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CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

GEOLOGICAL SURVEY OF CANADA
TOPICAL REPORT NO. 128

REPORT ON VISITS TO INSTITUTIONS ENGAGED IN THE ANALYSIS OF ROCKS, MINERALS AND RELATED MATERIALS; NORWAY AND ENGLAND

AUGUST 23 – OCTOBER 4, 1966

18

J. A. MAXWELL

OTTAWA
1967

NOT TO BE QUOTED AS A PUBLICATION
FOR DEPARTMENTAL USE

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REPORT ON VISITS TO INSTITUTIONS ENGAGED IN THE ANALYSIS OF ROCKS, MINERALS AND RELATED MATERIALS; NORWAY AND ENGLAND, AUGUST 23 - OCTOBER 4, 1966

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Report on visits to institutions engaged in the analysis of rocks, minerals and related materials, in Norway and England, during the period August 23 – October 4, 1966.

Norway

1. Department of Chemistry, University of Oslo.
2. Mineralogical Institute, Oslo.
3. Chemistry Division, Dept. of Industry, Trondheim.
4. Geological Division, Dept. of Industry, Trondheim.

England

1. International Mineralogical Association Meeting, Cambridge.
2. Warren Spring Laboratory, Department of Industry, Stevenage.
3. Department of Geology, University College of Swansea, Wales.
4. British Ceramic Research Association Laboratories, Stoke-on-Trent.
5. Central Laboratory, Pilkington Bros. Ltd., St. Helens.
6. Department of Geology, University of Newcastle upon Tyne.
7. Department of Geology, University of Durham.
8. Geological Survey of Great Britain, London.
9. Overseas Geological Surveys, London.
10. Department of Mineralogy, British Museum (Natural History), London.

In 1962 the writer was privileged to make a tour of rock and mineral analysis laboratories in Norway, Sweden, Finland, England and Scotland (see Topical Report No. 75, 1963) to learn about methods, techniques and equipment in use in these laboratories, to meet analysts engaged in this work and to discuss problems both common and peculiar to our respective laboratories. The results of this visit have been very fruitful and the initial contacts have been maintained both by correspondence and by return visits of Scandinavian and British analysts to the Geological Survey.

In the succeeding four years there has been a notable increase in the application of instrumental methods to rock analysis, particularly that of X-ray fluorescence spectroscopy and, more recently, atomic absorption spectroscopy. It seemed worthwhile, therefore, to take advantage of a planned vacation trip to revisit certain laboratories, and to visit some new ones, in which these instrumental and other methods are being applied in order to obtain first-hand knowledge of their procedures and techniques. The Survey generously concurred in this proposal and the writer devoted sixteen working days to carrying it out.

A. Visits in Norway

1. Department of Chemistry, University of Oslo.

A return visit was made to the laboratory of Dr. F.J. Langmyhr, chiefly to discuss the reaction to his recently-published new scheme of silicate analysis ("A contribution to the analytical chemistry of silicate: a scheme of analysis for eleven main constituents based on decomposition by hydrofluoric acid". F.J. Langmyhr and P.R. Graff, Norges Geologiske Undersokelse No. 230, 128 pp., 1965) and to inquire about new work along these lines. The following pertinent observations arose out of the discussion:

- there has been much interest expressed in the new scheme but no trials of it have as yet been made apart from its use at the Geological Survey of Norway (see on). I think that it is overshadowed by the vast amount of work being done on instrumental methods and that it will eventually be found most useful for the analysis of minerals in limited sample supply. It does not require expensive bombs for the routine decompositions and the method for the determination of silica is particularly interesting. I was able to obtain more information about the decomposition vessels used, and to clarify some ambiguous statements in the scheme as published.

Other items of interest from Dr. Langmyhr were:

- Dr. Zdenek Sulcek (Chief Chemist, Central Geol. Institute, Praha 7, Kostelni 26, Czechslovakia) has written (with Drs. Dolezal and Povondra) a book on the decomposition of minerals.

- E. Merck AG, Darmstadt, Germany, are marketing a high-purity hydrofluoric acid (max. 0.005% SiF_4, 0.5 ppm Pb).

- a graduate student is studying the determination of Fe^{2+} and Fe^{3+} by orthophenanthroline (Dr. J.M. Moore and I investigated this unsuccessfully in 1960-62); he may try a system involving two colour-forming reagents.

- sintered porous Teflon discs (of varying porosity) inserted in Teflon crucibles for filtering HF solutions are available from

  Gachot,
  179, Ave de la Division Leclerc,
  Enghien (Siene et Oise),
  France.

