories, Warren Spring and General Electric, have the SP 700 recording spectrophotometer (UV, visible, near IR) which is used in these laboratories for special work and for research on colorimetric methods. The Hilger Uvispek is used at the Warren Spring, Overseas Surveys and Geological Survey of

Great Britain laboratories (according to Ojanpera (Finland) the flame attachment is not good), and the Hilger Spekker, a simple filter photometer, has proven very satisfactory as well (Finland, Br. Museum, Overseas Surveys). The Beckman DU spectrophotometer, with attachments for UV, nephelometric and flame work, is used at Bolidens and in Langmyhr's laboratories (Oslo), but other laboratories use it almost solely for flame photometry. The Beckman B instrument is also occasionally used for this purpose.

A Zeiss PMQ II non-recording spectrophotometer with flame attachment is used by Langmyhr (Oslo) and it is a very handsome instrument. It utilizes a quartz prism and costs about $4500; it has been used for 3 years now with no troubles. An Eppendorf Photometer (German), a new type of filter instrument with 8 Hg-line filters and vacuum photocell, is used in the chemical laboratories of the Geological Survey of Sweden; it has an AC electronic system, several sensitivity steps, uses Hg, Cd or Hg-Cd lamps, can be used for automatic titrations (galvanometer), and has a lid which closes during use, thus eliminating all stray light. Dr. J.P. Riley (Liverpool) is very pleased with his Optika (Italian) spectrophotometer, a grating instrument giving a band width of 1 Å over most of the range. He uses it for special studies, such as the determination of the perchlorate complexes of Pr, Nd and Sm in monazite, using home-made micro-cells. It is not an instrument for routine use, being somewhat slow in operation, but is very stable to temperature and humidity changes, with a steady dark current, and is easily converted to a flame photometer.

Little work is being done in the infrared region. The Macaulay Institute and Warren Spring Laboratories use a Grubb-Parsons GS 2 Infrared Spectrophotometer (Walkergate, Newcastle upon Tyne 6); this instrument is very different in appearance to other similar instruments, uses a double beam with a very complex system of lenses and vibrating mirrors, takes gases, solids or liquids, has a range of 1-23 microns with a limit of resolution of 1 $cm.^{-1}$ and is said to be the most sensitive IR instrument available. The very narrow chart results in an undesirable squeezing of the IR spectra. The General Electric Laboratories use a Hilger double beam IR spectrophotometer with spectral reflectance attachment for the study of semi-conductors, synthetic crystals and manufactured materials, using the KBr disc technique.

(c) Flame Photometers, all types

Undoubtedly the most popular instrument for the determination of the alkalies by flame emission spectroscopy is the EEL (Evans Electroselenium), a popularity which is well deserved. Its simplicity and reliability make it ideal for routine use; the low temperature flame minimizes interference from other elements. For other elements the Unicam SP 900 is widely used in England and Scotland; Riley (Liverpool) warns against aspirating organic liquids into the spray chamber because of the danger of explosion. The Beckman DU spectrophotometer with flame attachment and recorder is used in several laboratories (Geological Survey of Sweden, Finland, Pilkingtons (2)) although Pilkingtons have not had much success in their attempts to use the recorder trace for quantitative measurements. Only one Perkin-Elmer Model 146 was seen (Geological Survey of Great Britain). The Macaulay Institute uses two home-made instruments based on the Lundegaard flame system; 3 photomultipliers are used to measure Na, Ca and K in an air-acetylene flame, and a special baffle in the spray chamber eliminates large droplets (which cause interference by P in the determination of Ca). The acetylene is moistened before use to prevent the accumulation of air-borne material on the acetylene jet in the spray chamber.

Only one laboratory is using atomic absorption spectroscopy in routine work. The Macaulay Institute uses a home made instrument (Hilger Uvispek, Walsh power pack, Hilger burner and hollow cathode (Co), EEL atomizer and Lundegaard spray chamber and baffle) for the determination of Co in soil extracts (0.2 to 10 ppm); it is regarded here as essentially a single-element technique and simpler than doing Co by the DC arc, since sample extracts can be used. General Electric is studying the use of this technique, using their own burner and detector; they also make their own hollow cathode lamps (Mg, Zn, Au, Mo and Cu) and are working on one that will be continuously evacuated. The Warren Spring Laboratory expect to acquire the necessary equipment soon, starting with a Hilger Uvispek. Hilger and Watts now offer the complete instrumentation for this work; they are now able to do Mg but still cannot do Ca, Al or P.

A visit to Unicam Instruments Ltd., Cambridge, was very rewarding. All of the manufacture, excluding the chassis castings, of their complete line of instruments is done at the Cambridge plant and one is impressed by the careful work that is done, most of it by hand. Demonstrations of several instruments were made. The spray chamber of the SP 900 flame photometer is very efficient; successive atomizing of a solution containing 10,000 ppm. K, water, and one containing 0.1 ppm. K gave a recorder peak for the 0.1 ppm. solution that was only slightly higher than that given when only the 0.1 ppm. solution was atomized. Zeroing of the instrument is rapidly and consistently achieved with water even after the use of the 10,000 ppm. solution. Added advantages of the SP 900 are the venting of the drain to air, a monitoring system for detecting the source of trouble and a simple detachable scanning device. The SP 100 Infrared spectrophotometer is a really magnificent instrument with many remarkable features and amazing versatility.

(d) Automatic Titrators, all types

The EEL Quantitrator, a simple apparatus for automatic colorimetric titrations using various sizes of containers, is found in a number of laboratories (Geological Inst., Norway; Br. Museum; Liverpool; Macaulay Inst.) where it is used principally for EDTA titrations. Other instruments in use include the Radiometer Titrigraph at the Swedish Geol. Survey and at Bolidens where it is used to determine chloride in electrolytic Cu (they plan to get a Kemula attachment for anodic stripping studies), the Lumetron Colorimeter (works well but stirring is slow) and the Baird and Tatlock Potentiometric Titrator at the Finnish Geol. Survey, a Pye autotitrator (B.C.R.A. Lab.) and the Technicon Autoanalyzer at Imperial College; the latter is used routinely for the determination of Zn (250 per day) and the entire operation takes about 15 minutes to process a sample starting with the automatic aliquotting of the sample.

The writer was impressed by the Teflon piston burettes, developed by Metrohm Ltd., Switzerland, which are in use in a number of laboratories. They are available as single or double burettes, with both manual and motor-driven operation. Their capacity ranges from 5 to 20 ml., with scale graduations of 0.005 to 0.02 ml. At Bolidens concentrated NaOH is used as titrant with no leakage; in Finland they are used for potentiometric titrations, at the B.C.R.A. Labs. for EDTA titrations. They are easy to handle and give excellent control over the addition of titrant in very small increments. A similar, home-made piston burette is in use at the Geological Survey of Great Britain (Sergeant and Wilson, Analyst 87, 152-153 (1962)).

(e) Spectrographs and Plate-reading equipment, all types

Sixteen of the laboratories visited use Hilger and Watts quartz and/or glass spectrographs of the small, medium or large varieties and often all three. Most of these are of the plate-reading type but several direct readers are in use also. Dr. Manheim (Swedish Geological Survey) uses a 8-channel direct-reader attachment with a medium Hilger quartz spectrograph to do Cu, Pb and Zn in ore-prospecting samples; at the B.C.R.A. Laboratories a Hilger and Watts 3-metre grating Polychromator (Polyprint) with typewriter print-out, with 30 channels, has just been installed in an air-conditioned room; a similar instrument, modified for DC arc work, is used by Mr. Ward in the Pilkington laboratories, in company with 4 other Hilger spectrographs; the Macaulay Institute has 7 Hilger spectrographs, of which two are of the direct-reader variety, one with 12 channels and the other having only two channels and used for the determination of Mg in plant extracts; the General Electric Laboratories has a 13-channel direct-reading attachment for a medium Hilger quartz spectrograph, and uses it for a variety of special analyses.

At the Warren Spring Laboratories clever use is made of the available ranges of the Hilger spectrographs by using three of them (large quartz and glass, large quartz and medium quartz instruments) with one arc stand. This makes maximum use of small samples; the first two instruments cover the range 2100 - 3500 Å while the medium spectrograph, by means of an automatic shutter, records the volatiles present in the first few seconds of arcing.

Among the other spectrographs in use are a Zeiss Q-24 quartz instrument used for major element analysis in rocks by a pellet-spark technique (Swedish Geological Survey), a Jarrell-ash 3.4 metre plane grating instrument at the Geological Survey of Finland, and a Mannkopf double glass prism (60° and 30°) instrument used for the determination of all the rare earths except holmium.

At Bolidens extensive use is made of an A.R.L. Quantometer with a tape machine attachment. The tape method of Danielsson was developed at Bolidens and is used to determine 30 elements in rocks, sulphides and ion exchange resins. Alkalies are done by means of a Beckman flame attachment and Zeiss triple glass prism connected to the A.R.L. recorder.

The Judd-Lewis Comparator and the Hilger and Watts non-recording densitometer were the most popular plate-reading instruments found. The Jarrell-ash console densitometer is used at the Finnish Geological Survey, and also (in the slightly different model made by Hilger for Jarrell-Ash) in Mercy's laboratory (Edinburgh). Riley (Liverpool) uses a Joyce-Loebl Recording Microdensitometer, as does the Geological Survey of Great Britain; this instrument has an automatic scanning facility and plots the density vs wavelength, the scanning rate slowing automatically for high density areas of the plate, and both laboratories speak highly of it. The Macaulay Institute also uses a Leeds and Northrup recording densitometer. The only other instrument observed was a Schnellphotometer (VEB Carl Zeiss, Jena) of which Dr. Manheim (Sweden)spoke highly.

(f) X-ray Powder, Diffraction and Fluorescence Equipment

Observation of this equipment was incidental only and the following comments are by no means exhaustive in nature.

The Geological Institute in Oslo has acquired a new Philips Vacuum X-ray Fluorescence Spectrometer, with Diffractometer unit, but it was not yet in operation. The Philips equipment was found in many laboratories, including those of the Swedish Geological Survey, Bolidens (at Rönnskar and at Laisvall), the Geological Survey of Finland, Manchester, Pilkington, Mercy (Edinburgh), Oxford, Cambridge, Imperial College, Warren Spring and the Overseas Geological Survey.

Bolidens has installed an A.R.L. VXQ 70000 X-ray Fluorescence Quantometer with 5 fixed channels (Ni, Cu, Zn, Pb, Fe) and 3 scanning channels; they hope to use an ion exchange technique in the preparation of the sample. The Dept. of Geology at Leeds is working out methods for their new Siemens - Halske Vacuum X-ray Fluorescence Spectrometer (LiF and Gypsum crystals only) which is also equipped to do diffraction studies; it is to be used for rock analyses (major elements except Na and K). The B.C.R.A. Laboratories are to acquire an instrument similar to the Philips autrometer to compliment the coverage given by their new Polychromator.

Three electron microprobes were observed in operation, all of them Cambridge instruments. Pilkington Brothers use their instrument for the study of new glasses and refractories; at Imperial College it is used by the Departments of Metallurgy and Physics as well and will handle polished sections only. The original instrument in the Dept. of Mineralogy and Petrology at Cambridge is still in use but a newly-designed one is in the course of construction; a study of iron meteorites is in progress.

