

Nuclear Activation Analysis

Recent developments in the technique and its inherent attributes give promise of wide applicability.

Richard E. Wainerdi and Normand P. DuBeau

Man has always felt challenged by anything he can't accurately measure and describe. He feels a need to know what his surroundings, both near and remote, are made of. The relatively new technique of nuclear activation analysis may prove to be one means of satisfying this need.

Advances in activation analysis made during the past 5 years (1, 2) have brought a technique which, 25 years ago, had been little more than a curious laboratory phenomenon (3) to the threshold of becoming a valuable and widely available tool for elementary analysis. It is likely to be used for analyzing large numbers of samples of blood or soil, for surveying the Mohole, even for analyzing the surface of the moon. The advances made are the result of the research of many dedicated workers (1) in several parts of the world.

Basically, activation analysis is a method of making qualitative and quantitative elementary analysis by means of techniques of nuclear transmutation. When a material is irradiated by the nuclear particles produced in a nuclear reactor, particle accelerator, or other source, some of the atoms in the material interact with the bombarding particles and are converted into different isotopes of the same element or into isotopes of different elements, depending on the nature of the bombarded material and the bombarding particles. In many cases the isotopes produced are radioactive. If each induced radioactivity can be distinguished or separated from all the other radioactivities present, then the amount of each radioactivity is a measure of the quantity of the parent isotope present in the bombarded material.

The activating particles can be neutrons (4), protons (5), deuterons (6), tritons (7), helions (8), or even high-energy gamma photons (9). If the par-

ticle is of the proper type and energy, some of the atoms bombarded will undergo transmutation and become radioactive, emitting characteristic radiation that can be measured and identified. The radiation may be emitted either during the instant of bombardment or in the course of radioactive decay. In either case it is a useful analytical tool.

The nuclear processes chart (Fig. 1) is used to predict which isotope will result from a given nuclear transmutation. For instance, a target nucleus of mass M (square 6) will be converted to the next heavier isotope of the same element (square 7) after capture of a neutron. Similarly, a nuclear process which causes an alpha particle to be given off by a target nucleus will cause that nucleus to have an isotopic mass of $M-4$ and all the nuclear properties of the isotope in square 1.

At present, there is no type of analysis that can be made by the nuclear activation method that cannot also be made by some other method. On the other hand it can be shown that all the elementary analyses that can be made by other methods can, potentially at least, be made by nuclear activation analysis.

In this technique there is no interference from the matrix; thus, sample preparation and handling are greatly simplified and the analytical chemist is released from many of the routine tasks associated with other methods of elementary analysis. Independence of the form of the matrix also makes it possible to analyze samples in the solid, liquid, or gaseous state.

Activation analysis is an extremely sensitive method (10). With the present techniques, sensitivity of analysis is in the 0.01- to 0.0001-percent range. For amounts of material in the sub-microgram range, precisions of 5 percent have been reported (11). The

ultimate sensitivity, which can be calculated for all techniques, has yet to be reached for activation analysis. As we discuss later, when larger numbers of bombarding particles are available the sensitivity of the method will be increased; the increase in sensitivity will be proportionate to the increase in bombarding-particle flux.

Activation analysis, especially in its instrumental applications, is essentially a nondestructive technique. The number of atoms transmuted during bombardment to develop detectable radioactivity is so small as to be insignificant—perhaps a few million atoms per gram. Since usually no chemical analysis is required before activation, the reagent contamination problem, common to most methods of wet-chemical trace analysis, is eliminated.

Activation analysis can be very selective, especially when the analyst has access to a variety of subatomic particles and detection devices. It can also be quite general in its applicability. Thus far it has been used in determinations for 72 elements (12) with thermal neutron bombardment.

To date, Greenwood (13) has measured the characteristic spectra of the radiation emitted in the instant of neutron capture by 46 elements. It appears probable that each of the other elements will also have a unique gamma-ray spectrum in the instant of capture, which can be used analytically. Steele has listed (2) the nuclear reactions for 48 elements bombarded with neutrons of 14-Mev energy. Measurements for all the naturally occurring elements have been made by some variety of nuclear activation analysis.

