

GEOCHEMISTRY, ISOTOPE AND NUCLEAR GEOLOGY

73. PROJECT: PREPARATION OF SYNTHETIC MIXTURES OF MINERALS FOR INFRARED TESTS

PERSONNEL: S. Abbey, H.R. Steacy, W.H. Champ, and E. Schiller

Specimens of the purest available quartz, albite, microcline, and muscovite were ground to the usual fineness for chemical analysis. Portions of each were weighed out to give three mixtures of compositions characteristic of granite, quartz monzonite, and granodiorite. After thorough mixing, portions of the mixtures were ground exceedingly fine, as were also portions of each pure mineral and of a rock sample for which modal and normal analysis were available. Part of each finely ground material was sent to each of four manufacturers of infrared spectrometers, who had offered to attempt to determine the mineralogical composition of the mixtures and the rock by using the pure minerals as reference standards.

Results from one manufacturer were entirely unsatisfactory, since they did not treat the samples in the manner requested. A second manufacturer provided a detailed report, at least half of the results on which were good. The data is being studied with a view to improving the remaining results. The other two manufacturers have not yet reported.

(S. A.)

74. PROJECT: LEAD AND SULPHUR ISOTOPE GEOLOGY OF KENO AND GALENA HILLS, YUKON TERRITORY

PERSONNEL: R.W. Boyle, R.D. Stevens, and R.K. Wanless

The lead isotopic analyses of 150 samples of galena and other lead minerals from the region have been completed. All of the samples studied have anomalous lead isotope abundance ratios, indicating the addition of excess radiogenic lead. The isotopic ratios fall into two main groups. One group, comprising about 75% of the samples, is confined within small limits whereas the majority of the remaining samples have accumulated varying, large concentrations of radiogenic lead.

The lead isotope data is to be combined with the results of the sulphur isotope studies. All sulphides have been converted to SO<sub>2</sub> and the first isotopic analyses (25) have been completed.

(R. K. W.)

75. PROJECT: DIRECT-READING SPECTROCHEMICAL  
LABORATORY

PERSONNEL: E. M. Cameron and Mrs. T. D. Dawes

In recent years it has become apparent that progress in many areas of pure and applied geochemistry is dependent on the development of rapid analytical techniques capable of producing large numbers of precise analyses of rocks. A direct-reading optical spectrometer was obtained to fulfil this need for a number of elements of geochemical interest.

Since this instrument was installed in December 1961 a method has been developed for the rapid analysis of Si, Al, Fe, Ca, Mg, Mn, Ti, and Na. The rock samples are fused with lithium tetraborate and strontium carbonate and are briquetted with graphite, before being excited in a high voltage spark. Similar techniques for the analysis of certain trace elements are presently being investigated.

(E. M. C.)

76. PROJECT: MAJOR-ELEMENT CONTENT, GRANITIC ROCKS,  
NORTHWESTERN ONTARIO

PERSONNEL: E. M. Cameron, R. F. Emslie, R. H. C. Holman,  
and Mrs. T. D. Dawes.

The major-element content of several hundred samples of granitic rocks, collected during the "Roads to Resources" project in northwestern Ontario, were analysed late in 1962. These analyses were made using newly developed direct-reading spectrochemical techniques, which are both rapid and precise.

The project was undertaken to provide information on the genesis of the various granitic rocks within the area studied and also to evaluate the use of rapid chemical analyses as a tool in mapping granitic areas.

(E. M. C.)

77. PROJECT: OPERATION OF HIGH PRESSURE APPARATUS

PERSONNEL: K. L. Currie

During 1962 the apparatus was assembled and successfully tested at design pressure and temperature of 50,000 psi and 650°C. Sealing difficulties were encountered in certain

pressure ranges and modifications of the sealing rings have been made. Experiments on quartz and albite solubility are in progress.

Specimens for experimentation were collected from the Crow Lake dome, a granitic body in the Grenville structural province. Initial chemical results indicate that this is a favourable location for determination of the role of water in granite formation.

