

GEOCHEMISTRY AT CALTECH

The Division of Geological Sciences moves into another new field of research, in its program to integrate the efforts of geologists, physicists, chemists and biologists.

by SAMUEL EPSTEIN

IN RECENT YEARS students of geological problems have felt an increasing need for the type of data that can only be obtained by methods developed and employed by physicists and chemists. Recognition of this need prompted physicists and chemists to become interested in geological problems and their contributions to the field of geology have been well established.

On the other hand, it has not been a common practice in universities to integrate the efforts of the geologists, the physicists, and the chemists towards training students or towards the solution of geological problems. Caltech has been among the first institutions to incorporate a coordinated program of physics and geology, both in teaching and research, in its geophysics and geology programs.

Two years ago, under the directorship of Professor Harrison Brown, a geochemistry program was set up to enable undergraduate and graduate students and staff members to undertake a variety of problems and to carry on this research under favorable conditions for exchange of ideas among the students of the various branches of geology. As a result of this conducive academic atmosphere, geological research related to paleontology, geomorphology and metamorphism has already elicited cooperative efforts involving geologists, geophysicists and geochemists.

The new facilities available in the Division of the Geological Sciences satisfy the needs of a large range of geological interests. Rock crushing, sizing and mineral separation facilities have been established for the purpose of preparing analytical samples under conditions where contamination is controlled carefully. The mineral separation processing employs heavy liquids,

electromagnetic separators, centrifuges and other equipment. The principal use of these facilities recently has been made in research on acid soluble trace elements in igneous rocks.

Wilbur J. Blake, a silicate analyst formerly with the United States Geological Survey at Denver, is in charge of facilities for making the highly specialized and difficult silicate analyses. Such analyses have become very important to geologists in efforts to precisely delineate chemical compositional differences of different rock types as related to the environment in which they are found in the field.



Prof. Harrison Brown and Sam Epstein, Senior Research Fellow, check the recorded results of the mass spectrometer in the Institute's new geochemical laboratories.

In many cases rapid qualitative chemical analyses for a series of rock samples are desired. For this purpose an emission spectrograph has been installed, with spectroscopist Arthur Chodos, formerly of the U.S.G.S., in charge. By recording photographically the intensity of characteristic light frequency emitted by the different elements of a vaporized rock sample, rapid analyses for most chemical elements present can be readily made.

This instrument is particularly useful in determining concentration of minor constituents of a rock. Concentrations of as low as a few parts per million can be readily determined by using the spectrograph, whereas such determinations by wet chemical means are practically prohibitive. The importance of data on minor constituents as clues for evaluating the genesis and history of rock formations is well recognized, and is evidenced by the number of members of the Division using this instrument as a tool in their research.

Mineral analysis

X-ray equipment including powder cameras, goniometer-spectrometer and Brown recorder are available for mineral analysis. This equipment has been extensively used for studying calcareous fossil remains for their aragonite-calcite composition in Professor Heinz Lowenstam's pioneering work. Important relations between the mineral compositions have been observed.

In addition to these facilities there are two modern general chemistry laboratories which are equipped for qualitative and quantitative wet chemical analyses. These have been in continuous use by chemists Walter Nichiporuk, Richard Kowalkowski and Aiji Uchiyama in studies pertaining to chemical composition of meteorites and in studies pertaining to uranium and thorium distribution in igneous rocks.

For those interested in radioactivity studies, four low

level alpha counters and two beta counters are available. The counters have been used extensively in determinations of natural radioactivity in rocks and in radioactive tracer techniques.

The facilities described above have been available, to some degree, to many geologists throughout the country, either in having been set up in some geology departments or else in having been provided as a service by the United States Geological Survey.

Among the most recently developed techniques and tools now set up at Caltech are those associated with isotope work. Two mass spectrometers for lead isotope analyses and one mass spectrometer for isotopic analyses of the lighter elements have been constructed under the supervision of C. R. McKinney, Senior Research Fellow, and will be available for research.