(g) Heating equipment, all types

Good-looking muffle furnaces, with a convenient method for opening the door, are made by Heraeus, Germany, who also make a quartz infrared lamp that is useful for evaporation. Surface evaporation is favoured by Dr. Riley (Liverpool) and he uses fused silica radiant heaters (Thermal Syndicate, England) that cost about $50 for one 6" in dia., 750 watts. Muffle furnaces from Rudolph Grave AB, Stockholm, are circular well-type furnaces that will reach 1100°C, with a simple temperature control and will hold eight 1" graphite crucibles; they cost about $80 each. A small electric furnace for both ashing and ignition, which takes the ordinary size of crucible, is made by Erich Tschacher, Laboratoriumbedart, Bielefeld, Germany, and is very useful. Sergeant, at the Geological Survey of Great Britain laboratories, uses a Radyne Ltd. Induction Furnace for the determination of water and, possibly, for CO_2 as well; it can also be used for the ignition of Pt crucibles. A very nice Ultrathermostat, made by Haake, Berlin, is used by Langmyhr (Norway) for potentiometric titrations with a Zeromatic pH meter; it is good to ± 0.05°C.

(h) Miscellaneous equipment, all types

Mr. Brunfelt, Geological Institute, Oslo, is using a foil machine for the determination of rare earths by neutron activation. The irradiated rare earths are separated by ion exchange and then eluted; the eluate drops on a moving strip of aluminum foil which passes under infrared lamps, the spots are dried and then pass under a Geiger counter connected to a recorder which measures the radioactivity of the eluate.

The chlorination apparatus described by Moss, Hey and Bothwell (Mineralog. Mag. 32, 802-816 (1961)) was demonstrated at the British Museum (Natural History). It is necessary that all water be absent before chlorination of the meteorite sample is started, otherwise the silicates are also attacked. The chlorination is now started 'cold' with a mixture of nitrogen and chlorine after an initial heating with nitrogen to flush out water, so that the reaction can start (150°C) in dilute chlorine.

At the B.C.R.A. Laboratories a small desk calculator (VEB Trumphator, Werk-Molkau, Leipzig, East Germany) has been found very useful; it costs approximately $75.

Polarographic methods were in use at only three of the laboratories visited. Riley (Liverpool) uses a Tinsley recording instrument for the simultaneous determination of Ni, Co, Zn, Pb and Cu in sea-water after preconcentration by solvent extraction; at Imperial College a Barker square-wave polarograph is used for the determination of Cd; it was originally intended for the analyses of natural waters but the preliminary sample preparation proved too cumbersome. The D.S.I.R. Laboratories (Jeffery) employ a K 1000 cathode ray polarograph to do Cu, Ni, Pb and Zn (5-20%, ± 5% accuracy).

Langmyhr (Norway) uses a home-made polyethylene weighing pipette for taking weighed aliquots of HF solutions; the tube is about 6" long, with a bulb at one end and a cap at the other, and is suspended from the balance arm by fine wires. For the determination of FeO the sample is decomposed in a Teflon cup on a small hot plate, over which is placed a Teflon hood through which nitrogen is forced. When the aliquot is taken for the colorimetric determination of Fe" both the weighting pipette and beaker into which the aliquot is placed are filled with nitrogen. It was interesting to see that stiff feathers are still being used to brush off the balance pans.

Dr. Runnholm (Sweden) reported that they obtain 100 ml. Teflon crucibles, with lid, from a concern in Stockholm for $10 each. He also uses an automatic dispenser, capacity 0-10 ml., made by Wiklund Glass, Stockholm, that is very accurate.

Ruff-shaped cooling fins made of copper strips fastened around a suitable copper ring are used at Bolidens to cool the ends of combustion tubes; we have tried them and they are very effective.

Mr. Nurmi, at the Geological Survey of Finland, has made a special spoon for sampling material in the geochemical laboratory; it is shaped like a pair of scissors or a candle-wick trimmer and one can scoop up the sample and level it off to a predetermined amount in one motion.

Dr. Mercy (Edinburgh) uses a Scorah automatic all-glass water still (L.V.D. Scorah, 44 Northfield Rd., Kings Norton, Birmingham 30) that is fully automatic; see Nature, Feb. 16, 1963, p.CCXLV, for picture.

At the Macaulay Institute use is made of a number of home-made items. A hot plate is made from a sheet of Al (with 5% Mg), with 6 ordinary heating elements strapped to the underside; it rests on a transite box fitted with an Al reflector, and can be heated to 400°C. Bunsen burners have had the barrel replaced by a silica tube fitted with a Teflon sleeve, to eliminate Cu contamination.

The Warren Spring Laboratories use small Kjeldahl flasks for the rapid decomposition of ore samples; the flasks are supported in 2" dia. steel rings (3" deep) embedded in a sandbath; 0.1g. can be decomposed in 30 minutes with 3 acid dehydrations.

MISCELLANEOUS USEFUL TECHNIQUES

Most laboratories have developed, in addition to specialized equipment and apparatus, some special techniques or laboratory practices that are often very useful to others but which are little publicized. A few of these 'helps' are included here.

Langmyhr (Oslo) prepares his standard ferrous iron solution by dissolving Johnson, Matthey and Mallory iron sponge in acid and reducing the ferric iron with a silver reductor, and stores it under CO_2 in a Kipp generator operating on marble chips and HCl.

Mr. Bothwell (British Museum), in the NaOH fusion of a sample for the preparation of Solution A of the rapid methods scheme, places the sample on one side of the bottom of the nickel crucible; the crucible is placed over a burner in a tilted position such that the sample is away from the direct flame, and NaOH pellets are placed at the lowest point of the crucible interior, melted over a low flame and then mixed with the sample by restoring the crucible to a level position. Spattering of sample during melting of the NaOH is thus avoided.

At Pilkington Brothers Ltd., the practice is followed of quenching platinum crucibles after a fusion to enable easy removal of the cold melt; the crucibles become very deformed (cuspidor-shaped) but last about two years before replacement is necessary. Much analytical time is saved by this procedure. They also find it better to sample foundry sands when the sand is damp; when the sand is dry the fines tend to segregate and, since feldspar tends to concentrate in the fine fraction, it leads to misleading values for trace constituents such as iron.

In the Geology Department at Oxford the practice is followed of submitting a piece of mica and feldspar, picked from a rock submitted for Rb-Sr age determination, for quick qualitative analysis by emission spectrography to determine if the rock warrants further treatment. To reduce the amount of dirt tracked into the laboratory used for the lead extraction and Rb-Sr separation work, a layer of 'tackymat', a linen mesh covered with viscosine, serves as a mat. Dr. Hamilton also concentrates the Pb present in feldspars by adding a small amount of Dowex 1 resin (80 - 100#, 8% cross-linkage) to the solution of the sample in 1N HCl, stirring and then filtering and washing the resin. He thinks that a resin-impregnated membrane would also do this.

The writer was impressed with the facilities for sample irradiation in the neutron activation analysis laboratory at Harwell (Mapper). Two slots in the DIDO and BEPO piles are connected by pneumatic tubes between the piles and the laboratory; the course of the 'rabbit' containing the sample is regulated and followed by a control board in the laboratory, giving rise to truly 'instant' irradiation.

Mr. Scoon (Cambridge) finds that the filtration rate is speeded up by fabricating a vertical loop into the funnel stem.

At the Overseas Geological Surveys, Mr. T. Deans uses an ultrasonics bath to break up soil samples without destruction of delicate crystals; he also finds that immersion of a pycnometer in the bath will facilitate the removal of air bubbles during the determination of powder density, and that good dispersion of material in centrifuge tubes, prior to centrifuging, is obtained in this way. A dental drill is very useful for showing the characteristic streak of minerals in hand specimens. Specific gravity determinations are made with manual spring balances of various sizes and a bucket of water; the determination can be done so rapidly that no porosity problems are encountered and results are quotable to the second decimal.

RECORDING OF ANALYTICAL AND SAMPLE DATA

Inquiries were made into the methods used for the recording of sample and analytical data in the laboratories visited. Most of them have no problem because they deal with a relatively small number of samples, and the information can be easily recorded on small-printed cards. Only one laboratory used a form of automated retrieval of data.

The card used by the Geological Survey of Finland is typical of those in general use, and is a condensed version of the Rock Analysis Record form still in use at the G.S.C. General information is placed on the face of the 4" x 6" card, together with the analytical results if the card is for an ore sample; silicate and spectrographic analyses are placed on the back of the card. One copy is sent to the geologist, the other is retained in the laboratory and any pertinent notes are attached to it.

Some 30,000 samples are handled yearly by Pilkington Brothers Ltd., and Mr. Hartly considers his sample and analysis record system to be simple and foolproof. Books of vouchers, each stamped with a code letter and serial number and colour-coded as well, are sent to the seventeen firms who submit samples; the individual requiring analyses fills in the details of sample nature, number and analysis requested on a voucher, tears from it a perforated, gummed label bearing a duplicate set of the code letter and serial number and attaches this to the sample container, and sends both voucher and sample to St. Helens. Samples and vouchers are collected and examined by a senior chemist, who distributes them to the appropriate analyst. The analyst writes his results in pencil on the back of the voucher; if more than one analyst is involved, the additional results are submitted to the first analyst on a preliminary analysis form and the latter collates the results with his values. The voucher is returned to the senior chemist who reviews the results for correctness and completeness and then sends it, via pneumatic tube, to a typist who transcribes the data on a standard analysis form for return to the works chemist who submitted the sample, with copies to the works manager and the central laboratories files. Analyzed samples are stored in basement rooms and systematically reduced in number over a year until only a few representative samples are left as a permanent record.

In the Department of Spectrochemistry at the Macaulay Institute (Aberdeen) the sample data are entered by hand in both a register and laboratory record book, the samples being numbered in numerical order as received. The analytical data are entered by hand in the register and in the laboratory record book the elements are ticked off when done. When the results of an analysis are ready for release the values are ticked off in red; when typed the results are crossed out. No special report form is used and results are sent by letter. The sample, incidentally, is kept in the sample preparation room and weighed aliquots are brought up to the laboratories. A card index file for methods, maintained since 1920, has a 3 way index - catchword, subject and author.

Service Request and Sample Advice forms are required at the Warren Spring Laboratory of D.S.I.R., Stevenage. The Sample Advice form, a small simple form which requests description of sample, analysis required and project number, accompanies the sample to the sample preparation room and from thence to the laboratory. The sample is assigned a serial number (e.g. 61/2120, 61/2121, etc.) and this, together with the date and other data are entered by hand in a register. A rough work sheet is maintained which shows the progress of work on all samples; when the analysis is finished the results are checked by a senior chemist against the Service Request form and the handwritten results are sent or telephoned to the submitter. Results are also entered on a small analysis record card, with a notation about the methods used.