Construction of apparatus to reach 150,000 psi and 600°C, using the present apparatus as a first stage is now under way.

(K. L. C.)

78. PROJECT:      RADIOCARBON DATING

PERSONNEL: W. Dyck, R.K. Wanless, and others

Carbon 14 measurements completed during 1962 with the 2-litre counter provided 64 radiocarbon ages of samples the majority of which were collected by officers of Geological Survey of Canada. Most of these ages have been submitted for publication in Radiocarbon, vol. 5, 1963.

Twenty-one C-14 measurements were performed on samples of known age and are a part of two research projects initiated in 1961 and 1962.

One of these projects has as its objective a study of the variation of the C-14 content of terrestrial plants with time and involves the C-14 analysis of tree rings of known age and annual growth of present day plants. Analyses to date show that the C-14 concentration in tree rings during the past 1,100 years has not varied by more than  $\pm 2.5\%$ . 1961 and 1962 maple leaves from the Ottawa area have a C-14 concentration of 22% and 33%, respectively above that of the pre-thermonuclear bomb testing era.

The second project deals with the C-14 concentrations of shallow-water sea shells of known age from Canadian shores<sup>1</sup>. Shells collected before the advent of thermonuclear explosions have a C-14 content of approximately 1% below that of terrestrial plants, whereas shells collected since show an increase in the C-14 concentration of 1% to 2%.

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<sup>1</sup>Most of the samples involved were collected and/or selected by W. Blake Jr.

In addition to the routine analyses, an all-metal filling line, a 5-litre radiocarbon counter, and a matched set of electronics were assembled and tested. The background count of this arrangement was found to be 3.0 c/min., 3.7 c/min., and 4.4 c/min. at 1, 2, and 3 atmospheres of CO<sub>2</sub>, respectively, and the modern wood count 33 c/min/atm. CO<sub>2</sub>. Calculations show that this counter will permit the extension of the present age limit of 40,000 yrs. to 47,000 yrs.

(W.D.)

79. PROJECT: GEOCHEMISTRY OF THE CANADIAN SHIELD -  
REPORT OF PROGRESS

PERSONNEL: K.E. Eade, W.F. Fahrig, and J.A. Maxwell

The object of this study is: (1) to determine the major and minor element composition of the Canadian Shield; (2) to determine the composition of individual cratonic segments of the Shield so that they can be compared, and geochemical evolutionary trends evaluated; and (3) to relate element abundance to lithology within each segment in order to gain increased knowledge of primary lithology and tectonic events.

Composite samples representing roughly 150,000 square miles have been prepared and are ready to be sent forward for chemical analysis. The composites were derived from several thousand individual hand specimens.

(W.F.F.)

80. PROJECT: MOBILE SPECTROGRAPHIC LABORATORY

PERSONNEL: R.H.C. Holman, P.J. Lavergne, and C.C. Durham

The construction of a specially designed mobile spectrographic laboratory was completed. The unit is mounted in a modified house trailer (24' x 8') and can be easily hauled by road with a truck. Essential spectrographic equipment includes a 1.5 metre Jarrell-Ash grating spectrograph, a D.C. arc source, a photo-processor and an A.R.L. densitometer/comparator. Power is obtained from the domestic hydro-electric supply available along nearly all roads navigable with the unit. Water is pumped from a nearby well, lake, or stream. At the close of the season the spectrographic equipment is taken into the laboratory in Ottawa for use during the winter; the spectrograph is mounted on a movable stand to facilitate this operation. During field trials in the summer of 1962 several technical problems were solved and the mobile laboratory is now fully operational for the

routine semi-quantitative determination of some twenty trace elements in rocks. The unit is intended to provide spectrochemical data with the minimum delay while field programs are underway, so that sampling may be more efficiently planned during geochemical studies.

(R. H. C. H.)