2. Mineralogical Institute, Oslo.

This was also a return visit but I was able this time to meet Dr. Bruun who does the classical silicate analyses, assisted by Mrs. B. Jensen. They are using atomic absorption spectroscopy (AAS) for Mg over a wide concentration range and are to use it for trace element determinations in the near future. The instrument is a Beckman, with Westinghouse (Hilger) lamps. Together with Mr. A.O. Brunfelt they are using an instrumental neutron activation and gamma-ray spectrometry procedure to determine rare earths, P, Co, Zn, Ga, Ag, Cr, Cd and Se in rocks (Geochimica et Cosmochimica Acta, 30 (9), 921, 1966) which are to serve as internal reference samples for routine determination of rare earths by X-ray fluorescence (XRF) following a group separation on an ion-exchange column. The resin will be pelleted.

Dr. Olav Christie and Mr. S. Bergstol are using an X-ray fluorescence method extensively now for the analysis of rocks and Dr. Christie has written a computer program to provide a matrix correction. The full correction is made for Al, Ca is corrected for Fe, Si for Ti, Fe, Mg and Ca (my notes are confused here – further details have been requested from Dr. Christie); no correction is made to values for Ti, Fe, Mg (if less than 2%) or K. The sample (1 g) is fused with 2 g of lithium tetraborate, but no La_2O_3 is used. Dr. Christie expressed interest in the two papers by

Dr. Traill and Mr. Lachance published in Canadian Spectroscopy and copies have been sent to him.

3. Chemistry Division, Department of Industry, Trondheim.

The Department of Industry consists of the Chemistry, Geological (formerly Geological Survey) and Geophysics Divisions. The Chemistry Division has a staff of 35, headed by Director Aslak Kvalheim, and combines the analytical service functions of the GSC and Mines Branch, in that services are provided to geologists (approx. 25) and, fairly extensively, to industry (the latter on a fee basis). The Professional: Technician ratio is approximately 1:2; graduates from the Technical School are hard to get because of the competition from industry, and the academic qualifications of the others are similar to our Branch technicians.

The Chemistry Division is divided into the Spectrographic, Chemical, Geochemical Prospecting, Refractories and Metallurgy Sections, with a staff of 8-10 in the spectrographic laboratories. Dr. Kvalheim estimates that it takes about 1 year to adequately train a new technician in spectrographic methods. The working day (summer) is 8:$\underline{30}$ A.M. – 3:$\underline{00}$ P.M., with 20 minutes for lunch; they also work every other Saturday from 8:$\underline{30}$ A.M. to 1:$\underline{30}$ P.M. The spectrographic laboratory staff includes three professionals (including the Supervisor).

All samples to, and results from, the Chemistry Division are processed by Mr. Bersvensen; Dr. Kvalheim advises on the analytical needs when sample is other than routine.

The Spectrographic laboratories (named after V.M. Goldschmidt and L.B. Strock, respectively) do qualitative, semiquantitative and quantitative determination of major and trace constituents. The chief instrument (the spectrograph built by Goldschmidt and a Mannkopf prism instrument are also in use) is the ARL Quantometer, equipped with a Tape machine, and an ARL digital voltmeter-typewriter readout (just being installed). The instrument has two arc-stands; one is equipped for DC arc-air jet operation, using a 20 mm. flat pellet (0.5 mg sample mixed with Li_2CO_3 and cellulose) in the electrode, and the other operates with the tape. A switching mirror is used to make either arc stand instantly operational. Other samples are mixed with $SrCO_3$ as buffer, or in a 1:2 ratio with carbon only. Trace element/ major element analyses are done in the ratio of about 1:1. There is also a single channel Philips X-ray fluorescence spectrometer in use; at the time it was determining Cu and Zn in mill tailings, in the range 0.2-30%.

The chemical laboratories do silicate and ore analysis, and the usual geochemical determinations. I was shown one interesting method for sulphur in pyrite (40-50% S); after acid decomposition the pH is adjusted to 4.5 (pH meter), sodium alizarin sulfonate is added and the solution is titrated rapidly to a pink end-point with barium chloride solution.

Samples are routed to the appropriate laboratory on the basis of the type of work and the precision required. Little applied research is done because of lack of space and time for it; more is done in the spectrographic than in the chemical laboratories.

There is evidently no reclassification procedure as we know it. An individual can usually rise through two levels, provided that a vacancy exists.

4. Geological Division, Department of Industry, Trondheim.

My visit was principally to the rock and mineral analysis laboratory and its Head, Dr. P.R. Graff. The laboratory, with a potential staff of 5-7, avoids routine work (this is usually handled by Kvalheim's laboratories) and serves the needs of the geologists (Public samples are sent to the appropriate geologist, who may request help from the laboratory). There are now, in addition to Dr. Graff, two technicians and a Praktikant; one more professional and Praktikant are being sought. The Praktikant is one who has finished High School and is ready to enter a Technical High School, but who is working for one full year in a laboratory at his chosen speciality; the Technical High School requires one year of practical experience in order to obtain a degree (or diploma?) and, instead of working this off in four summer periods of three months each, students prefer to work a full year at the beginning and thus be able to take holidays each year. The advantage to laboratory is obvious and there are evidently many applications for suitable openings.