Activation analysis can be very rapid. There are other techniques that may be as rapid, or even more rapid, in analysis for certain elements under certain conditions, but with these other techniques, if the sample conditions change even slightly, associated procedures, such as spectrographic analysis, may become difficult or even impossible. Since activation analysis is fast and releases the analytical chemist from sample preparation, its cost is comparatively low.

The speed so far attained can be greatly increased, and the cost greatly reduced, as new, quick, radiochemical procedures (14) and new instrumentation (15) are developed. For instance,

Dr. Wainerdi is associate dean of engineering and professor of engineering science at the Agricultural and Mechanical College of Texas, College Station. Mr. DuBeau is director of information and publications of the Texas Agricultural and Mechanical College System.

Table 1. Comparison of approximate experimental and calculated sensitivities*; mode of chemical separations. [After Fukai and Meinke (32)]

Element	Sensitivity (g)† (flux: 1×10^{12} n/cm ² sec)		Mode of chemical separation	Chemical yield (%)	Time required for separation (min)
	Experimental	Calculated‡			
Vanadium	2×10^{-9} § (10 min irradi.)	1.2×10^{-10} (satur.)	Cupferron-chloroform	90	~4
Arsenic	5×10^{-8} (10 hr irradi.)	9×10^{-10} (10 hr irradi.)	Co-ppt. with phosphomolybdate	~60	30
Molybdenum	5×10^{-7} (15 min irradi.)	3.2×10^{-8} (satur.)	(C ₆ H ₅) ₄ AsCl-chloroform	60	30
Tungsten	5×10^{-9} (10 hr irradi.)	1.2×10^{-9} (10 hr irradi.)	Thiocyanate-ethyl acetate	30	40
Rhenium	1×10^{-9} (10 hr irradi.)	1×10^{-9} (10 hr irradi.)	(C ₆ H ₅) ₄ AsCl-chloroform	70	30
Gold	5×10^{-10} (10 hr irradi.)	3×10^{-10} (10 hr irradi.)	Ethyl acetate	80	30

* For exact experimental conditions, see 32. † This sensitivity includes a cooling period of one half-life for long-lived isotopes of arsenic, tungsten, rhenium, and gold, and correction for chemical yield. ‡ See 33. § This value was estimated on the basis of counting with a gamma-scintillation well counter. || This time includes a waiting period of 15 minutes.

the feasibility of constructing a fully automatic system capable of analyzing up to 500 samples a day has recently been demonstrated (2). This system (Figs. 2 and 3) has a nuclear reactor as an activating source, a multichannel gamma-ray spectrometer, control and timing circuitry, and a high-speed digital computer.

Nuclear activation analysis is based on the work of Hevesy (3), Seaborg and Livingood (16), and Clark and Overman (17). It was used to a limited extent by other workers prior to World War II. With the development of high-flux reactors during and after World War II it became apparent that the technique of activation analysis offered promise as a means of detecting trace

elements, and techniques in which reactor-produced neutrons were used developed rapidly (18). Meinke (19) and Steele (20) have shown that the technique is highly useful in the analysis of macro-constituents; in their studies they used tritiated targets in neutron generators of comparatively low output that produced neutrons of 14-Mev energy by accelerating deuterium (19).

Method

Activation analysis is a method based on the application of nuclear data that have been accumulated over the years in many laboratories. These data include tables of nuclear cross section

(21), compilations of nuclear parameters (22), and information about the isotopes (23). There is some mathematics involved in the analytical application of these data, but computation can be quite simple and rapid. In automatic activation analysis systems these nuclear data are utilized automatically.

The literature indicates that when oxygen-16 is bombarded by neutrons of 14-Mev energy, the activation probability for the production of radioactive nitrogen-16 is 49×10^{-27} square centimeter.

Decay of the product nitrogen-16 is accompanied (in 80 percent of the disintegrations) by the evolution of characteristic gamma rays of approximately 6- to 7-Mev energy. The half life of the nitrogen-16 is a little more than 7 seconds. Thus, if one assumes a detection efficiency of 8 percent of the gamma rays evolved, the number of atoms of oxygen which will produce 1 measured gamma-ray pulse per second after exposure to bombardment by 10^9 fast neutrons per square centimeter per second for 70 seconds is 3.19×10^{17} atoms—less than 10 micrograms of oxygen.