81. PROJECT: THE CHEMICAL AND SPECTROGRAPHIC ANALYSIS OF ROCKS AND MINERALS

PERSONNEL: J. A. Maxwell and staff of the Analytical Chemistry Section

This project covers the regular work of the Section, which in 1962 resulted in the analysis of 2,908 samples, involving 10,571 chemical determinations and 26,353 spectrographic determinations. It also covers the investigation, adaptation, and development of new methods and techniques required to facilitate the work of the Section, among which, in 1962, were the following items:

1. a colorimetric method for traces of thorium in rocks;
2. titrimetric and colorimetric methods for small amounts of sulphur in rocks, especially in basic and ultrabasic varieties;
3. adaptation of a colorimetric method for titanium;
4. adaptation of an extraction-colorimetric method for small amounts of aluminium in the presence of large amounts of iron and titanium;
5. extension of the general quantitative spectrographic procedure for minor and trace constituents to cover some 40 elements in aluminosilicates, basic and ultrabasic rocks, iron ores and limestones;
6. improvement of the general semiquantitative spectrographic procedure to cover about 50 elements in almost any material to the 0.01% level;
7. the construction of a dual apparatus for the determination of  $\text{CO}_2$  in rocks;
8. the construction of an apparatus for the determination of water by the Penfield method;

Development work is continuing on a micro-combustion method for the determination of CO<sub>2</sub> and H<sub>2</sub>O, the flame-photometric determination of aluminium, the pyrolytic-colorimetric determination of small amounts of fluorine, and the quantitative spectrographic analysis of trace elements in specific minerals at the lowest possible limits of determination. An investigation is also continuing into the potential use of infrared spectroscopy in the mineralogical analysis of rocks.

(J. A. M.)

82. PROJECT: INVESTIGATION OF POSSIBLE VARIATIONS IN  
SAMPLE COMPOSITION AS A RESULT OF SAMPLE  
PREPARATION

PERSONNEL: J. A. Maxwell and S. Courville

Three different types of rocks were chosen for this experiment, a diabase, a granite, and a limestone.

The bulk samples, each weighing approximately 20 to 30 pounds, were reduced to chips of about 1/4" diameter in the sample preparation room. The samples were screened and quartered in the chemistry laboratory and then were returned to the sample preparation room to be processed as ordinary samples. Each sample was separated into five portions, four of which comprised the quartered 1/4" diameter material, and the fifth the fines, which were separated before quartering the sample.

Analytical work on the limestone samples is nearly complete and work will start soon on the diabase and granite. Unforeseen analytical requirements have increased the amount of work required and the length of time needed to bring the project to an adequate conclusion, but it is hoped to complete the project early in 1963.

Arising out of the analytical work on the limestone has been the need for a better method of determination of aluminium in low concentration. Two very different techniques were applied to verify the Al<sub>2</sub>O<sub>3</sub> values obtained by difference - a flame photometric method and a colorimetric method employing alizarin Red. The flame photometric method will be useful in other aspects of the work of the laboratory as well.

(S. C.)

83. PROJECT: PREPARATION OF AN ULTRABASIC ROCK STANDARD FOR ANALYTICAL USE

PERSONNEL: J. A. Maxwell, W. H. Champ, C. H. Smith, and staff of chemical and spectrographic laboratories

There is a need in our laboratories for a reference sample of ultrabasic rock in connection with the analytical work of the Upper Mantle Project. Such reference material having a well-established composition (including major, minor, and trace elements) would be of great value in the testing of new methods for their application to the analysis of similar material, and to the checking of current analytical work, not only in Survey laboratories but also in other laboratories engaged in similar work. It was decided that the Survey would obtain about 200 pounds of suitable material, would crush, grind, and homogenize it, and would retain about 20% of it, the remainder to be turned over to the Non-Metallic Standards Committee of the Canadian Association for Applied Spectroscopy, for analysis, certification, and distribution, similar to the way in which this committee is handling the syenite and sulphide standards now in process of certification.