The laboratory handles 200 – 300 samples per year, using mainly classical methods. Total Fe is done on an aliquot of the pyrosulphate fusion of the R_2O_3 group; a 0.4 g sample is decomposed with acids and aliquots taken for the colorimetric determination of Ti, Mn and P, and the flame photometric determination of the alkalis. Fluorine is done by the usual distillation, followed by measurement of the bleaching of a peroxidized titanium solution. The Penfield method is used for total water, but the PbO flux portions are individually weighed into small, corked test-tubes in readiness for a number of determinations. Some assaying for Pt, Au and Ag is done. An atomic absorption instrument is to be added; spectrophotometric measurements are made with a Zeiss Optica spectrophotometer, and with a Beckman DB. Analytical balances (Mettler) rest on cork pads supported by a solid cement bench.

Dr. Graff has made some preliminary use of the new rapid scheme described by Dr. Langmyhr and himself but he has now realized that a method applied with reasonable success to 3 samples soon runs into difficulties when applied to a wider variety of samples. The method for Si has given the best results but, apart from its obvious usefulness when the quantity of sample available is small, the scheme must be used with caution, and Dr. Graff has not used the full scheme as yet.

Total U and Th is obtained by a counting procedure, and then U is determined fluorimetrically. They hope to get into gamma-ray spectrometry soon (Dr. D. Thorkildsen).

Geologists are usually assigned to a two-room quarters, consisting of an office (about 20' x 20') and a laboratory (10' x 20', complete with fume-hood). I gather that the laboratory is used chiefly for additional storage space. Those without the laboratory are given a larger office!

I had the pleasure of again meeting Mr. Ingvaldsen, the Administrative Director of the Department, who was interested in learning about recent developments in our Branch and Department. He asked that his regards be passed to Dr. Fortier, Dr. Lord, Mr. Hall and Dr. Gadd.

B. Visits in England

1. International Mineralogical Association, Cambridge.

The IMA Meeting was held in the Department of Mineralogy and Petrology, Downing Place, from August 30 – September 3 inclusive. I was able only to attend the last two days, September 2-3. There were about 350 Members and Accompanying Members and President Tilley and his colleagues are to be highly commended for a well-planned, well-organized and most successful meeting.

I attended the following sessions and heard 28 papers:

Friday, Session E: Synthetic minerals and miscellaneous.

Session G: Symposium II; Pyroxenes and Amphiboles.

Session H: Symposium II; Pyroxenes and Amphiboles.

Saturday, Session I: Symposium II; Pyroxenes and Amphiboles.

Session J: Symposium II; Pyroxenes and Amphiboles.

Copies of the Program, the Attendance List and the Program Abstracts have been given to Dr. R.J. Traill. The writer has nothing of significance to add to these latter, except to that of White, Henderson and Mason, "Secondary minerals produced by weathering of the Wolf Creek meteorite" (Friday, Session E); the two new minerals described are

Reevesite - $Ni_6Fe_2^{3+}(OH)_{16}\,CO_3 \cdot 4\,H_2O$

Cassidyite - $Ca_2Ni(PO_4)_2 \cdot 2\,H_2O$

Reevesite is isostructural with pyroaurite and cassidyite with collensite.

During the sessions much use was made of a Travelgraph. This is a device which enables the lecturer to write on the special translucent flat surface of a desk projector and his notes are projected, enlarged, upon a screen over his head.

The meeting afforded an opportunity to renew acquaintance with many people, and also to establish contact with others such as T. Hugi (Switzerland) and K.K.G. Hytonen (Finland).

The writer attended the Second Business Meeting of the IMA (Saturday, 4:$\underline{15}$ P.M.) as a delegate from the Mineralogical Association of Canada, but was not called upon to act in any official capacity whatsoever. The meeting was primarily for receiving the Reports of Commissions, as follows:

1. Abstracts (Hugi) - considered 3 proposals for abstract preparation and a subcommittee has been established to consider that proposed by Prof. A. Preisinger (Austria) which involves key-wards and an IBM 360 program.

2. Cosmic mineralogy (Sandrea) - meteorite studies; why is there no Canadian representation on the Commission?

3. Mineral Data (Strunz) - classification list of new minerals and supporting data (about 50% already published) will be distributed to members of the Commission.

4. Museums (Frondel) - to proceed with publication of World Directory of Mineral Collections as a small, inexpensive edition to stimulate criticism and promote more data. Mr. P.E. Embrey (U.K.) and Dr. R.H. Jahns (USA) to handle preparation. Dr. C. Guillemin (France) will replace Dr. Frondel as Acting Chairman. Much discussion about probable cost.