The activation analyst makes use of these known characteristics of the isotopes to provide the best chance of achieving the specific activation desired and to lessen the extent to which other substances in the matrix, which are not of interest, are activated. Thus he uses activating particles of different kinds and energies in different situations.

Let us, for example, assume that one has a sample which may contain cobalt oxide in the molecular form, CoO. An exposure of the sample to bombardment by thermal neutrons will produce characteristic cobalt peaks on a gamma-ray spectrometer (at 1.17 and 1.33 Mev). Only cobalt produces peaks at both these energies.

In order to survey for other elements, the sample is activated in a flux of neutrons of 14-Mev energy for about 20 seconds. This is long enough to determine the characteristic spectrum of oxygen. If the spectrum is measured every 3 or 4 seconds it becomes evident that the induced radiation is decaying in intensity with a characteristic half life of a little over 7 seconds.

This procedure (described here in somewhat simplified form), coupled with measurement of the level of the flux in which the oxygen was bombarded and the activation probability [available from the literature (24)] for the formation of N¹⁶ from O¹⁶, can be

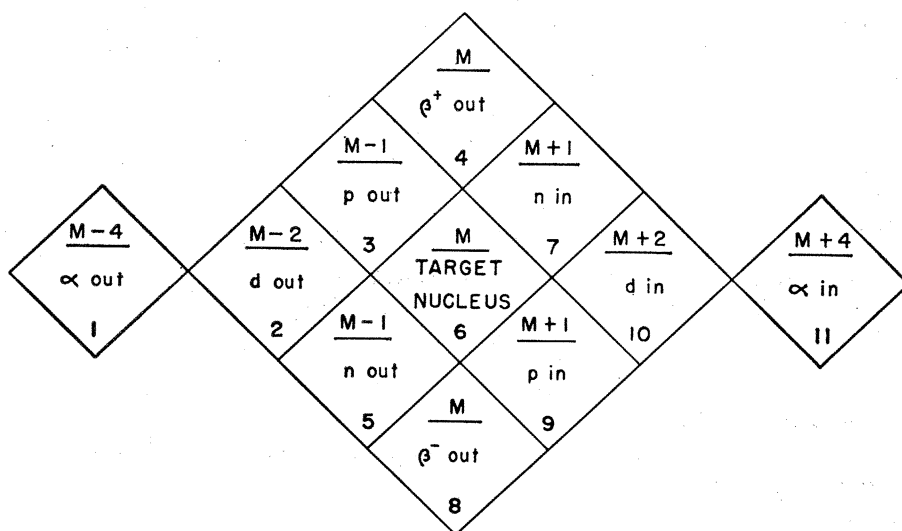
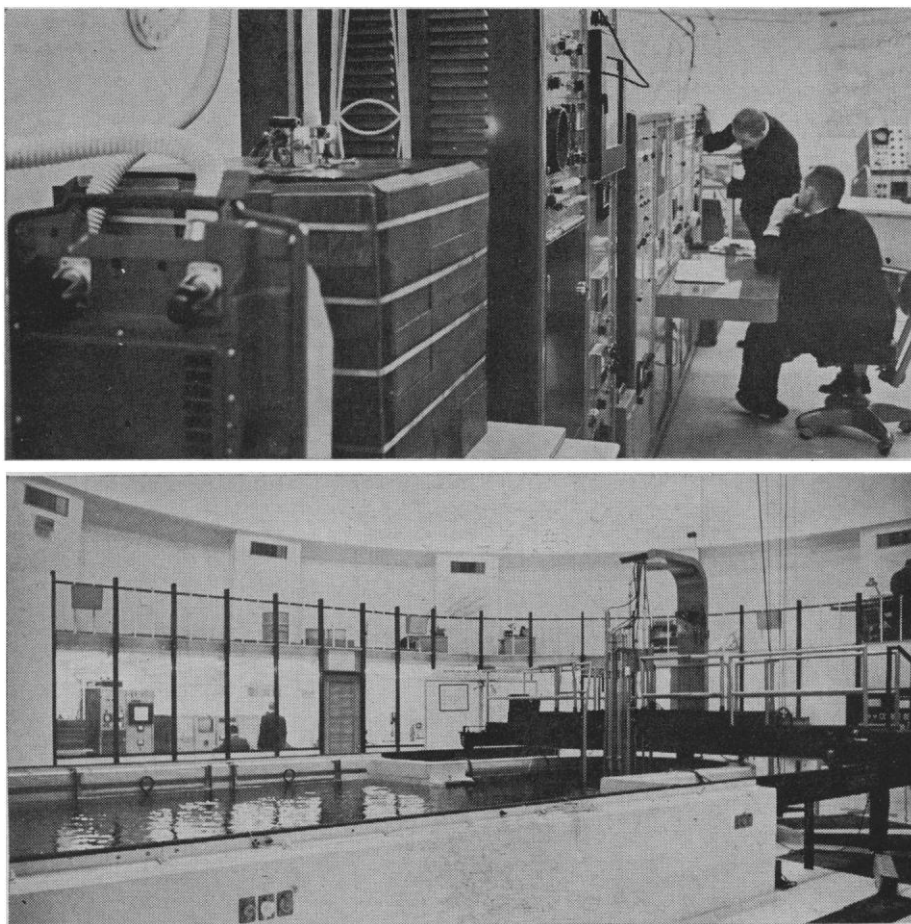