The Analytical Laboratories of the Overseas Geological Surveys have an elaborate system of coded cards for both sample and analysis data, and for analytical abstracts of pertinent methods and equipment. The system has two parts, (1) a Shannofan buff-coloured, small index card and, (2) a large Brich Vistem visible card index (also called a 'peek-a-boo' card). The sample is received by Mr. Bennett and, after a preliminary mineralogical examination by Mr. Deans, it is given a serial number (in numerical order as received, e.g. 3602, 3603, etc., but a series of samples from one customer is numbered 3602A, 3602B, etc.) and pertinent data are entered on the Shannofan card, including a code number to indicate where the sample is to go, and one to indicate where the sample is at the finish; the analytical data are added later. Cards are filed in numerical order in batches of 30, and coloured tabs in various positions on the top edge of the card facilitate rapid identification of any card. The Brich Vistem visible card index consists of large "feature" cards having 2,500 numbered squares printed on the body of each. When a feature card is applicable to a given sample, the country of origin, type of material, work done, elements determined and, where pertinent, the mineral name are recorded on the card by punching the appropriate square. If it is desired to know all samples of ilmenite from Tanganyika that have been analyzed for vanadium, the Tanganyika feature cards are placed over those for ilmenite, vanadium and chemical analysis and holes will go through all the cards in the squares corresponding to the required sample reference numbers. Further data are obtained by reference to the appropriate Shannofan cards. The results of the analysis are sent out on a special form which lists the constituents (27) both by name and chemical symbol.

ROCK AND MINERAL ANALYSIS - SAMPLE ANALYSIS AND RESEARCH

I. General

It was first planned to summarize the methods used in terms of the individual constituents of rock and mineral analyses but this led to a monotonous repetition of names and places. The methods used are summarized instead for each laboratory, following the order of the Itinerary, and the constituents are discussed in the conventional order in which they are usually tabulated.

II. Chemical methods

University of Oslo

Dr. Langmyhr is investigating a new scheme for the rapid and accurate analysis of silicates. A 200 mg. sample is decomposed in a Teflon crucible by

HF and $HClO_4$, under nitrogen. Aliquots are removed with a special weighing pipette for Si and Fe^{++} (only 0.2 mg. of SiO_2 is lost from 46 mg. SiO_2 present when a 1:3 HF solution is evaporated from 25 to 2 ml at 100°C), and the remaining solution is evaporated with H_2SO_4 to remove fluoride, diluted, transferred to a weighing pipette and aliquots taken for Mn, Ti, total Fe, P, Al, Ca, Mg, Na and K.

Si is done as the yellow silicomolybdate, using differential spectrophotometry. Al is separated from Fe and Ti in the R_2O_3 ppt. by extraction with cupferron and $CHCl_3$, then back-titrated with Zn (plus dithizone) after complexing it with EDTA. Fe^{++} is determined colorimetrically with orthophenanthroline, total Fe and Ti by Tiron, the Ti after reduction of the Fe with dithionite. Ca, Mg, Na and K were previously done flame photometrically on the filtrate from the ammonia separation but Ca and Mg are now done by EDTA titration, using Calcon carboxylic acid as indicator. Mn is done with KIO_4 (Nydahl's method with $AgNO_3$ and $(NH_4)_2S_2O_8$ did not give good results), P by the usual phospho-vanado-molybdate complex, and CO_2 and H_2O by standard methods. All work has been done on synthetic samples; one year was spent on the investigation of individual methods of determination and another on testing the effect of the various elements on each other. The scheme will eventually be tried on G-1 and W-1.

Studies are also being made of the decomposition of minerals by HF and $HClO_4$ in Teflon cups at steam bath temperatures, and of the determination of traces of Se with 2, 2-dianthrimide in 95 per cent H_2SO_4; in the latter there is a high reagent blank and colour development is slow, but it is specific for Se (IV).

Mineralogisk-Geologisk Museum, Oslo

The classical methods are used by Mr. Bruun but Dr. Ball is introducing the Shapiro and Brannock (hereinafter referred to as S-B) rapid methods, with his own modifications. Ca is titrated by EDTA using the EEL titrator with murexide + NaCl as indicator; Ca and Mg are similarly titrated at pH 13 with Calcon + Naphthol

Green B in methyl alcohol. Dr. Ball is also working on the determination of Si by a radiochemical method; a 200 mg sample is fused with NaOH in a nickel crucible after the addition of approximately 1 mg Si tracer, the SiO_2 is dehydrated, redissolved in NaOH and reprecipitated as the quinolate molybdate. The method is slow and high results have been obtained; it may be possible to omit the second precipitation. He hopes to do Al by a similar procedure, the Al to be precipitated as the oxinate or hydroxide following an ion-exchange separation.

Geological Survey of Sweden

In the chemical laboratories some classical rock analysis is done but more effort is devoted to the S-B methods, with few modifications. The molybdenum blue method for Si is used for most work such as the analysis of alum shale ash, but Bloxham's procedure (yellow complex) is used also, although it is very variable in behaviour and P interferes. The determination of Al with Eriochromcyanin is free from Ti interference, but V and high Ca cause trouble. They have had difficulty with the EDTA titration (with EBT) of small amounts of Mg. Mn is done by the Lingane-Karplus method.

Total sulphur (1-40 per cent) is done by combustion, with absorption in H_2O_2 and final acid-base titration. Suction, not pressure, is used to draw O_2 over the sample in a porcelain boat. Zn in sulphides is dissolved in H_2SO_4, made 2N in HCl, placed on Dowex 21 resin; Fe and Cu are eluted first with HNO_3, then Zn which is finally titrated with EDTA. Acid extraction and precipitation, rather than fire assay, is used to determine gold.

Bolidens Gruvaktiebolag, Skelleftehamn

No routine chemical rock analysis is done here but various elements are determined chemically in a variety of geological, geochemical and smelter samples. Total sulphur (0.01 - 30 per cent) is done combustimetrically as at the Geol. Survey of Sweden; for high sulphur (50 per cent or more) a double absorption is used and the combustion boat is placed in an inner short silica tube during combustion which is then washed with water, the washings being combined with the absorbing solutions before the titration; suction is used rather than pressure and the sample is either burned alone or mixed with asbestos. For samples with high As content, ZnO is added. Selenium is finished gravimetrically after distillation with HBr and hydroxide precipitation or, in trace quantity, is done colorimetrically with diaminobenzidine; the latter is specific for Se and no distillation is necessary. Zn is done by EDTA after ion exchange separation, Pb by ferrocyanide titration, with o-dianisidine as outside indicator, after separation as $PbSO_4$ and solution in ammonium acetate, Bi and Sb by precipitation on MnO_2, ascorbic acid reduction and colorimetric finish by KI (both on same solution by varying the KI concentration). H_2SO_4 in mine water is titrated directly with concentrated NaOH using a Metrohm Piston Burette; no leakage occurs around the Teflon piston. Au and Ag are done by fire assay, Cu by double electrolysis (raw Cu is plated out, redissolved and re-electrolyzed). S and Cl (in refined Se) are done nephelometrically as AgCl and $BaSO_4$; Pb and Zn in mine air (the paper collector is dissolved), and in tailings, are finished polarographically. Chloride in electrolytic Cu is determined by potentiometric titration using a Radiometer Recording Titrigraph which can be set to run overnight for slow reactions; they plan to get a Kemula attachment for anodic stripping-hanging drop work.

Fluorine is done by pyrohydrolysis in a wire-wound ASEA furnace, with an O_2 - H_2O flow of 200 ml/min (the flow rate recommended in the literature is too high) at a temperature of 75°C; the sample (1 g), mixed with 1 g WO_3, is placed in a porcelain boat with 0.5 g WO_3 as cover and pyrohydrolyzed at 1,000°C. The fluoride is absorbed in water only (without cooling); if NaOH was present then S (as SO_2) would be absorbed also. Final determination is by the chloranilate colorimetric procedure or by titration with $Th(NO_3)_4$; used for 0.01 - 1 per cent fluorine and good reproducibility is achieved.

Oxygen in metals, especially in Pb, is done combustimetrically with H_2, the H_2O formed being titrated with Karl Fischer reagent.

Mercury, when present in significant amount, is separated from Cd and Zn by sulphide precipitation; the precipitate is mixed with Eschka mixture and iron powder in a Selas crucible, covered with a weighed disc of Au foil and heated at 1,200°C for 1 hour with water cooling of the upper part of the crucible. The Hg collects on the foil and is weighed. Trace amounts of Hg are volatilized without prior precipitation and then dissolved from the foil and determined colorimetrically with dithizone.

Geological Survey of Finland

The Geochemical Laboratory in the Ore Prospecting Department has a staff of 3 and handles about 50,000 samples per year (soil, water, peat). No grinding is done, samples are dried in an oven, placed in a paper envelope, pounded briefly and then sieved through silk cloth. Acid extraction methods are used, followed by visual colorimetric determination of Cu and Zn with dithizone; other colorimetric finishes are used for Pb, Mn, Mo, Sn and other elements.

In the chemical laboratories a mixture of the classical and S-B rapid methods are used for rock and mineral analysis. Si is generally done gravimetrically but the Mo-blue method is preferred when appreciable fluorine is present. Al determination is usually gravimetric but some EDTA titrations have been tried and the use of ion exchange columns is contemplated. Total iron is done by the $FeCl_3$ method, now largely superseded by better methods. The Metrohm Piston Burette is used for the titrations of Ca (murexide) and Ca + Mg (EBT) with EDTA. The alkalies are determined flame photometrically after acid decomposition of the sample; Cs_2SO_4 is added as a radiation buffer for all alkalies (except Cs), K_2SO_4 is added for Cs. Total water is done both by the Penfield method and by combustion in a porcelain boat and absorption in anhydrone. Heating is by a propane flame, no flux is used for light-coloured samples, CaO + PbO is used for samples high in fluorine, PbO for those high in Fe. Fluorine is separated by distillation with $HClO_4$; no pyrohydrolysis. Cu, Ni and Zn in ores (0.05 - 15 per cent) are done simultaneously by a polarographic method; Pb is done separately and gravimetrically.

No laboratory manual, except for ore analysis, is available; methods are taken from the literature and from personal notebooks.

Peat ashing is done in a separate laboratory and the ash is analyzed for Ca, Mg, Mn, P and Fe, as well as for trace elements; nitrogen is determined on the peat by the Kjeldahl method.

British Museum (Natural History)

Silica is determined by the Mo-blue method, with a sulphite reductant. Mr. Bothwell mixes the tartaric acid and sulphite reductant before their addition; he has found that the longer the delay between the addition of the tartaric acid and the reductant, the lower the values for Si that are obtained. He uses Ni crucibles for the NaOH fusion of the sample; he tried Ag crucibles without success. The sample is weighed into the crucible by difference and a standard having a similar SiO_2 content is run also; a working curve rather than a factor is used.

Alizarin red S is used for the aluminium determination, with no correction for the effect of titanium. Iron is determined as the sulphosalicylate complex; no reduction needed, merely add reagent, NH_4OH and shake.

Total water is checked quickly by the S-B method, then done by the detailed Gooch procedure with Na_2WO_4 to seal the tube. The S-B method is also used as a quick test for CO_2.

Fluorine is separated by distillation with H_2SO_4 from the filtrate from a Na_2CO_3 - ZnO fusion and determined colorimetrically with Solochrome Cyanine R.