5. New Mineral Names (Fleischer) - presented by Dr. Guillemin in absence of Dr. Fleischer.

6. Ore Microscopy (Uytenbogaart).

The writer also visited the laboratory of Dr. J.V.P. Long, to see the new ion probe on which he is working. There were many visitors, and unfortunately Dr. Long was soon called away, so that I was able only to get a very cursory look at the device; an ion gun focusses ions on the sample, and the secondary ions are collected and analyzed mass spectrometrically. It resembles the laser microprobe in its functions (but the target area is 1-5 microns in width) and Dr. Long has actually considered the use of a laser beam. I was also able to see the new Cambridge Geoscan and the Microgeoscan (Corpuscular Beams, Inc.?) in Dr. Long's laboratory.

Mr. J.H. Scoon, the chemist with the Department, was unfortunately on holiday during the meeting. A quick tour of his laboratory on my own showed no significance changes in equipment since my 1962 visit.

2. Warren Spring Laboratory, Department of Industry, Stevenage.

This was a return visit to a group of laboratories whose work parallels ours very closely and which I found of particular interest in 1962. Dr. Paul Jeffery unfortunately was away on a lengthy administrative course but Messrs. Carr-Brion, Bakes and Gregory filled in admirably for him. Dr. A.J. Robinson, Deputy Director, was also very hospitable. The writer was accompanied on this visit by Professor E.B. Sandell, University of Minnesota.

The following developments are of interest:

- infrared spectroscopy is being used for semiquantitative analysis of mineral mixtures, but more emphasis is placed upon determination of adsorbed flotation agents on silicate minerals; the instrument used is a Grubb-Parsons Spectromaster. They are developing an on-stream IR analyzer using multi-attenuated total reflectance.

- a Philips 1212, fully automatic/manual, 24-channel X-ray fluorescence spectrometer is now in operation; both powder and solution samples are used. A computer program is being prepared to handle the data obtained on paper tape. An on-stream X-ray fluorescence analyzer has been designed to determine Pb and Zn in slurries, using Cd^{109} as the radioactive source; the reproducibility is 0.5% relative and the apparatus is also capable of sensing a change in particle size and correcting for it. (see pp 438-43, XII Colloq Spec. Int. Exeter)

- the 3 emission spectrographs operating simultaneously off one arc-stand have been reduced to two, and are used to determine those elements falling below the limit of determination by XRF. A direct-reading instrument is on order.

- the gas chromatographic method has not been extended beyond the determination of carbon dioxide (can determine down to 0.01% CO_2).

- both the Unicam SP-90 atomic absorption spectrophotometer, and the AA modification for the SP-900 flame photometer are in use here for the trace element determination of Ni, Co, Cr, Mg, Mn, Fe, Cu, Zn, Pb and Mo in a variety of materials; the signal intensity for Zn is affected by niobium.

- Mr. Gregory has developed a new method (see forthcoming Analyst) for trace Sn, using salicylideneamino-2-thiophenol with lactate as stabilizer. Only Cu interferes. Mr. Gregory also recommended the Koch-Light Laboratories, Colnbrook, Bucks, England, as a source of reliable organic reagents.

- a pyrohydrolytic method is now being used for fluorine determinations in the major and minor concentration ranges (Willard-Winter distillation is still used for trace concentrations). The heating temperature should be as low as is possible to avoid physical spattering; the flux is a Bi_2O_3-V_2O_5 eutectic mixture, and Alundum boats are used. A colorimetric finish using Xylenol Orange is now favoured.

- Mr. Bakes is assembling equipment for neutron activation analysis using fast neutrons. The source, a deuterium-tritium mixture in target enclosed by gaseous deuterium-tritium phase (target thus does not become depleted), is at bottom of 16-ft. hole and it is hoped that further shielding will be unnecessary. A neutron flux of 10^{11} neutrons/sec. is expected. F, O, Nb, Ti and Ba are among those elements to be determined first; a paper on the determination of fluorine (50% fluorite in tailings) has been submitted to the Analyst.

- among new major pieces of equipment now in the Laboratory are a Gem 7 electron microscope (Japanese), an ultramicrotome, and a Cambridge Geoscan microprobe analyzer.

3. Department of Geology, University College of Swansea, Wales.

Dr. Peter Hooper (Petrology) has been active in the X-ray fluorescence analysis of rocks and this visit was made primarily to see him. Dr. T.W. Bloxam (Geochemistry) was fortunately still there as well (he left next day for an exchange visit to Moscow) and I was able to spend an hour with him.