Fig. 1. Nuclear processes chart. The chart illustrates the results of various nuclear transmutations and guides the activation analyst in the selection of analytically useful reactions. The characteristics of the target atoms, bombarding flux, and transmutation product all must be considered in selecting the reaction desired.



Figs. 2 (top) and 3 (bottom). The automatic activation analysis system at Texas Agricultural and Mechanical College. Samples can be activated individually or in groups by means of the reactor shown in the foreground of Fig. 3; 2400 samples have been processed in 164 hours by this system.

used in the equation that follows to calculate the number N of atoms of oxygen which were bombarded—that is, to give a quantitative measurement of the oxygen present in the sample.

$$N = \frac{D}{\phi \sigma (1 - e^{-\lambda t})}$$

where D is the number of disintegrations per second after activation in a neutron flux ϕ (expressed in neutrons per second per square centimeter) for a time t ; σ is the activation probability that the material being bombarded will form the desired product; and $e^{-\lambda t}$ relates the decay constant λ of the radioactive product and the time t of the bombardment to the activity produced.

In the case described, when the oxygen atoms were irradiated, by neutrons of 14-Mev energy, some of the atoms captured neutrons and gave off protons to form N^{16} , which is radioactive, and which decays with a half life of 7 seconds.

In the case of cobalt (Co^{59} in its nonradioactive state), exposure to bom-

bardment by thermal neutrons causes many atoms to capture neutrons and to form the metastable isotope Co^{60m} ; this decays, with a half life of 10 minutes, to form Co^{60} , which has a half life of more than 5 years. Some of the Co^{60} atoms are formed and begin to decay immediately after irradiation, and it is this decay that can be measured and identified by characteristic gamma rays at energies of 1.17 and 1.33 million electron volts.

To find out how much cobalt is present, the analyst must determine the characteristics of the flux in which the material was activated—that is, find out how many neutrons of each energy impinged on the sample. Then it must be determined from the literature (23) what the energy-dependent activation probability for the production of Co^{60m} from Co^{59} is for that flux. Once the activation probability and the flux are known and the activation and decay times have been measured, the number of atoms of Co^{59} in the sample can be determined.

In the foregoing illustration, a simple

compound of two elements which have different half lives was considered: oxygen, with a half life of 7 seconds, and cobalt, with a longer half life.

The problem of distinguishing induced radioactivities can be approached in a number of ways. In what might be called “classical” activation analysis, the approach involves the complete radiochemical purification of the induced activities through complete separation of the desired radioactivity from all other radioactivities. Such a complete purification is exact, but it can be a tedious and strenuous task.

Another approach to this problem—a method not too complicated for relatively unskilled persons—involves the use of a gamma-ray spectrometry system in which radioactivities may be distinguished in terms of their gamma-ray energy. When necessary, this approach may be combined with simplified radiochemical procedures (25). When radiochemistry is not necessary, the approach is usually referred to as “instrumental” activation analysis.