Geology Department, University of Manchester

The Mo-blue method was used for the determination of silica but has been replaced by the method of Jeffery and Wilson (gravimetric plus a colorimetric determination). NaOH fusions are done in silver crucibles, and the solution is only used for the determination of silica.

The S-B Solution B is prepared as usual but alternative finishes are used if the sample contains resistant minerals. If the resistant minerals are known to be free of alkali metals (e.g. oxide minerals such as corundum, rutile, spinel) then the residue is separated by centrifugation, fused with $K_2S_2O_7$ and put into solution; the first solution is used for the determination of the alkalies, a mixture of the two solutions for the determination of other constituents. Resistant silicate minerals (e.g. staurolite, tourmaline) resisting the acid attack are first separated by centrifugation; if the alkali content is negligible the residue is fused with NaOH in a silver crucible, put into solution and treated as described for the resistant oxides above. If the resistant minerals contain alkali metals, then solution is achieved by acid attack (HF + $HClO_4$) in a sealed Teflon-steel bomb heated at 150°C for 3 to 4 hours in an oil bath; this solution is mixed with the first acid solution and used for all determinations except SiO_2 and ferrous iron.

Aluminium is determined as the oxinate following extraction in chloroform; Fe is complexed with dipyridyl. $BeSO_4$ is added to complex any traces of fluoride present. It is essential that the pH of the extraction be 4.9 - 5.0.

Total Fe is measured as the red ferrous dipyridyl complex, Mn as permanganate after oxidation with $(NH_4)_2 S_2O_8$ in the presence of H_3PO_4 and $AgNO_3$.

The method of J.P. Riley for the removal of interfering elements by continuous extraction of their oxinates with $CHCl_3$ prior to the EDTA titration of Ca and Mg was found to be too tedious and has been abandoned in favour of a single R_2O_3 precipitation; it is hoped to do them flame photometrically with a Unican SP 900 instrument, now used for Rb and Cs (Na and K are determined on an EEL flame photometer).

Phosphorus is determined by a single solution molybdenum-blue method, with ascorbic acid as the reductant. The simultaneous method of Riley is used for H_2O and CO_2.

Professor Vincent dislikes old-fashioned methods but prefers the classical approach rather than "Shapiro and Brannockry". He favours microanalysis, and mineral analysis over whole-rock analysis. He does an R_2O_3 precipitation and then determines all other constituents of the precipitate, including Pt, on aliquots of a separate sample to obtain aluminium by difference. Flame photometry is used for the alkalies but he prefers a neutron activation method for small amounts (he has experienced trouble with isotopic dilution methods). A neutron activation method is used for the determination of phosphorus; it is separated as ammonium phosphomolybdate and care must be taken to get the proper compound which must be dried in air, never in an oven. He thinks it may be possible to determine S by irradiation of the sample and measurement of the P^{32} formed. Ferrous iron is done by Wilson's method (decomposition in presence of ammonium metavanadate); Pratt's method was used but low results were obtained with olivines. Dr. Vincent has not yet found a good method for sulphur, especially when present in small amounts.

Department of Oceanography, University of Liverpool

Dr. J.P. Riley and his students have done much to introduce a new type of "rapid" method of rock and mineral analysis, methods which are semimicro in nature and of high accuracy but which, because they avoid many of the tedious steps of classical rock analysis, are faster and more conducive to batch analysis. He is now concentrating on methods for the anionic and cationic analysis of sea-water and his present studies include the following:

(1) Salinity, previously derived from the chlorinity measurement, is now determined by a conductimetric method having a precision of 1:15,000 when weighed samples are used. He thinks that the Ca content of sea-water varies from place to place.

(2) Fluoride is measured with alizarin complexone using the La salt rather than Ce and an acetone solution of the reagent to increase both stability and sensitivity. The distribution of F is not constant and greater concentrations are found in North Atlantic waters.

(3) $BO_3^{\equiv}$ is being done with curcumin in phenol, a modification which Dr. Riley finds gives high sensitivity; colour development is done in an oil bath at 110°C and the time of heating is not important.

(4) The cations Na, Ca, Mg, K and Sr are separated by ion exchange. Amberlite CG 120 acid form resin in a 14 cm-column is saturated with sea-water (30 ml), and Na + K are partially flushed through; 200 ml. 0.15 M NH_4Cl removes Na + K (combine extracts, add H_2SO_4, evaporate and fume off excess acid, weigh, determine K by tetraphenylboron and Na by difference), 0.35 M NH_4Cl removes Mg (EDTA titration), acetylacetone at pH 9.2 will remove Ca (EDTA titration), 2N NH_4Cl will remove Sr (flame photometry of acetone solution of evaporated extract). Dr. Riley tried to do ion exchange separations in silicate rock analysis (similar work is reported under way by A.D. Maynes at Cal. Tech.) but found that while it works for isovalent elements, it will not work well for a mixture of elements of different valences.

(5) Dr. Riley uses Sb as an accelerator in the Mo-blue method for Phosphorus (ascorbic acid reductant). He uses only redistilled water not purified by ion-exchange because water from the latter always contains some organic matter.

(6) Ni, Co, Sn, Pb and Cu in sea-water are to be preconcentrated by solvent extraction, then determined simultaneously by polarography.

Is the oxidation of <u>ferrous iron</u> during the acid decomposition (Pratt) of the sample as likely as is now believed? Dr. Riley removes the crucible cover and stirs the contents when necessary and decompositions have been made in uncovered crucibles without deleterious oxidation. A fusion method is preferred for refractory minerals; the sample plus metafluoroborate is fused in a 5 mm diameter vitreosil tube sealed at one end, while the tube is strongly evacuated to less than 0.1 mm Hg, then cooled (tube cracks), dissolved in air-free water and titrated using a semimicro burette. This works well for staurolite.

British Ceramic Research Association Laboratories

Material analyzed in these laboratories include glass sand, aluminous silicates, all types of glazes, bone ash and strange, new ceramic materials (inorganic polymers). Analyses are made by the modified classical methods as outlined in Methods of Silicate Analysis by H. Bennett and W.G. Hawley (British Ceramic Research Association, Stoke-on-Trent, 1958); a revised version is to be ready by 1964 and may include emission spectrographic methods also. All analyses are done in duplicate for Si, Al, total Fe, Ti, Mn, Ca, Mg, Na, K and Li, with S, CO_2 and total H_2O as extras; the loss on ignition is determined in a Pt dish, followed by fusion (sinter) of the sample in the same dish. One sample is finished about every 2¼ days. Two general methods are in use, one for precise work and the other for routine control.

In the precise method $\underline{SiO_2}$ is determined by a single dehydration (0.5 ml H_2SO_4 is added to the HCl solution of the sinter before dehydration to prevent precipitation of Ti) followed by colorimetric determination of the residual SiO_2 as the yellow phosphomolybdate complex or the Mo-blue complex if $PO_4^{\equiv}$ is present; the SiO_2 not recovered in the dehydration amounts to 0.1 - 0.3 per cent for SiO_2 = 95 per cent. An HF - H_2SO_4 solution of the sample is used for other determinations and the interfering elements are removed by a cupferron-chloroform extraction (it is essential that Fe and Ti be removed) prior to final determination of Al (1) gravimetrically as the oxinate (Mn does not interfere if precipitation is done in acid solution in presence of acetate buffer), or (2) by EDTA with a back-titration by Zn; the latter method is faster and more accurate.

The routine control method requires an accuracy of about 0.25 per cent for Al and Si and is applied to samples containing up to 99 per cent SiO_2 and 40 per cent Al_2O_3. SiO_2 is done as the yellow complex (1½ hrs) and gives very good results; HNO_3 is used after the NaOH fusion rather than HCl because $FeCl_3$ interferes with the measurement of the complex (a blank does not help). Hill's method (Anal. Chem 28 1419 (1956)) is used for aluminium; by the combined use of sodium thioglycollate and EDTA with blank and sample aliquots, aluminium may be determined directly and specifically by Eriochrome Cyanine R. Mr. Bennett prefers to use the HF solution of the sample rather than the NaOH solution (as in S-B) because of the tendency for Ni crucibles to absorb Fe and then transfer it to other Fe-poor samples. For very highly aluminous materials (e.g. 99 per cent Al_2O_3) a fusion is employed because of the incomplete action of HF on this material; the SiO_2 is determined spectrographically, and Al by the EDTA method mentioned previously.

A new analytical scheme has been devised for the rapid analysis of magnesite but is not ready for publication; the sample is dissolved in HF, the SiO_2 is determined by emission spectrography, Al as the oxinate, Mg and Ca by EDTA. For chrome-magnesite an extraction of the Fe and most of the Cr is made with isobutyl methyl ketone before proceeding as above.

When appreciable fluorine is present the quinoline silicomolybdate method for SiO_2 is preferred over the Berzelius procedure. Fluorine is done by a pyrolysis method.

Pilkington Brothers Ltd., St. Helens

Conventional methods of silicate analysis have been replaced by the S-B rapid methods when the latter are equal in accuracy and reliability to those replaced. A combination of methods is used, e.g. Si and Al (chem.), Ca, Fe, Mg (emission spec.) and Na and K (flame phot.), and an analysis is usually finished in 2 to 2½ days. Silica is measured as the Mo-blue complex, using differential spectrophotometry; they are considering use of the quinoline silicomolybdate method instead. Iron in foundry sand (5 to 10 grams) is titrated with $TiCl_3$ when high; lower amounts were done by emission spectrography but it was found that a colorimetric method (thioglycollate) was faster because of a simpler sample preparation procedure. The alkalies are done flame photometrically by a method almost identical with that used at the G.S.C.; an unsuccessful attempt was made to take measurements from a recorder trace (Beckman DU).

Fluorine is separated routinely by pyrohydrolysis on samples containing 0 to 5 per cent F, but even CaF_2 has been done. The sample, in a Pt boat, is mixed with U_3O_8 and heated in a ¾ - inch diameter silica tube at 1,000°C for 30 minutes, with a flow of 2½ to 3 ml/min of distillate, and collected in 0.1N NaOH. A final visual titration by $Th(NO_3)_4$ (0.005 M) is used, with monochloracetic acid buffer and alizarin red S. Amblygonite has been done and P does not interfere; for phosphates or Zn glasses a vitreosil boat is used.

W.H. Herdsman Laboratories, Glasgow

Analyses are done singly by conventional methods; attempts to use the S-B methods were unsuccessful. The Jeffery and Wilson method for SiO_2 (gravimetric and colorimetric) was tried but it was found just as easy to do a second dehydration. The facilities for the introduction of the newer methods are, however, limited.

Department of Geology, University of Edinburgh

Dr. Mercy had many comments to offer on the methods used in his laboratory. The Mo-blue method for SiO_2 is generally good but Mg interferes (our experience also) and close control of the pH (use pH meter, pH = 1.2) during initial complex formation is necessary; he adds the tartaric acid and reductant together. The principle source of error, he believes, is in the optical measurement; cells should be given minimum handling (fill by means of a pipette, empty by suction). He does not favour differential spectrophotometry as a method of measurement. No interference encountered from up to 10 per cent F, $PO_4^{\equiv}$ less than 0.5 per cent). He is enthusiastic about the quinoline silicomolybdate method but emphasized the need to test new methods on all kinds of rock types (amen!). The modification of the classical method using gelatin is also very good, a single precipitation being sufficient. The Bloxham method is spoiled by Fe^{+++} interference, and acetone stabilization of the complex is without effect.