Dr. Hooper was using an unfused powder method (300-mesh material) to determine Mg, P, Ti, Mn and Sr; the instrument is a Model PW 1540 Philips all-vacuum X-ray spectrometer, with a Cr tube for light elements, W tube for Al and others. He found that particle size introduced problems and abandoned the method. For major constituents the sample (1 g) is fused with 10 g borax glass (prefused Na tetraborate) in a graphite or, preferably, in a Palau crucible (a film of graphite on the bead caused trouble); the ground fused material is mixed with bakelite (1:5), pressed and then "cured" at 110°C for 15 minutes to harden the surface and prevent deterioration. The precision data are generally good (the std. deviation (1 sigma?) for SiO_2 is ± 1%). For minor and trace constituents the above method involves too great dilution and the introduction of too much impurity; he uses a 2:1 fusion with lithium tetraborate (to minimize the interelement effect). Each sample and standard is read 7 times, and each set consists of 3 samples and a standard. Reproducibility is no problem; Dr. Hooper feels that the greatest error lies in the sample preparation and questions the need for high accuracy in view of the nature of most samples.

The determination Ca, Mg and Fe in pyroxenes by XRF is being studied. A 50 mg sample is sprinkled on Cellotape; synthetic stds. are being used at the beginning and an accuracy of ± 1% is expected.

Dr. Bloxam supervises a rock and mineral analysis laboratory for the use of graduate students. The following are comments on the methods used:

- Si is done by the quinoline molybdate method.

- Riley's method (extraction with oxine) is used for Al; double shaking is necessary before extraction and the separatory funnel should be flushed twice with chloroform. All reagents should be buffered to 5.1 (pH meter).

- for a "blank" determination of MnO, decolourize the permanganate with H_2O_2 and use same solution.

- Ca is titrated with EDTA, using murexide screened with naphthol green B as indicator; Dr. Bloxam prefers Solochrome Black 6B to Eriochrome Black T for the titration of Mg (no screening necessary).

- the alkalies are determined by flame photometry, with Li buffer. The stability of the flame emission is improved by masking the flame.

- a Pregl semimicroapparatus is used for C and H_2O; CO_2 (inorganic) is done by usual evolution method. The sample is heated at 1000 °C in oxygen (350 ml used per sample) and the gas passed over Cu (700 °C) and plumbate - Ag (200 °C) to remove S and Cl.

- fluorine is done pyrohydrolitically using a silica tube to which a small condenser is attached at the exit; the effluent passes through a sandwich made up of glass beads between two layers of quartz wool. A small heating mantle is wrapped around the front part of the combustion tube to prevent condensation of water from oxygen-steam mixture (100 ml O_2 per min.). The sample + WO_3 (1:2) is heated at 900 °C and the effluent is absorbed in H_2O (200 ml flask is cooled in beaker of water). The absorbate is diluted to 200 ml, an aliquot (10-600 ppm F) taken and diluted to 100 ml, and the determination finished using cerium alizarin complexone. Two standards (NaF) are used to prepare the calibration curve for each series of determinations.

- $S < 2\%$ is done by XRF.

- Chloride is determined nephelometrically (see Boltz, p. 166) using an attachment for an EEL spectrophotometer. Dr. Bloxam sees no reason why chlorine could not be determined on an aliquot from the fluorine pyrohydrolysis.

- uranium and thorium are determined with a Nuclear Enterprises (GB) Ltd. Scintillometer, to 1 ppm. A 200 g sample is placed in an annular metal container that fits over the crystal.

The university buildings are new and the campus, situated on Swansea Bay about 20 minutes walk from the city centre, is very impressive.

The Geology Department has a new, imaginative and extensive "museum", in the style of Logan Hall. Dr. Bloxam designed and helped build a floor to ceiling coloured geological map of Great Britain (6 1/2 miles to 1 inch) in which the various formations are lighted by pressing the appropriate button.

4. British Ceramic Research Association Laboratories, Stoke-on-Trent.

This was a repeat visit made primarily to discuss the progress made in their use of the direct-reading emission spectrograph that was being installed at the time of my last visit, and to discuss general silicate analysis with Mr. H. Bennett.

Most of the day was spent seeing and discussing the emission spectrographic methods with Mr. Bennett and Mr. R.P. Eardley. The range of materials analyzed by the chemical and spectrographic laboratories is very wide and as a result they are unable to use general methods. Much of their effort is devoted to the complete decomposition of the sample. Much work has been done on the rotating disc electrode (rotrode) technique and they are able to get excellent results for the major and minor constituents in such varied samples as high-silica material, burned magnesite and chrome-bearing refractories; the instrument is a Hilger and Watts 3-metre grating Polychromator, with typewriter readout. The original solution procedure (1964; fusion with NaOH in a nickel crucible, dissolution in HCl with Co added as internal standard) for high-silica materials has been replaced by a combination of acid decomposition (HF, HNO_3, $HClO_4$), fusion of the total residue with Li_2CO_3 - $Li_2B_4O_7$ flux and dissolution in HCl. This method enables the flame photometric determination of Na and K in an aliquot of the solution and reduces the loss of Fe to the crucible. Burnt magnesites and chrome-bearing materials are fused with a Na_2CO_3 - H_3BO_3 mixture in a platinum crucible and dissolved in HCl.