A sample of about 200 pounds of picrite from the Muskox intrusion has been collected by D. C. Findlay and processing of this material will start early in 1963.

(J. A. M.)

84. PROJECT: A CRITICAL EVALUATION OF THE GRAVIMETRIC, TITRIMETRIC, AND COLORIMETRIC METHODS FOR DETERMINATION OF SMALL AMOUNTS OF TOTAL SULPHUR

PERSONNEL: J. B. Sen Gupta

With a view to developing a fast and accurate method of determining small amounts of total sulphur in a large number of rock samples, the existing methods developed in the past half century by various researchers were critically reviewed and 103 references were cited. It was concluded that the conventional barium sulphate precipitation method is unsuitable because of incompleteness of precipitation and interferences from foreign ions. Direct or indirect determination of sulphur by volumetric or colorimetric methods using various reagents such as barium chloride and chromate, lead nitrate, ethylenediamine tetra-acetic acid (EDTA), benzidine, 4-amino-4'-chlorodiphenyl, thorium borate amaranth dye, barium chloranilate, barium molybdate-thioglycollic acid, and others are considerably influenced by the presence of foreign ions, especially chromate, phosphate, silicate, and fluoride, which if present will also interfere by giving high or low results.

Combustion with vanadium pentoxide in a tube at 900-950°C. in a current of nitrogen and subsequent reduction of sulphur trioxide to dioxide by hot copper and determination of sulphur dioxide by iodate-iodide titrimetric or p-rosaniline hydrochloride colorimetric method appeared to be a promising one, though reported in the literature only for determination of materials other than rocks. This method was successfully applied in our laboratories after suitable modification for determination of traces of sulphur in acidic, basic, and ultrabasic rocks with rapidity, high precision, and accuracy.

A paper on this project is being prepared for publication in 1963.

(J.G.S.G.)

85. PROJECT: AGE DETERMINATIONS OF ROCKS AND MINERALS

PERSONNEL: R.K. Wanless, R.J. Traill, J.C. Paris, S.C. Robinson, J.A. Lowdon, J.H.Y. Rimsaite, G.R. Lachance, R.D. Stevens, W.D. Loveridge, and J.K. Van Peteghem

Potassium-Argon Age Determinations

K-Ar age measurements have been continued on a routine basis. To date, a total of 636 samples have been processed (603 of these being priority samples) and the results have been published or are to be published in the following papers:

| <u>G. S. C. Paper</u>       | <u>No. of Determinations</u> |
|-----------------------------|------------------------------|
| 60-17                       | 98                           |
| 61-17                       | 152                          |
| 62-17                       | 206                          |
| 63-17                       | <u>147</u>                   |
|                             | 603                          |
| Experimental Determinations | <u>33</u>                    |
| Total                       | 636                          |

Rubidium-Strontium Age Determinations

The experimental techniques required to process mineral concentrates and whole rock samples for Rb/Sr age measurements have been tested. Enriched rubidium and strontium isotope solutions used for the isotope dilution determination of the concentration of these elements have been prepared and calibrated. Ion exchange columns for the separation of the



rubidium and strontium fractions have been set up. Isotope analyses are carried out on a solid source mass spectrometer equipped with an electron multiplier. A three-filament source assembly is used for all Sr analyses in order to minimize the contribution of Rb<sup>87</sup> to the Sr<sup>87</sup> ion current. Contamination levels are monitored by processing all samples in duplicate using different samples-to-spike ratios for each fraction.

A preliminary study based on the Rb/Sr ratio in whole rock and mineral concentrates from various phases of the White Creek Batholith in S. E. British Columbia is nearing completion. It is hoped that the results, combined with K-Ar age measurements on the same rocks, may be of assistance in interpreting the age relationships between the various phases of the Batholith.