Instrumental activation analysis can be used for most qualitative analyses, but it is difficult to make quantitative measurements of samples containing several elements by instrumental analysis unless some sort of sample preparation or postactivation treatment of the spectrometer data is devised to separate one gamma-ray activity from another. Data treatment can be accomplished by mathematical procedures, such as those illustrated in Fig. 4. This figure illustrates alternate methods for separating a composite gamma-ray spectrum into its components. This “unscrambling” can be done either mathematically or electronically, but the basic assumption—that the law of superposition applies—requires that the conditions under which these spectra are accumulated be such that the spectra actually do add linearly. This approach has been integrated into computer programs to preserve the simplicity of operation that is one of the method’s principal attractions (2, 15).

Choice of Procedures and Equipment

The choice of procedures and equipment for performing a given analysis is governed by what one wants to determine. To analyze a few samples in great detail, the analyst follows one approach, but if he is mainly interested in general analyses of several elements in large numbers of samples, he elects quite different procedures.

Table 2. Magnesium in high-alloy steels. [From Hoste, Bouten, and Adams (38)]

Sample	Certified concentration	Activation analysis measurements	No. of analyses
BCS steel 266 PMA: Co, 23.4%; Ni, 13.3%; Al, 7.95%; Cu, 3.3%; Ti, 0.1%; Fe, 49.7%	0.27 ± 0.012*	0.276 ± 0.005	9
BCS steel 233 PMA: Co, 23.7%; Ni, 11.2%; Al, 6.98%; Cu, 5.09%; Fe, 51.15%	0.235 ± 0.005	0.245 ± 0.017	12
NBS No. 155: Cr, 0.485%; W, 0.517%; Ni, 0.1%	1.25 ± 0.015 1.24 ± 0.01	1.24 ± 0.050	20
NBS No. 101d: Cr, 18.68%; Ni, 9.05%	0.739 ± 0.004	0.746 ± 0.021	16
BCS cast iron 149: S, 0.011%; P, 0.010%	0.023 ± 0.0018	0.025 ± 0.001	28

* Standard deviation computed from certificates.

If the goal is very detailed information about a small number of samples, one approach might be to divide the sample into several parts and expose the various pieces to bombardment by different activating particles, then to separate the active products chemically to the point where simple beta- and gamma-ray spectrometers can provide analytical information.

To process complex samples with such care and precision would involve an investment in dollars, skilled analysts, and time which is incompatible with large-scale surveying (26). A typical intricate radiochemical separation can cost thousands of dollars. The cost of a typical instrumental analysis is in the range of tens of dollars or less. (Admittedly, only certain elements in certain matrices can be analyzed instrumentally.) In overlapping cases, where both methods are applicable, the data can be compared to determine the relative precision of the methods.

It has been suggested (27) that a computer program be set up to guide the analyst in selecting the best possible

conditions for activation analysis on the basis of the equipment and facilities available and the analytical situation in question. At present, such selection requires considerable effort and skill on the part of the analyst.

For analytic situations requiring great patience and precision, radiochemical separation procedures are at present often the approach of choice. It appears that anticipated improvements in instrumentation will make it possible to analyze more complex samples by automated systems. At Oak Ridge National Laboratory, 57 elements were identified and measured in a single sample through a combination of instrumental and radiochemical procedures (28). Much ingenuity will be required to equal this performance through purely instrumental procedures, but the promise of such an eventuality, or even a reasonable approach to it, is enough to inspire the workers in this field to considerable further effort.

Automated activation analysis can be used at present for mass analyses for specific elements in suitable matrices.

Samples for an automated system might include hydrocarbons, sea water, blood, drill cuttings from the Mohole or from oil wells, soils, transistor materials, and metal alloys. In each case the type of activating particles and the way in which the details of the procedure would be programmed would be governed by the specific elements of interest in the matrix.

To illustrate, let us suppose that an analysis of sea water for trace elements is desired. The sodium and chlorine macro-constituents will interfere with the analysis if thermal neutron activation is used. Any of at least four alternate procedures can be used.

1) The sodium and chlorine could be removed chemically prior to thermal neutron activation, but this might also remove some of the trace elements as well, or even add some.

2) A chemical separation of sodium and chlorine from the trace elements could be performed after thermal neutron activation. Such a separation would be less likely to upset the total composition of the sample than procedure 1, since the separation procedures could be monitored radiochemically.