Since Professor Aston, the English physicist, first showed that the lead isotope method promised to be a method of absolute time determination, great interest in this method has existed. Naturally occurring uranium isotopes 238 and 235 and thorium 232 decay radioactively, with known characteristic rates, to produce lead isotopes 206, 207 and 208, respectively. By precisely determining the ratios of these isotopes by means of the mass spectrometer, the time of formation of the rock sample in which the lead is found can be determined.

There are of course many complicating factors, including the existence of non-radiogenic lead, which may affect the accuracy of dating by this method, but in any case the method promises to be a powerful tool to evaluate chronologically and geochemically many geologic events.

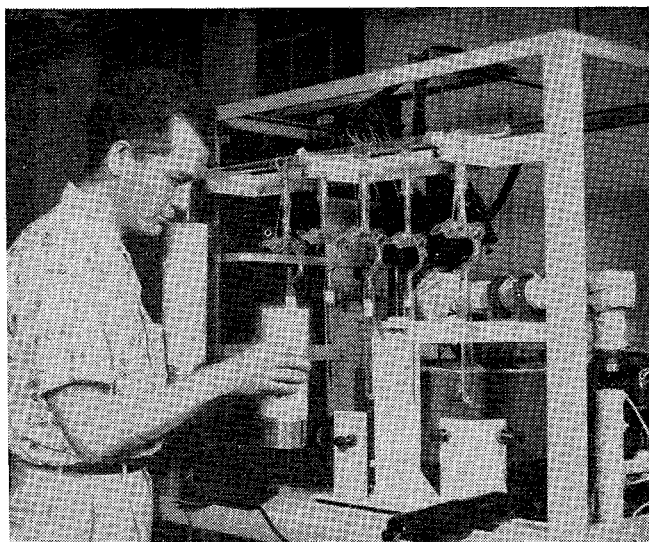
Lead isotope data

The scarcity of good lead isotope data is primarily due to the difficulty and expense in constructing specialized equipment and to the difficulty of the chemical techniques. In recent years, Claire C. Patterson, Senior Research Fellow, Professor Brown and their associates at Caltech have developed techniques whereby they are able to extract microgram quantities of lead from commonly occurring rocks and minerals. These lead samples can then be analyzed mass spectrometrically.

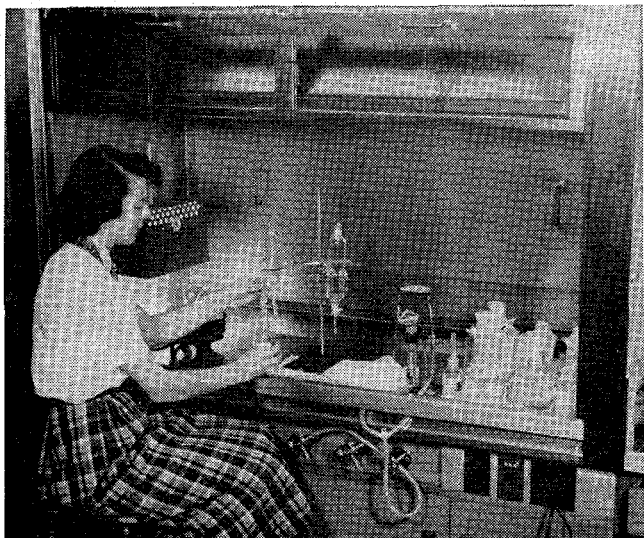
Most of the lead data now available are from lead ores whose origin is usually a complex one and do not represent the typical widely occurring rock formations. The development of these refined techniques for lead extraction from common rock types like granites and basalts represents an important step in increasing the usefulness of the lead method of dating. In addition, they promise more powerful methods for investigating many geochemical processes.

The extraction of such small quantities of lead from large rock samples introduces a serious problem of avoiding contamination with isotopically different lead.

Lead has become so commercially important and widespread that we virtually live in a cloud of lead. As a result, under normal conditions, the chemical steps taken



Sam Epstein freezes out a sample of carbon dioxide from the gas sample line of the mass spectrometer.