Mr. Eardley stressed the importance of factors involving the rotrode, such as source (Johnson-Matthey are consistently good, Ringsdorff are too hard), the depth of immersion of the disc in the solution dish, and the proper form of dish (Hilger is good, ARL unsatisfactory).

There is a need for rapid semiquantitative method as well, but because the wide range of materials precludes the use of solutions, a pelleting-AC arc technique is now in use. The sample (50 mg) is mixed with 100 mg Li_2CO_3 and 800 mg pelleting graphite and pressed at 2 tons load (Hilger Press). A 20-second integration time is used and 23 elements are read; the accuracy is about ± 25% at best. The direct-reader is, incidentally, regarded at BCRA as part of the 'wet' chemical analysis instrumentation.

The British Non-Ferrous Metals Research Association (Euston Road, London, N.W. 1) has recently prepared a series of Cu alloys specifically for the study of mass absorption effects in X-ray fluorescence spectroscopy. This was drawn to the attention of Mr. Lachance.

Mr. Bennett described a new minimum-flux procedure for the classical determination of SiO_2 that is fairly rapid. The sample (1 g) is fused for 5 min. (30 min. for bauxite) with 1.5 g fusion mixture and 0.2 g boric acid at 1200 °C; the cake is dissolved in 5 ml 1:9 H_2SO_4, 15 ml HCl and 15 ml H_2O, containing 1 g Sorbitol. The solution is evaporated and a single separation made of SiO_2. The effect of the Sorbitol on the R_2O_3 precipitation is not known.

There were few other new developments in chemical methods. The potential use of atomic absorption spectroscopy is being investigated, using an EEL instrument as a method for determination of small amounts of Ca (0.005%) and Mg (0.0001%). They are considering the purchase of a Techtron instrument. The Fuels Section is investigating the determination of CO_2 in clays, etc., by gas chromatography, using a conductivity cell to measure the separated gas.

Mr. Bennett is using a new automatic liquid dispenser (Jencons, made by Quickfit and Quartz) that should be very useful in our FeO method.

5. Central Laboratory, Pilkington Brothers Ltd., St. Helens.

This also was a second visit, in this instance made chiefly to observe the development, over the past four years, in the use of X-ray fluorescence spectroscopy for the analysis of glass and related products and materials. Mr. Frank Hartley (Chief Chemist), Mr. W. Ward (Asst. Chief Chemist), Mr. F.J. Cliffe (Head, Instrument Section) and Mr. Derek Ashley (X-ray fluorescence) were most hospitable and helpful.

X-ray fluorescence spectroscopy is now being used extensively and the importance of emission spectroscopy has diminished. No changes have been made in the latter operation since 1962, except the introduction of a general semiquantitative method for major constituents involving fusion of the sample with lithium tetraborate (Co and Sr as internal standards). A rotrode method is used for some types of samples.

Because the samples have a relatively small compositional range, both individually and collectively, it has not been necessary to develop a fusion method for their Philips X-ray fluorescence spectrometer. For fibreglass, sands and silica brick, the sample is mixed with bakelite (1:10) and cured by heating to give strength to the 1 1/4" disc; it also improves the reproducibility of readings. A simpler procedure using a 1:3 ratio of sample/bakelite, with mowiol (polyvinyl acetate (Farbwerke Hoechst, Frankfurt) as binder, is used when speed, rather than accuracy, is required. Chemically analyzed materials (e.g., glass) are used as standards and these are prepared at about 2-week intervals. It is believed that the surface of the disc changes with time, possibly due to the absorption of sulphur compounds.

The stainless still mold cap of the press has been replaced by tungsten carbide because of the pick-up of iron at the disc surface. The harder surface requires less polishing.

SiO_2 is still done chemically by the Jeffery-Wilson procedure, except for cathode-ray glass for which an accuracy of ± 0.1-0.2% is required.

There are no changes in their methods for fluorine and chlorine.

Since 1962, an impressive new administrative building and employee recreation area has been added, and also a Museum of Glass Technology (open to the public) that requires much more than the hour spent by the writer to do it justice.

6. Department of Geology, University of Newcastle upon Tyne.

A visit was made here to Dr. M.H. Battey, whom the writer had met before in England and during his visit to the Survey in 1965. Dr. Battey is developing a method for X-ray fluorescent rock analysis based upon the method of Norrish and Hutton (CSIRO, Adelaide, Australia). The sample (3 g) is fused with a special mixture (Li_2CO_3, $Li_2B_4O_7$, La_2O_3 and $NaNO_3$) in a gold-lined Pt crucible (Au surface is non-wettable, produces a fluid melt that pours easily) and poured on to a graphite disc (1 1/4" dia., slightly dish-shaped) on a hot plate, surrounded by a brass ring and pressed into a thin glass disc with an aluminium plunger. The disc is then annealed between asbestos sheets on a hot plate (some breakage occurs). The surface has the appearance of the skin of an orange but this is said not to interfere in the reading, although positioning of the disc is important. Dr. Battey has been getting erratic results for Al, particularly for ultrabasic rocks; this could be due to pick-up of Al from the aluminium plunger used to form the disc.