The determination of Al by difference has been abandoned in favour of Riley's method involving extraction following an initial cupferron-chloroform separation of interfering elements. Fusion of the sample and leaching to separate Al is questioned on the grounds that magnesium hydroxide adsorbs Al strongly. Fe^{+++} interferes and the use of a a-dipyridyl is recommended instead of $NH_2OH \cdot HCl$ when Fe is high.

Total Fe is determined by fusion of the sample with 1:1 Na_2CO_3 + $Na_2B_4O_7$ in a Ag crucible, solution in HCl, reduction by a Ag reductor and titration with $K_2Cr_2O_7$. If acid decomposition is used, HNO_3 causes less spraying than HCl. Dr. Mercy is investigating the relative usefulness of four methods (o-phenanthroline, thioglycollic acid (thiovanadic acid is less unpleasant), salicylic acid and a-picrolinic acid) for total Fe determination, including the effect of possible interferences. Ca and Mg are titrated visually with EDTA, following R_2O_3 separation; Dr. Mercy wants to use an EEL titrator instead. For low-Ca samples (olivines and ultrabasics) the Ca + Mg is done as usual (EBT), then a known amount of std. Ca solution is added, and the Ca precipitated as oxalate (tungstate is no good); filter, ignite and dissolve, add $MgCl_2$ solution until the Ca/Mg is approximately 1:1 and titrate Ca with EDTA (calred). When the alkalies are determined by the EEL flame photometer, no interference of Na and K with each other is encountered but interference occurs when the Unicam SP 900 is used.

Dr. Mercy has devised a Teflon crucible for the preparation of Solution B of the rapid methods scheme. It is machined from a 1-inch Teflon rod, leaving a 3/16 inch centre pole which projects about 1/4 inch beyond the open end of the crucible. A cover with a central hole fits snugly over the centre pole and can be raised or lowered to any desired position. A similar crucible has been fabricated in the GSC instrument shop.

The rapid methods used by Dr. Mercy are highly accurate and are 'rapid' in the sense that they are largely instrumental, not in the sense of the Shapiro-Brannock scheme. The laboratory completes 4 to 5 samples each week, reported only to one decimal place; if the rocks vary much in composition, work is slowed.

Department of Chemistry, King's College, Aberdeen

Dr. R.A. Chalmers has discontinued his studies of silicate microanalysis in favour of inorganic chemistry studies.

He mentioned a recent study yet unpublished which investigates the determination of Si by the silicomolybdate heteropoly complex. The beta-form is stabilized with acetone and a larger aliquot than that used in the Mo-blue procedure can be taken. The procedure is simple and a 10-fold variation in the acid concentration is permissible. It is possible to do As and Ge by this procedure also.

Macaulay Institute for Soil Research, Aberdeen

The bulk of the analytical work is by emission spectrography, following chemical separation and preconcentration, and this will be discussed in a later section. Flame photometric methods are used for Na, Ca and K; both instruments are home-made and use the Lundegaard flame system. Baffles are inserted in the spray chamber to eliminate large droplets and the acetylene moistened before entering the spray chamber, in order to eliminate certain undesirable effects.

As mentioned previously, rock analyses are made in a small laboratory belonging to the Department of Pedology, by a mixture of classical and rapid methods. Ca and Mg are done by EDTA titration, using an EEL titrator.

Department of Geology, University of Oxford

A small laboratory is used by students who require rock analyses. The methods are those of E.A. Vincent (Methods in Geochemistry, 1960, A.A. Smales and L.R. Wager; Interscience Publishers, New York, pp. 33-80) and J.P. Riley.

Dr. N. Snelling has a simple procedure for the determination of K in mica; a 25 mg sample is dissolved in HF + H_2SO_4 and diluted to give approximately 8 ppm K, then run with an EEL flame photometer hooked up with a recorder to measure the peak height. Standard solutions of pure K salt are used to bracket the unknown; it was found that Na, Al, Fe, Ca and Mg had no effect (low temperature flame?), so simple standard solutions are used. Precision is said to be ± 2 per cent. Comparison of values obtained in this manner and those obtained by Riley's method, which separates interfering elements by ion exchange, suggest that a matrix effect occurs in solutions containing more than 200 ppm of rock.

Notes were made on a study of the radiochemical determination of Na and K in silicate rocks and minerals, in particular in ultrabasic rocks, by Miss Barbara Peel (unpublished thesis, University of Oxford, 77 pp. 1962) working with Dr. Vincent. Emphasis is placed upon the need to avoid contamination of the ultrabasic sample by air, instruments and hands. Four weighing forms were investigated - chloroplatinate, cobaltinitrite, perchlorate and tetraphenylboron - and the chloroplatinate found to be the best.

Department of Geology, University of Cambridge

Mr. Scoon prefers classical methods but has used some of the S-B rapid methods, such as the Mo-blue method for SiO_2. He prefers to bring down the Mn by the addition of bromine after precipitation of the R_2O_3 group; an aliquot of the pyrosulphate fusion of the ignited precipitate is used for the determination of Mn which is reported as Mn_3O_4. Mr. Scoon has tried the vanadate method of A.D. Wilson for FeO and he finds that the longer the sample solution remains in the plastic bottle, the lower are the final results; Fe is adsorbed on the walls of the container and later washing with HCl will show the characteristic yellow colour of the ferric complex. Total H_2O is done by the Penfield method but using a flux (Na_3WO_4) only when the sample is known to be refractory; no correction is made for F or CO_2!

Royal School of Mines, Imperial College, London

Both classical and 'rapid' (colorimetric but with high precision and accuracy) methods are used by Dr. Carmichael. SiO_2 is done by the Jeffery-Wilson method. One sample solution is used for R_2O_3, CaO and MgO, a second one for Na_2O, K_2O, MnO, TiO_2, P_2O_5 and total Fe. Ca and Mg are finished gravimetrically except when less than 2 per cent, when an EDTA titration (calcein, EBT) is used following an R_2O_3 separation with hexamine. Total iron is determined colorimetrically by thioglycollic acid if the sample contains less than 15 per cent Fe_2O_3, by titration with $Cr_2O_7^{=}$ after $SnCl_2$ reduction if more than this. FeO is done by Wilson's method which Dr. Carmichael likes and finds applicable to almost any range of Fe^{++}. He obtains his Al_2O_3 by difference in rock analysis but determines it directly, with aluminon, on minerals.

Overseas Geological Surveys, London

In general the chemical laboratories use the classical methods of rock analysis, with some addition of the methods of J.P. Riley. Ca is done gravimetrically as the oxalate (weighed as CaO) as usual but Mg is done titrimetrically with EDTA and EBT on an aliquot of the calcium filtrate. The alkalies, P_2O_5 and MnO are done on the HF - $HClO_4$ solution of a separate sample; the aliquot for the alkalies is simply diluted to volume and run, as is, on the EEL flame photometer. Total water is done with a simplified form of the closed-circulation system of Jeffery and Wilson.

Laboratory of the Government Chemist, London

Mr. Wilson is not doing analytical work pending a move to new laboratories. He discussed various methods including the microdetermination of $CO_3^{=}$ (in small beaker, CO_2 released by H_2SO_4 precipitates $BaCO_3$ which is filtered on a filter stick and weighed), the determination of S (add V_2O_5 to acid mixture when organic matter or pyrite is present, and add Ca if absent in order to tie up $SO_4^{=}$ in preference to Na), the microdetermination of Al with pyrocatechol violet and the use of sodium (not ammonium) succinate to precipitate the R_2O_3 groups.

Geological Survey and Museum, London

The methods used here are largely classical with some newer procedures, chiefly those worked out by Jeffery and Wilson (both of whom at one time worked in these laboratories) such as those for SiO_2 and total H_2O. Mr. Sergeant is investigating a procedure that will permit the titration of Ca and Mg separately in the same solution; Ca is titrated with EDTA to a blue-grey end-point with methylthymol blue (pH 12), the solution is buffered to pH 10 and the titration is repeated to a green-grey end-point. The R_2O_3 group is removed by Wilson's succinate procedure and residual interferences are complexed with triethanolamine and ascorbic acid. Mn will interfere. Ca and Mg in minerals are determined also with the Unicam SP 900 flame photometer.

Warren Spring Laboratory, Stevenage

The samples analyzed by the chemical laboratories are very varied (including ores and rocks) and chiefly involve the determination of specific elements; over 8,000 determinations on 4,000 samples were made in 1961 and the most popular elements are Pb, Zn, Cu, Co and Ni. Dr. Jeffery is very much interested in rock analysis (formerly with Geological Survey of Great Britain) and has 2 reference samples (Shetland Granite (R 117) and Carbonatite (R 93)) which he will make available to laboratories interested in providing analytical data on them. He is presently investigating a gas chromatographic method for the measurement of gases in rocks; CO_2 and H_2O are readily determined and he has also isolated and measured traces of hydrocarbons.

Fluorine is separated by the Berzelius distillation method with perchloric acid; instead of bubbling steam through the acid mixture he simply drips water in from a separatory funnel. For large amounts of fluorine he prefers the lead chlorofluoride method.

III Emission Spectrographic Methods

Geological Survey of Sweden, Stockholm

The Geochemical Institute, with 5 spectrographs, has an extensive program of spectrographic analysis in operation. Rock samples are analyzed for Si, Al, Ca, Mg, Fe, Mn, Ti, Ba and Sr by a spark technique, using a ¼-inch diameter pellet prepared from a 1:1 mixture of graphite and sample (sample fused at 1,000°C with B_2O_3, Li_2CO_3 and internal standards Co_2O_3 and BeO, bead ground in agate); a Zeiss Q-24 quartz spectrograph is used (3Å displacement at 2,000Å) and the pellet is sparked (approximately 1 minute) at 13-15 Kv. Dr. Manheim uses a Hg vapour lamp to improve ionization at the electrode, and uses a thyraton controller in a Hilger source to control the spark, although he prefers the Feussner rotating control. An optical 3-step filter is used (4, 20, 100). Spectra are recorded on Ilford plates and standards, both synthetic and natural, are put through the same process. The goal is ± 3 per cent accuracy per element and 6 or 7 standards are put on each plate. This method is used for all types of samples except sulphides; a study of iron ores is in progress.

A part of the ground, fused sample is dissolved in nitric acid and Na and K are determined with a Beckman B spectrophotometer and flame attachment.

For geochemical prospecting samples a solution technique is employed to determine Cu, Pb and Zn. The sample is digested warm overnight with 2N nitric acid in a loosely-stoppered test-tube and the supernatant liquid used. A vacuum electrode equipped with a nylon or Teflon cup holds the liquid for a 30-second sparking. A study is being made of shales in Northern Sweden that are high in Cu but not in Zn and it is hoped to eliminate false anomalies by this procedure.