This lead-free laboratory has filtered air, lead-free paint, stainless steel work benches and fume hoods.

to extract the minute quantities of lead from the common type of rocks will introduce sufficient quantities of common lead to invalidate the subsequent isotopic determinations and time measurements.

To avoid such contamination, a special lead-free laboratory was constructed. The air entering the room is cleaned and filtered. The air pressure in the room is kept somewhat higher than the outside so that there is little danger of air diffusing into the room through small openings or while doors are being opened. The room is painted with lead-free paint and the work benches and fume hoods are constructed of stainless steel so that they may be washed with nitric acid and kept scrupulously clean at all times.

Recent work by Dr. Patterson has borne out that these precautions are indeed effective. He was able to extract minute quantities of lead of unique isotopic compositions from meteorites, with insignificant contamination with common lead, and as a result was able to assign the oldest experimentally determined age to the earth; namely, 4.6 billion years.

Non-radiogenic geology

Finally, facilities have been set up to do research in the field of non-radiogenic isotope geology. This field of endeavor consists of studying the isotopic composition of the lighter elements, such as oxygen and carbon, as they occur in nature.

In many cases the isotopic composition of these elements reflects the origin and history of the compounds in question because isotopic fractionation (a preferential separation of an isotope of an element relative to the other isotopes of that element) accompanies many chemical and physical processes.

Thus, for example, the isotopic composition of the oxygen of water and ice is affected by the cycles of evaporation and condensation; processes involved in

the formation of igneous, metamorphic and sedimentary rocks are in most cases accompanied by isotopic fractionation of the elements of which these rocks are composed.

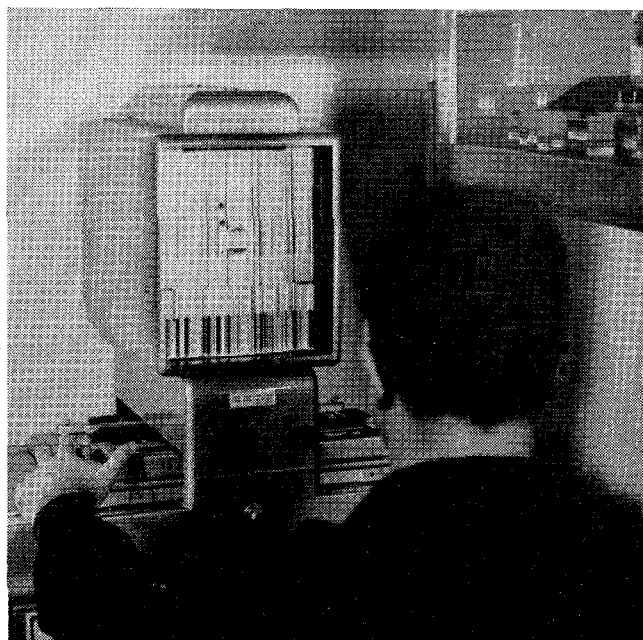
A modified Nier (60°, 6 inch) type of mass spectrometer for high precision isotope analyses and sample preparation apparatus developed by Professor Harold C. Urey and his associates at the University of Chicago have now been set up here and research in this field is now in progress.

Joint research

Drawn from many scientific disciplines, the members of the Division are actively pooling their training and interests in joint research where sound basic data have been limited or unavailable in the past. The availability of excellent equipment has given the cooperating workers the tools necessary to attack their problems more effectively.

The graduate and the undergraduate geology student have also become aware of the existence of a geochemistry group. The new two-year graduate course encompassing lectures dealing with subjects ranging from astronomy to biology includes many geochemical topics.

Undergraduate courses dealing with both theoretical geochemistry and laboratory experiments are now available, where the students have the opportunity to do a variety of experiments involving the use of most of the instruments discussed in this article. In most cases the student may not be able to do independent work with some of the more complex instruments and techniques, but in many cases the mere awareness of what these things can do should prove very useful in subsequent work involving either field or laboratory problems.



Arthur Chodos evaluates plates from emission spectrograph to analyze for chemical elements in rock sample.