There are also small chemical and spectrographic laboratories connected with the Department. The University is, to the writer, an unimpressive collection of heterogeneous buildings (Victorian to ultramodern) surrounded by, and flowing into, the city. Much new construction is in progress.

Dr. Battey showed me a single pocket-book version of "An introduction to the rock-forming minerals", of Deer, Howie and Zussman, of which I was unaware. It is published (1966) by Longmans, and sells for about $7 in the United Kingdom.

7. Department of Geology, University of Durham.

Dr. Battey accompanied the writer on this visit to the Science Laboratories of the University on South Road. These buildings are new, well-situated, and very impressive. Unfortunately, most of the members of the Department were away at the time of my visit and I met only Dr. Emeleus and Mrs. Kaye, a spectrographer who had studied with Professor L.H. Ahrens and who was returning to join Professor S.R. Taylor at Australian National University after two-years at Durham . Mrs. Kaye kindly acted as my guide.

Mrs. Kaye has been using a Hilger quartz spectrograph for chiefly quantitative analysis; the methods are DC arc with anode excitation. The instrument is used in conjunction with the XRF spectrometer, to do those elements not done by the latter and to analyze very small samples. Mrs. Kaye has been determining trace fluorine by emission spectroscopy. No internal standard, CaF_2 band head, 9 arcings; the precision is comparable with that obtained by pyrohydrolysis (e.g., 450 ± 58 ppm).

The electron microprobe is a Cambridge Geoscan. It has been in use for 1 year and has evidently given much instrumental trouble.

Dr. M.J. Solomon (University of Tasmania, Hobart) has been at Durham on exchange for a year. At Tasmania he has been using a Techtron AAS to determine Ni and Co (5-10 ppm) in sulfides, chiefly pyrite. The sample is first roasted slowly in air to get rid of bulk of the sulfur before dissolving in acids. Fe may cause erratic results (molecular absorption?) but precision is generally satisfactory. A paper on this work is in preparation.

The X-ray fluorescence spectrometer is a single channel Philips instrument, with Cr tube. Dr. J.G. Holland has worked out an "as is" method for determining 9 elements (Na is not too good) and has written a computer program (in Extended Mercury Autocode for an Atlas Computer) that has proved very successful. Dr. Holland was unfortunately away at the time (he was presenting a paper on this subject at a meeting of the Yorkshire Geological Society; publication ("A self consistent mass absorption correction for silicate analysis by X-ray Fluorescence") is planned in Spectrochimica Acta, but Dr. Holland has sent me a copy of the computer program) but Mrs. Kaye showed me a copy of the paper prepared by Holland and Brindle, from which the following is summarized.

The sample is ground as finely as possible and is then pelleted at 5 tons/sq. in, using 3 drops of 2% Mowiol as binder. The method of analysis is an iterative one, involving reference to one standard (W-1) which is used to calculate the approximate composition of G-1; using this, the total mass absorption is determined for each operational wavelength and the corrections used to make a more accurate estimate of the composition of the unknown (G-1). This is in turn used to obtain a more accurate value for the total mass absorption. It thus gives a self-consistent chemical composition which is then corrected for other deviations from proportionality by reference to a set of standards, using a quadratic regression relationship. It is applied only to whole rock analyses.

8. Geological Survey of Great Britain, London.

The future role of the analytical laboratories of the Survey is in some doubt because of the amalgamation of this and other organizations under the National Environment Research Council. This latter covers Oceanography, Nature Conservancy, Geological Survey and the Overseas Geological Surveys. The largest unit is the Institute of Geological Sciences, with Dr. K.C. Dunham (Durham) as Director Designate. As a result of this uncertainty, the Survey laboratories are marking time until decisions are forthcoming.

The fluorine method, involving ion-exchange purification of the distillate and a spectrophotometric finish (Eriochrome cyanine R, or Solochrome cyanine RS #279) has proven satisfactory. Purity of reagent is essential (Na_2SO_4 present) and Mr. Sergeant has found that supplied by George T. Gurr Ltd., London, SW 6 (thought to have been recently taken over by British Drug Houses Ltd.) to be the best.

Some thought has been given to atomic absorption spectroscopy but no action is likely at present.

A new variant on the Titan Yellow method for Mg (0.1-20% Mg) has been developed. Following a basic succinate separation of Fe and Al, the Mg is determined as the Titan Yellow Lake, glycerol and sucrose being added to suppress Ca and polyvinyl alcohol to aid in suspension of the lake.