Dr. Manheim plans to use the solution technique for the analysis of sulphides (As and Sb to come), with S and Fe being done in the chemical laboratories.

A medium Hilger quartz spectrograph, having an 8-channel direct-reader attachment, is housed in an air-conditioned room, and has been used recently for both the rock analysis and the geochemical prospecting work.

Dr. Manheim tried the tape machine but thinks that the solution technique is better for a small laboratory lacking the facilities for the very important, preliminary sample isoformation.

Bolidens Gruvaktiebolag, Skelleftehamm

The outstanding feature in the Physical Methods section is the A.R.L. Tape Machine and Quantometer. Full details of the method have been published by Danielsson and Sundkvist. The room is air-conditioned and warmed air is circulated through the Quantometer which has 30 integrators using 42 lines for Cd, Co, Zn, Pb, Mn, Cu, In, Ag, Fe, As, Sb, Bi, Sr, Ge, Cr, Hg, Si, Be, Mg, Ca, Tl, Ba, Ti, K, B, Mo, Cs, Ni, Na, Al, Sn and Li. HF solutions of samples, and synthetic standards, are analyzed with a Beckman flame attachment connected to the ARL recorder by a Zeiss 3-glass prism; no correction is made for interelement effect.

For rock analysis the sample is fused at 1,350°C in a Pt crucible (graphite was used but it melted(?) and spoiled the sample bead); Palau crucibles are also being tried. The bead is ground in the Schwingmühle using tungsten carbide mills, and then is fed on to the tape.

Cu, Pb and Zn are determined by precipitating them from solution, after addition of Cd as an internal standard, by saturated H_2S water, filtering them on a Buchner funnel, covering the precipitate with a filter paper, washing it with acetone and air-drying. The sulphides are shaken in a mixer mill and are ready to be fed on to the tape.

Concentration of minor and trace elements is done by ion exchange. Cd perchlorate (internal standard) and K_2CO_3 (special buffer) are added to the solution of the sample followed by an anion exchange resin (20#, by volume) and a cation exchange resin (100#, by weight). The mixture is boiled, allowed to digest, macerated filter paper is added and the whole is filtered through a double polyethylene filter funnel by suction. The coarser anion exchange resin is removed first (it is regenerated for further use) while the lower funnel retains the finer cation exchange resin. A filter paper is placed on top of the resin, washed with acetone, dried and ground in the Schwingmühle for analysis by the tape method.

About 20 complete analyses can be finished each day by three people. The results are very acceptable for rocks; loss on ignition is used to obtain a reasonable total, ignoring oxidation of ferrous iron.

Mr. Sundkvist reported that In is a good internal standard for Al in different matrices.

Geological Survey of Finland

A Jarrell-Ash model 7101, 3.4 metre plane grating spectrograph, with a JACO console densitometer and varisource are used for trace analysis of peat ash and glacial till by DC-arc methods. The samples are mixed with carbon powder only, and cathode excitation is used. It is also used to finish analyses on material supplied by the chemical laboratories, such as rare earth concentrates, and platinum metals (in bead). A Beckman flame attachment is available for the determination of alkalies.

University of Leeds

Miss Rooke has a Hilger large quartz and glass instrument, with a Hilger power source. A Judd-Lewis comparator and a Hilger non-recording densitometer are used. Neither of the laboratories are air-conditioned.

The electrode is a ¼-inch rod machined to 1/8-inch, with a 1/16-inch hole drilled to 1/4-inch depth; the sample and carbon powder (1:1) plus Pd and In are mixed in a small agate mortar and loaded through a paper funnel. The electrode is filled to the top and packed firmly; no weighing of the charge is done. Samples are run in duplicate, by anode excitation, using a first and second burn arrangement to take advantage of a plateau of emission that is related to the concentration level of the elements. Miss Rooke is studying the effect of matrix on selected line pairs, using 10 samples ranging from very acid to very basic. She has concluded that certain line pairs are better than others and presents quantitative data to support her views.

British Ceramic Research Association Laboratories

In an effort to be able to handle a large number of samples requiring a wide range of element determination in a wide range of materials the B.C.R.A. Laboratories have installed a Hilger and Watts 3 metre grating direct reading spectrograph (Polychromator) with typewriter read-out (Polyprint) in an air-conditioned room with the regular Hilger quartz spectrograph. The 30 channels of the Polychromator are being set up for Li, Co, Zr, V, Mg, Ca, Ba, Pb, Sr, Cr, Sn, Al, K, Zn, Ti, Na, Cu, Si, Fe, Mn and B; Co and Sr are used as internal standards. The high voltage spark technique is to be used, with pelletizing of the sample as received or after fusion with $Li_2B_4O_7$. About 50 elements are determined semi-quantitatively with the Hilger spectrograph, using cathode layer excitation; the sample is mixed with carbon powder for the determination of the alkalies, with KCl and C for most other elements except Zn, P and F (as SrF_2) for which $CaCO_3$ and C are used. Standards are either synthetics (a base of $Al_2O_3 \cdot 2SiO_2$ plus pure reagents) or analyzed silicates.

Pilkington Brothers Limited, St. Helens and Lathom

Mr. Ward has 4 Hilger large quartz spectrographs and a Hilger 3 metre grating direct reading 30-channel spectrograph (Polychromator) which he has modified for DC-arc methods. He uses the same methods on the direct-reader that are used on the other instruments. His laboratories are a model of organization, with each spectrograph assigned for certain work; to find the proper method to use an analyst consults a list of methods for all types of samples which refers him to the proper file containing a complete description of the method and the appropriate working curves, as well as the necessary standards already prepared for use. Descriptions of the methods in use, and of the modifications made to the Polychromator, have been published. As mentioned previously, chemical and spectrographic methods are used to provide rapid analyses on a variety of samples; on sand they determine Fe, Al, Ti, Cr, Zr, Mn, Ca, Mg, Na, K (Si by difference), on sheet and plate glass Fe, Al, Ti, Cr, Zr, Ca, Mg, Ba and V, on opal glass Ca, Fe, Al, Mg, Ba, Zn, Mn, Mo, Zr and Ti, and on Crookes glasses Al, Zn, Ca, B, Sb, Pb, rare earths. Fifteen different classes of material are handled in this way. The internal standards used are Rb for Na and K, Sr for Ca and Co for others.

University of Liverpool

Dr. Riley uses an early Hilger quartz spectrograph which takes three 10-inch plates to cover the range 2,100 - 9,000Å. Cathode excitation is used for samples which are generally mixed only with C powder, occasionally with $CaCO_3$ also. The standards are matched to the matrix of the unknown. The work is mainly qualitative and includes the checking of chemically determined material, e.g. Cd in sea-water is extracted with dithizone and measured spectrophotometrically, then the extract is evaporated to dryness and the residue examined spectrographically for the presence of Cd and the absence of Tl. This method for Cd has been extended to sea organisms and sediments and the lower limit is 20 ppb (± 3 per cent) Cd.

Macaulay Institute for Soil Research, Aberdeen

Details of the methods used have been published and few comments are necessary here on these. The equipment and facilities have been described in previous sections.

Dr. Mitchell pointed out that useful life of the counter electrode used for porous cup work (e.g. with the 2-channel Hilger used for determination of Mg in plant material) can be increased five times by cleaning the used electrode in acid, drying it at 250°C and resharpening it. Separate working curves are used for the acetic acid or ammonium acetate extracts of plant material; readings are made with a galvanometer and as little as 0.1 ppm can be detected accurately.

The preconcentration procedures are done simultaneously in the same beaker; the curdy precipitate ashes well in a porcelain crucible and the ash is easily removed. The ash is weighed, allowed to come to equilibrium in air and 5 milligrams are then fused with Na_2CO_3 and the total Fe determined colorimetrically with sodium salicylate. The Fe thus determined is used as the internal standard for the analysis of the ash (K_2SO_4, with Li and Cr, as buffer) by the rotating disc method; the elements determined are Zn, Si, B, P, Cr, Fe, Mg, Mn, Al, Li, Cu, ranging from 0.1 ppm B to 0.1 per cent Si or Al. The 12-channel direct reader is used for this work.

Royal School of Mines, Imperial College, London

Mr. R. Berlin, of the Pure Geochemistry Section, uses the methods of Louis Ahrens, with whom he studied in Capetown. Cathode excitation is largely used, with Pd and Fe as internal standards and C powder only (no buffer). Electrodes are filled by inverted packing, and synthetic standards are preferably prepared from appropriate minerals whenever possible. No air-conditioning used in laboratory or dark room but Mr. Berlin arcs and develops an accumulation of samples at one time and thus avoids the problem of reproducing environmental conditions. He also prefers the Micro-Mill (agate vials with agate balls, enclosed in steel capsules, Agate and General Stonecutters, 25 Hatton Garden, London, E.C.1) to the Wig-L-Bug mixer because the latter tends to cause separation in powders.

He does not believe that it is possible to transpose a method from one laboratory to another without making changes in the method. Fluorine has been done as CaF_2, using Kodak P 1600 plates, when it is greater than 200 ppm.

A special Mannkopf spectrograph, with 2 prisms (60° and 30°), having a dispersion of 3Å/mm at 4,000Å, is used for determination of the rare earths, except holmium. The rare earths are first separated as fluorides, then as oxalates using homogeneous precipitation with methyl oxalate (Th is included); 20 milligrams of the ignited residue is mixed with C powder and analyzed.

Miss Lewis, in the Applied Geochemistry Section, is using methods originally set up by Dr. J.A.C. Fortescue of the Survey. The sample is mixed with Li_2CO_3 and C powder, with Ge as internal standard, and then analyzed with a large Hilger quartz spectrograph, using anode excitation and a partial 20-second burn. Elements determined are Pb, Sn, Ga, Bi, V, Cu, Ti, Ag, Ni, Zr, Co, Mn and Cr, occasionally Si, Ca, Mg, Al, alkalies also; 13-14 samples are put on each plate and 40 samples, for 15 elements, are run daily, including a standard series. There are 12 standard series and 4 series are done daily so that the whole series is covered in 3 days (one series on each plate). Calculations are made to see if the runs are acceptable (within ± 40 per cent). Samples are prepared, loaded and 1 plate is shot in morning, the other 3 plates in the afternoon; they are dried overnight and read (Hilger version of JACO console used as comparator only).

Warren Spring Laboratory, Stevenage

The use of 3 spectrographs with one arc-stand has been mentioned previously. The work is mostly quantitative but includes some qualitative analysis for mineral identification; the samples include air pollution specimens and material from the beryllium analysis laboratory.

General Electric Laboratories, Wembley

One large and 2 medium Hilger quartz spectrographs are used for a variety of analyses, mostly on metals. A 13-channel direct-reading attachment to one of the medium Hilger instruments is used for the determination of Be in air, using the porous cup technique with Au as internal standard; In, Al and Ga in metals are also done this way.

Overseas Geological Surveys, London

The samples are very varied and the work is chiefly semiquantitative at present. The standards are selected to resemble the sample as closely as possible, e.g., for Nb use pyrochlore as the basic material for dilution; DC arc only, anode excitation, total burn. A buffer of $CaCO_3$ plus graphite in 1:1 ratio is used for Nb work (with HfO_2 as internal standard). Cr, Ni, Pb and Cu are done semiquantitatively (Li_2CO_3 + C, 1:1) as a preliminary geochemical examination prior to a chemical analysis.