#### U-Pb, Th-Pb Age Determinations

During the early stages of development of isotopic studies in the G. S. C. laboratories all lead samples were converted to a gas, lead tetramethyl, and were analyzed on a gas source mass spectrometer. This method is dependent on relatively large samples (5 to 10 mgm.) and consequently its application is limited. Much smaller samples (from 1 to 10 micrograms) may, however, be analyzed by employing solid source mass spectrometric techniques. With this increased sensitivity, the problem of lead contamination originating in chemical reagents, glassware, laboratory air, etc. becomes a serious one and special precautions must be taken to reduce the contribution to negligible proportions. To facilitate this phase of the work a laboratory has been equipped with an electrostatic air purification system, plastic plumbing fixtures have been substituted for all fixtures containing lead, and the walls and ceiling have been painted with tygon paint. Special precautions have been taken to purify all reagents to render them free of lead contamination.

Plans are being made to apply these techniques to the determination of age based on the U-Pb and Th-Pb ratios in zircons.

(R. K. W.)

86. PROJECT: ISOTOPE CHEMISTRY OF SULPHUR IN ROCKS AND MINERALS

PERSONNEL: R.K. Wanless, R.W. Boyle, and R.D. Stevens

Four minor sulphur isotope studies, essentially of a reconnaissance nature, have been completed. These include studies of the sulphides from the Sullivan Mine, Kimberley, B.C. for G.B. Leech; sulphides from the Blind River region of Ontario for S.M. Roscoe; sulphides in radioactive deposits in the Bancroft area for S.C. Robinson; and a preliminary survey of the sulphides from various iron deposits in Canada for G.A. Gross.

(R.K.W.)

87. PROJECT: ISOTOPIC STUDIES OF CANADIAN ORE LEADS

PERSONNEL: R.K. Wanless, G.B. Leech, R.W. Boyle, and R.D. Stevens

This project was completed with preparation of a paper published in the Buddington Volume of the Geological Society of America, 1962.

Ref.: Leech, G.B. and Wanless, R.K.: Lead Isotope and Potassium-Argon Studies in the East Kootenay District of British Columbia; in Petrologic Studies: a Volume in Honor of A.F. Buddington; Geol. Soc. America, Buddington Volume (1962).

(R.K.W.)

88. PROJECT: ADSORPTION STUDIES

PERSONNEL: R.A. Washington

Studies were carried out of the variation with pH of adsorption of Co by quartz and feldspar, using Co<sup>60</sup> tracer. Little or no adsorption took place on either mineral from solutions having pH < 3. From solutions having higher pH, the adsorption increased with pH to a maximum (at pH 6 to 6.5) of ca. 0.5 %/g. for quartz (-250+325 mesh) and 1.5 %/g. for feldspar (-250+325 mesh). The adsorption apparently decreases at pH values greater than 6.5, possibly because the Co begins to precipitate as a colloidal hydrous oxide or basic salt. A peculiar feature of the adsorption (which is usually, but not always reproducible) is a marked decrease in adsorption at a pH of ca. 5.5, relative to higher and lower pH values. Before an interpretation of these observations can be attempted, further experiments will be necessary, including establishment of an adequate experimental method for measuring the surface area of the substrata.

Attempts to measure the adsorption of Zn failed because of a lack of sensitivity and accuracy of the counting technique for the  $Zn^{65}$  used as a tracer, and for other reasons. These experiments will be repeated after improvements have been made in the experimental procedures.

(R. A. W.)

89. PROJECT: PRELIMINARY STUDY OF NEUTRON ACTIVATION ANALYSIS

PERSONNEL: R. A. Washington

A preliminary study has been made of the analysis of Cd in rocks of the Keno Hill area by neutron activation. The results indicate that the sensitivity is better than that of the spectrographic method by an order of magnitude or more. Further improvements in the technique are being developed, including a method for more complete sample attack and a better procedure for chemical yield determination. Extension of the method to include other elements (e.g. gold, rare earths, etc.) is also planned.

(R. A. W.)