Boron is determined as follows: the sample is fused with K_2CO_3 and dissolved in 1:1 HCl, acetic acid is added and an aliquot taken; to this is added a mixture of acetic and sulfuric acids, acetic anhydride (to remove H_2O) and the reagent (curcumin in sulfolane), and the boron complex extracted with methyl isobutyl ketone and the O.D. measured at 555 mμ. The sensitivity is 1 microgram in about 5 ml and the accuracy, based on synthetic standards, is acceptable (no reliable natural standards are available).

A new pulverizer is used in the emission spectrographic laboratory – Grindex, GR-200, by Research and Development Instrument Company, 17 Stannary St., London, SE 11. It is like the Wig-L-Bug in operation but larger, and takes 2 capsules (agate, steel) holding up to 2 g each. The price, without capsules, is about $750.

9. Overseas Geological Surveys, London.

This is now the Overseas Division of the Institute of Geological Science (Dr. S.H. Shaw, Officer I/C, Overseas Division). The emphasis is changing from Commonwealth service to aid to newly independent countries which are losing trained foreign staffs; much training of chemists from these countries is also being done.

This repeat visit was to the Chemical Laboratories only (Mr. W.H. Bennett, Chief Chemist).

Dr. M. Rowlands has been working an identification of clay minerals by infrared spectroscopy for 2 years. The instrument is a Unicam SP 100 which has evidently given much trouble; it is no longer being made.

A TEMA Laboratory Disc Mill is now used to prepare samples. This is similar to our Bleuler mill but has a tungsten carbide liner and puck for the single large head. It is made by Tema (Machinery) Ltd., Banbury, England.

A Zeiss Elko II filter, double beam spectrophotometer is used for the colorimetric determination of major amounts of Fe, Si and P (phosphate rocks), in 2N H_2SO_4. The photometer cell is thermostatted.

Mr. J. Moore is using atomic absorption spectroscopy (Unicam SP 900, modified for AAS) to determine a number of elements. He has experienced difficulty with Mg in the 8-10% range, which may be due to 'salt' concentration (air-acetylene); aluminium depresses the sensitivity and Sr reduces the effect of Al but the net result is still a depression. Zn, Cu and Pb are determined with an air-propane mixture; he finds that better results are obtained with the 2170Å line than with that at 2833Å.

A Radiometer "Polariter" is used for the polarographic determination of Eu in monazite and Cd in zinc concentrates. Nd, Pr and Sm are determined in various materials with a Unicam SP 800 UV spectrophotometer; Al as a major constituent is also determined with this instrument.

The emission spectrographic laboratory is still concerned chiefly with trace element determination only. $SrCO_3$ is now being used as the buffer.

I had an opportunity to talk briefly with Dr. P. Bowden, formerly with the Geological Survey of Tanzania; he has been, and still is, involved with the evaluation of the data on the Tanganyika Tonalite (T-1).

10. The Department of Mineralogy, British Museum (Natural History), Cromwell Road, London SW 7.

This repeat visit was made chiefly to meet Mr. A.J. Easton, who has replaced Mr. D.I. Bothwell as a chemist with the Department, and to renew contacts with Drs. Max Hey and A.A. Moss. Much of the analytical work of the Department is for meteorite study.

Mr. Easton described an apparatus for accomplishing the decomposition of a small sample (5 mg) in acid in the absence of oxidation, prior to the determination of ferrous iron. The crucible plus sample is inserted into a hole in the bottom half of a plastic bottle and placed on a steam bath; acid is added and the top half is placed over it, nitrogen being forced in through the mouth and escaping through the join. After decomposition is complete the contents of the crucible are washed quickly into a flask containing 2, 2'-dipyridyl (Riley) or, better, 20% sulphosalicylic acid (4ml reagent, 4 ml aqueous ammonia).

The chlorination procedure has been improved. The attack of silicates by HCl in the chlorine has been minimized by inserting a tube containing marble chips dusted with $CaCO_3$ into the front of the train. A bubbler with concentrated HNO_3 will insure the recovery of all sulphur. The preparation for chlorination is as follows: crush the meteorite sample to about 24-mesh and remove metal with a weak magnet; crush the residue to 100-mesh in an agate mortar and remove another magnetic portion if desired. Chlorinate the magnetic portion and obtain silicate impurity (and some taenite, which can be separated magnetically) in the boat, the Fe and S in the collecting tubes. Chlorinate the non-magnetic portion as above and obtain the silicates and some sulphides in the boat, the Fe and some S in the collecting tubes. It is recommended that the nitrogen be admitted first, then nitrogen plus chlorine.

In the Shapiro-Brannock method for the determination of total iron by 0-phenanthroline, it is recommended that double the given concentration of reagent be used.

Atomic absorption spectroscopy is done with a Unicam SP 90. Mg, Zn and Cu have been determined to date; the Pb hollow-cathode tube proved ineffective.