Geological Survey and Museum, London

The spectrographic laboratory uses the methods of Harvey and Murray (The Analyst 1958, p. 136), employing a large quartz and glass Hilger instrument, with DC arc and anode excitation. Standards are made by addition of Spec-Pure chemicals to a base of calcium silicate and quartz; an attempt was made to use a felsite as base but the spectrum proved to be too complex. Some use has been made of the spark technique but it was not found to have any advantage over the DC arc.

IV. X-ray Fluorescence Spectroscopy

None of the laboratories visited were using, nor planning to use, an X-ray fluorescent spectroscopic method similar to that now in use in the Survey for the major element analysis of rocks but some use is being made of the technique.

The Geological Institute in Oslo are using a Philips Vacuum X-ray Fluorescence spectrometer for qualitative work but have done V in magnetites ($>$0.1 per cent) and some work on rare earths.

At Bolidens, the Rönnskar laboratories use this technique for determining Cr, Cu, Fe and As in wood preservative; the Laisvall laboratory uses it for the automatic analysis of mill tailings (Pb and Zn) and a new 4-channel Philips X-ray Fluorescence spectrometer is to be installed.

Mr. Hoffren, at the Geological Survey of Finland, is using a Philips vacuum instrument for the semiquantitative determination of Cu, Ni, Zn and Pb in ore samples without fusion of the sample. All available crystals have been obtained, including topaz for the rare earths.

At the University of Leeds a Siemens-Halske Vacuum X-ray Spectrometer is being used for rock analysis (major elements, except Na and K). Dr. Hornung mixes the sample with cellulose (1:1) and pelletizes it in a 30-ton press. No internal standard is used; tried V but found it not satisfactory. Working curves are prepared for each rock type (e.g. basalts, granites) based on chemically-analyzed material. Dr. Hornung has found that control of the voltage on the gas flow counter is very critical; a 20V variation can eliminate an element.

Dr. Snelling at Oxford uses a simple method for total Sr and Rb in granites; the sample (200-400 mesh) is pressed into a 1-inch diameter pellet and analyzed with a Philips instrument, using chemically analyzed (isotopic dilution) samples as standards. The method is applicable to samples having up to 600 ppm Rb and 250 ppm Sr, with an accuracy of ± 20 per cent. Dr. Hamilton has applied the method to 100 mg samples of feldspar and biotite also, as well as rock samples available in only small quantity, by using a boric acid backing; matrix effects are eliminated by the use of similar material as standards. For a quick method, Dr. Hamilton dissolves the sample in HF - $HClO_4$, evaporates to near dryness and extracts the Sr + Fe with ethylacetate, leaving the Rb with the Al as a sludge;

the Sr is separated from the Fe by ion exchange, the K and Rb perchlorates (left after treating sludge with H_2O) are converted to sulphates.

A new 12 channel Philips Vacuum X-ray spectrometer was just being installed at Cambridge. The sample will be mixed with starch and pelletized and they expect to cover the whole range of major elements in rocks.

An A.R.L. PXQ X-ray Fluorescence spectrometer is being used for analysis of rocks and minerals at the Royal School of Mines. A 2-gram sample of rock (100#) is mixed with cellulose and pelletized; mineral powders are backed with cellulose.

A single-channel Philips Vacuum X-ray Fluorescence spectrometer, at the Warren Spring Laboratory, is used chiefly for ore analysis. It is possible to get Mg (poor) and Al (better), but determinations are made usually of Nb, Sn and rare earths. Samples are run either as a powder, or as a pellet (with and without cellulose).

Mr. Hunt, at the Overseas Geological Surveys, has a single channel Philips instrument (air path) which is used mainly for qualitative work. The sample is fused with borax (800-900°C) and poured on to a hot (400°C) Al plate to give a button with one flat side; it must be allowed to cool slowly overnight.

Dr. Zussman, at Manchester, also used the Claisse borax fusion technique but preferred a mechanical mixture with borax instead. He used the method for the determination of Ni and Fe in serpentine, and of Mo in scheelite.

V. Infrared Spectroscopy

Dr. Farmer, at the Macaulay Institute, Aberdeen, was the only one encountered who was using infrared spectroscopy for mineralogical studies. He is interested in clays and does semiquantitative work (KBr disc) with IR; X-ray diffraction picks out the crystalline forms but the IR shows more, such as the amorphous constituents (silica, allophane, hydroxides). He finds IR useful for certain work as a complement to other techniques, such as X-ray, TGA and DTA.

The technique has been used to show difference between two calc-silicate minerals (one contained zeolitic water) and it can detect 1 per cent of carbonate in a silicate, in any form. For mineralogical work a good instrument is necessary (most silicates tend to have absorption bands in the same region), with a range of 2-25 microns and with a sensitivity of 1 wave number in the OH^- region. Better results are obtained with ultraviolet absorption spectroscopy in the OH^- region.

OTHER RELATED ANALYSIS

I. Geochemical Analysis

A brief mention of the geochemical work done at the Geological Survey of Finland has been made in the previous section. The only institution visited where an extensive program of geochemical prospecting analysis by chemical methods is in progress is the Royal School of Mines, London. Here the Applied Geochemistry Group use standard geochemical methods, employing a visual standard series, for a number of elements including Co, Mo, Pb, Sn, As, Be, Cu, Ni, Zn, C, Cr, Mn, W, K, Fe, Nb and U in soils, rocks and sediments, and Cl^-, $CO_3^{\equiv}$, $HCO_3^{\equiv}$, $SO_4^{=}$ and U in natural waters. Mr. Stanton also uses a Technicon Auto-Analyzer (unfortunately not working) for the routine analysis of Zn (250 samples/day); a 0.5N acid solution of the bisulphate fusion or acid leach of the sample is used, and the whole procedure, from the taking of an aliquot to the final recorder trace, takes about 15 minutes to complete. A smaller one is also available which was used both for testing methods for manual use, and for the determination of Mo, Pb and Ni.

II. Special Analysis

This will include material which cannot be accommodated in the other sections.

Dr. Filby, at the Mineralogisk-Geologisk Museum in Oslo, is studying the coprecipitation of Zn and Fe as sulphides, with Fe^{55} and Fe^{59} as tracers, and

$BaSO_4$ in the presence of Pb, Sr, Ca, Rb, Cs and Ag, the latter present as tracers; he hopes to correlate the distribution coefficient of each element with ionic size and the kinetics of the precipitation. He is also doing bomb work, particularly a study of the incorporation of Pb into sulphate lattices under varying conditions.

Dr. Hamilton, at Oxford, has used various means of reducing the level of contamination in his laboratory for Pb extraction and Sr-Rb separation. Reconstruction is going on and the problem is considerable. A triple filter (viscosine-covered linen mesh, viscosine-covered interlocking metal cones and a fibreglass sheet) in the door is the only entrance for the air drawn into the laboratory by the fume hoods, themselves fitted with similar filters. Mats of the sticky linen mesh are placed at the entrance to remove dirt from shoes, and the windows are covered with a single layer of this material. Hood space is reduced to a minimum by the use of an internal polyethylene hood and a similar curtain protects the decomposition vessels from a direct current of air. Table taps are black Formica which shows dust more readily. In the new laboratory the hoods will have an 'air curtain', ceilings will be low and double doors will be used. Air will be drawn from the basement through a series of filters.

Dr. Hamilton advised that, if Pt dishes are used for sample decomposition, the Pt dish must be made from new ('juvenile') rather than from reclaimed Pt. Dithizone extraction works well with small samples (relatively high Pb) but for larger samples the precipitation of Al spoils the extraction; the ion exchange method is better in the latter case but 10 to 20 per cent $HClO_4$ must be used as the eluting agent if good yields are to be obtained.

In the Pure Geochemistry Section at the Royal School of Mines, London, Dr. Butler is using neutron activation methods to determine a number of trace elements such as Cs, Rb, Tl and Cd.

III. The Analysis of Meteorites

A conference on analytical methods for meteorites was held at the British Museum (Natural History) on the 5th and 6th of September, with twenty participants from Canada, Finland, France, Great Britain and the U.S.A. Mme. Dyakanova (U.S.S.R.) was unable to attend but sent a summary of methods in use by the Committee on Meteorites of the Academy of Sciences of the U.S.S.R., on behalf of herself and Professor E.L. Krinov. A list of participants is given in Appendix III. There were four sessions, all of which took the form of a round-table discussion, and all proved to be highly interesting, informative and congenial. The conference was the first of its kind to be held and it was noted that just over one hundred years ago M.H.N. Storey-Maskelyne began his study of meteorites (1861).

The first session (Chairman - Dr. M.H. Hey) dealt with the selection and preparation of material, and the determination of the principal constituents of irons (Fe, Ni, Co, P and S). All participants agreed on the difficulty of obtaining a truly representative sample of an iron meteorite; methods varied from the use of shavings taken with a milling cutter to thin slabs which are used without reduction to fragments, but it is better to sample inclusions separately and a metallographic study of the surface is a necessary first step. Discussion centred on the use of various solution techniques and magnetic separation methods for separating the silicate, metal, phosphide and sulphide phases in stones and stoney irons, and the chlorination procedure used at the British Museum is said to give excellent results. Dr. Wiik (Finland) obtains excellent results with a boiling solution of mercuric chloride (Friedheim's reagent, mercuric ammonium chloride, has no particular advantage) for the total Fe of the metal phase. For all solvent attacks it is desirable that the meteorite be finely ground; chilling in liquid air helps by making the metal brittle and Dr. Mason has used ultrasonics to break up composite grains (in this case crushed, not ground, material is better). Magnetic separation is not favoured (it is a standard procedure with Russian analysts) because it produces two concentrates, the one of metal plus a little silicate and the other of silicate plus a little metal, and certain unjustifiable assumptions must be made about the distribution of constituents in these concentrates. The amount of material necessary to obtain a representative sample depends on the homogeneity of the meteorite and elaborate sampling procedures may be needed; Russian and British analysts generally prefer 15 to 20 grams of a normal meteorite for analysis but Drs. Mason and Wiik prefer 40 grams. There was general agreement that analysis of the separated components is preferable to an analysis of the whole meteorite. A suggestion that a large sample of an iron meteorite be prepared and distributed for comparative study was not favoured; except for certain trace elements, a standard steel sample will serve as well. No radically new methods are in use or were proposed for the determination of the major constituents in iron meteorites; the methods used in the various laboratories range in nature from gravimetric to neutron activation. Much discussion was spent on the determination of carbon, which is rarely determined and then often unreliably. Dr. Axon (Manchester) pleaded for C determinations, even if only to show that it is not present. The methods available for the determination of microgram amounts of C (and CO_2 in general) were discussed; the uncertainties of absorption tube procedures were recognized and the possibilities of a micro-gasometric method which employs the freezing out of CO_2 (good for study of phases) were enlarged upon.

Dr. E.P. Henderson (Smithsonian) was Chairman of the second session which dealt with the determination of the principal constituents of stones, with particular reference to Al, Ca, Na and K. Classical methods, with flame photometric determination of the alkalies, are in general use. Dr. Maynes (Caltech) prefers to leach the sample in HCl and then fuse the insoluble (metal- and sulphide-free) residue with Na_2CO_3; Dr. Wiik fuses the whole sample with Na_2CO_3 at 1,100°C in an electric furnace having adequate access of air, and finds no contamination of the Pt crucible by iron in the sample. Aluminium is the constituent most difficult to determine because of its low amount and its determination by difference was generally condemned. A comparative study of the various procedures suggested would appear to be badly needed. Mme. Dyakanova wrote that the Russians are working on the direct determination of Al by a complexometric method using 3, 3'-dimethylnaphthidine or benzidine as indicators. Much discussion, with few results, occurred on the subject of determination of the state of oxidation of iron in the non-metallic phases; the isolation of magnetite from some stony meteorites indicates that the assumption that all the iron is ferrous no longer holds. The chlorination method holds much promise in this regard. No use is being made of X-ray fluorescence spectroscopy for the analysis of stones because no distinction can be made between the constituents of the various phases if the uncrushed sample is used, but Dr. Maynes is planning to do some work on this.

The third session considered the determination of trace elements, with Dr. E.B. Sandell as Chairman. Much emphasis was placed upon danger of contamination from groundwater (if specimen was in the ground for any length of time), cutting and polishing (abrasion, tap water, soluble oils), sulphur printing, or etch solutions. Dr. Axon mentioned that schreibersite generally has a cracked appearance and invasion of the specimen is thus easily achieved. Ideally the sample for trace element study should be <u>broken</u> from the interior and the case history of the mass should be well-known.

Neutron activation methods were strongly put forward (Mr. Smales) but Dr. Sandell pointed out that, where facilities are not available for such work, colorimetric methods compare well with neutron activation in sensitivity, precision and convenience. In this connection discussion took place on the general unreliability of organic reagents and the need to test each new batch.

Mr. Mapper (Harwell) described the neutron activation methods used for determining Ge, As, Sb, Cu, Cr, Mo, Ag, In, Zn and Pd (these elements were selected on basis of interest only); Ga is done colorimetrically. Mr. Smales emphasized that much chemical separation work is necessary in neutron activation analysis and also pointed out possible errors in the method. Dr. Webster (Harwell) discussed an isotopic dilution method for the determination of Rb and Cs in stony meteorites, the results of which compared well with those obtained by neutron activation. He found that the Cs content of chondrites varies by a factor of 50 (0.2 - 0.004 ppm), Rb by a factor of 3 (1 - 3 ppm). Cold water leaching of the sample will remove much of the Cs.

The final session, with Dr. Brian Mason as Chairman, included a discussion of the use of the Castaing microanalyzer for meteorite studies by Dr. Sandréa (Paris). In a study of the Chassigny iron he was able to trace variations in the S, Cr and Fe content of spherules of troilite and chromite. A similar instrument for meteorite study is being installed at the Max Planck Institute in Heidelberg.

In the general discussion which followed many subjects were raised. Mr. Smales spoke of the potential usefulness of spark source mass spectrometry; Dr. Henderson recommended a general study of a large meteorite and offered to prepare a reference sample (he has a pallasite, octahedrite and some stones available for this purpose); Dr. Wiik warned against the practice of describing a meteorite just because it is available, since this leads to many descriptions of similar specimens and rare specimens go unstudied (supported by Dr. Mason); Dr. Moore (Nininger) suggested a listing of all good thin sections held in collections, but Dr. Henderson pointed out that he always hesitates to make thin sections for fear of contamination; Dr. Wahl (Finland) urged that studies should be made on the large groups of meteorites that are available (so that material is available to others) rather than on single specimens. Much interest was expressed in the Abee meteorite and several requests for specimens were forwarded to Dr. Dawson.

IV. The Spectrochemical Analysis of Feldspars

Dr. W.E. Fraser, Marischal College, Aberdeen, has devised a procedure for the rapid spectrochemical analysis of feldspars which gives results for Na, Ca and K as per cent albite, anorthite and orthoclase respectively. The sample, unmixed with any buffer or graphite, is burned (cathode excitation) and visual estimation of the intensities of selected lines for Na, Ca and K are made. The estimated intensities are substituted in two empirical functions and from the algebraic values of these two functions the composition of the sample is obtained from a chart. The chart was prepared from results obtained on chemically-analyzed material. A less rapid and more accurate procedure involves the flame photometric analysis of a HF - $HClO_4$ solution of the sample.

Dr. Fraser's method is deliberately empirical for, as he says, "the working rule throughout the investigation has been to circumvent, rather than to investigate, difficulties" and it was hoped that the method could be applied by relatively unskilled personnel. At his suggestion the writer took Muskox feldspar samples for testing the method but unfortunately Dr. Fraser's data cover only the microcline-albite-labradorite field and the G.S.C. specimens were all in the anorthite range. Dr. Fraser, however, has made good use of these analyzed samples to extend the coverage of his data. Dr. Pouliot is interested in the potential usefulness of this method and he is to supply Dr. Fraser with more analyzed feldspars. The paper giving all details of the method will be published shortly.

APPENDIX I

Summer School in Analytical Chemistry

The fifth Summer School in Analytical Chemistry, sponsored jointly by the Royal Institute of Chemistry and The Society for Analytical Chemistry, was held September 9 - 15, 1962, at the Manchester College of Science and Technology. This was the first time the Summer School was held in Manchester, and the first time that it was open to non-members of the participating societies; it was also the largest Summer School yet held, with 360 enrolled of whom 36 were overseas visitors chiefly from countries in Western Europe. The four courses offered were: I. Physical Methods of Organic Chemistry; II. Recent Developments in Inorganic Analysis; III. Determination of Toxic Substances in Air and Effluents; IV. Newer Instrumental Methods. Two lectures were given in each course in the morning; four sections of practical work were also organized for the afternoon and each member attended one section each afternoon and then spent the final afternoon in a section of his choice. The writer applied for enrolment in course IV but because of the popularity of this course (II and IV were both oversubscribed) was not able to gain a place in the laboratory work.

The lectures attended by the writer during the week are as follows:

Monday: Atomic Absorption Spectrophotometry. Mr. J.A.F. Gidley. Recent Developments in Methods for the Analysis of Ceramic Materials.
Mr. H. Bennett.

Tuesday: Ultrasonic Techniques in Chemistry and Applications.
Dr. W.H. Lee.
Electrometric Methods of Analysis. Mr. E. Bishop.

Wednesday: Spark-source Mass Spectrometry. Mr. R.D. Craig.
Spectrophotometry of Emulsions and Suspensions. Dr. J.D.S. Goulden.

Thursday: Infrared Spectroscopy in Inorganic Chemistry. Prof. J. Lewis.
Thermogravimetric Analysis and Differential Thermal Analysis.
Dr. J.P. Redfern.

Friday: X-ray Fluorescence Analysis. Mr. J.B. Stansfield. Electron Spin Resonance. Dr. J.F. Gibson.

These lectures, apart from the one by Mr. Bennet (Monday), dealt with fundamental theory and touched only lightly on application and practical detail. The lectures on ultrasonics, spark-source mass spectrometry and DTA and TGA were most fruitful, in particular that by Dr. Lee on ultrasonics from which many applications of interest to us have suggested themselves. The lectures on atomic absorption, electrometric methods and X-ray fluorescence analysis contained nothing new to the writer while that on electron spin resonance never succeeded in penetrating the writer's intelligence. It was hoped that Professor Lewis' lecture on I.R. spectroscopy would provide some information on the use of this technique in mineralogical studies but the lecturer had no experience in this aspect of the work. The lecture on the spectrophotometry of emulsions and suspensions contained nothing of particular interest to the writer.

An opportunity was provided for the writer to see and examine the very large amount of equipment that had been loaned by various firms for use in the practical sessions.

The inability of the writer to take part in the laboratory sessions much reduced the benefit to be derived from this Summer School, both from the opportunity to test various types of instruments and to discuss methods, techniques and instrumentation with others in the same course.

APPENDIX II

The Preparation of Meteorite Models.

At the request of Dr. K.R. Dawson, Geological Survey of Canada, the writer inquired into the methods used in the British Museum (Nat. History) for making casts of meteorites. Mr. A.G. Hayward generously devoted time and effort to demonstrate the method, and supplied the writer with information as to possible sources of supply for the material needed.

Mr. Hayward prefers to make a rubber mold of the surface as follows: the specimen is embedded in plasticene and on the exposed surface is brushed a coat of Latex (60 per cent, centrifuged, pre-vulcanized) which is then reinforced with layers of cotton batting and then dried with an IR lamp; 3 layers are similarly built up and a plaster backing is then made and allowed to dry; the plasticene is removed from the other half of the specimen and the above procedure is repeated on it; polyester resin with a 70 per cent metal filler is then poured into the rubber mold enclosed in the plaster (less than one-half volume) and the whole rotated slowly to form the cast; the cast is painted with oil paint in turpentine. If a plaster cast is desired, a longer time of rotation is needed and the cast should be given a coat of shellac before it is painted; plaster casts may be made solid if desired, a more certain procedure than that for hollow casts. Mr. Hayward tried silicone and polyvinylchloride molds and found them to be less satisfactory.

When shown a plaster and a plastic cast of a meteorite, the writer was able to distinguish the original only by its weight.

APPENDIX III

Analytical Methods for Meteorites Conference

September 5-6, 1962.

The British Museum (Nat. Hist.), Department of Mineralogy

Participants:

Dr. H.J. Axon,
Dept. of Metallurgy,
Univ. of Manchester.

Dr. M.H. Battey,
Dept. of Geology,
King's College,
Newcastle upon Tyne.

Mr. D.I. Bothwell,
British Museum (N.H.).

Dr. G.F. Claringbull,
British Museum (N.H.).

Mr. Roy S. Clark,
Smithsonian Institution,
Washington, D.C.

Mr. M.T. Frost,
Dept. of Geology,
Univ. of Manchester.

Dr. E.P. Henderson,
Smithsonian Institution,
Washington, D.C.

Dr. M.H. Hey,
British Museum (N.H.).

Mr. D. Mapper,
United Kingdom AEC,
Harwell.

Dr. Brian Mason,
American Museum,
New York.

Dr. J.A. Maxwell,
Geological Survey of Canada.

Dr. A.D. Maynes,
California Inst. of Technology,
Pasadena.

Dr. C.B. Moore,
Nininger Meteorite Collection,
Arizona State Univ.
Tempe.

Dr. E.B. Sandell,
Univ. of Minnesota,
Minneapolis.

Dr. A. Sandréa,
Musee d'Histoire Naturelle,
Paris, France.

Mr. A.A. Smales,
United Kingdom AEC,
Harwell.

Dr. W. Campbell-Smith,
British Museum (N.H.).

Prof. Walter Wahl,
Helsinki, Finland.

Dr. R.K. Webster,
United Kingdom AEC,
Harwell.

Dr. H.B. Wiik,
Geological Survey of Finland,
Otaniemi.