3) The sea water could be activated with particles that do not make sodium and chlorine very radioactive—for instance, neutrons of 14-Mev energy. The activating particle selected would have to be of a type that would easily activate each trace element of interest.

4) An electronic system could be used to resolve the sodium and chlorine spectra from the composite spectrum obtained during accumulation (2). Such a system could be incorporated into a conventional multichannel gamma-ray spectrometer for this purpose.

If, on the other hand, the goal is to

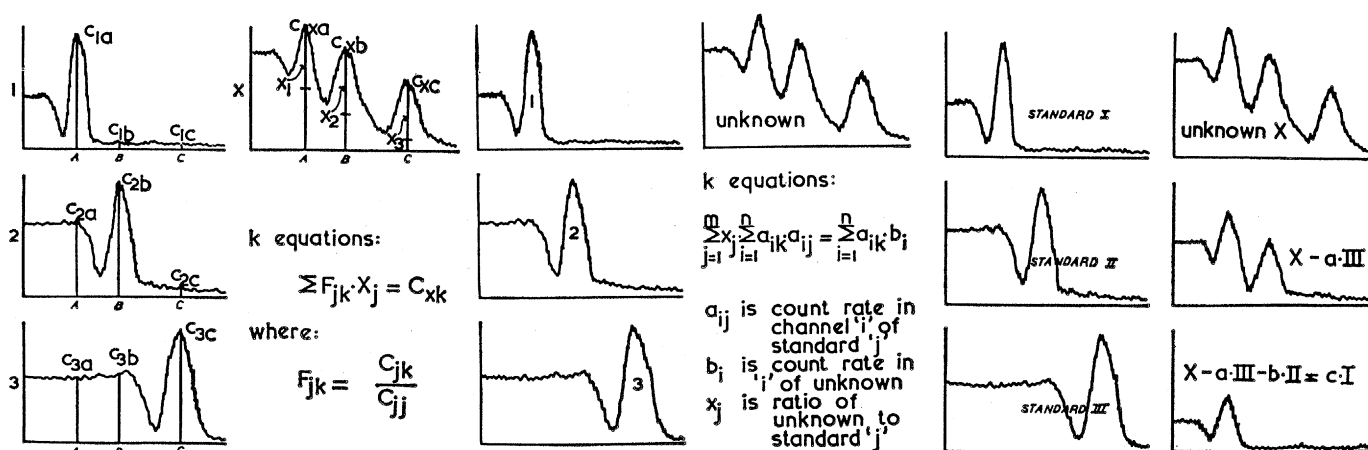


Fig. 4. Three illustrations of the law of superposition as it applies to gamma-ray spectra collected under carefully controlled conditions.

determine how much sodium, chlorine, and other macro-constituents are present in the sea water samples, activation with thermal neutrons, without preparatory chemistry, will be the method of choice.

Applications of Activation Analysis

Just as the development of computers opened vast new areas of mathematical exploration, so activation analysis has made possible the exploration of many new problems of wide interest and importance. More than 2000 applications are listed in the literature (29).

The several specific applications of activation analysis given here were selected to illustrate various aspects of radiochemical, combined radiochemical and instrumental, and instrumental activation analysis.

The use of nuclear activation analysis for determining the oxygen in silicon by irradiation with alpha particles in a

cyclotron was recently described by Nozaki, Tanaka, Furukawa, and Saito (30). These investigators made use of the $O^{16}(\alpha, pn)F^{18}$ reaction, and succeeded in determining the presence of as small an amount of oxygen as 0.3 part per million.

Their approach involved extensive radiochemical processing. After irradiation, the surface of the activated silicon was washed with dilute hydrofluoric acid containing a small amount of nitric acid to remove the surface contamination. The silicon was then pulverized and added, in small portions, to fused potassium hydroxide (25 g) containing sodium fluoride (10 mg) and disodium hydrogen phosphate (5 mg) as a carrier. Sodium nitrate (0.5 g) was then added to convert radioactive phosphorus produced by $Si^{28}(\alpha, p)P^{32}$ and $Si^{30}(\alpha, pn)P^{32}$ reactions into the phosphate, which served as the internal flux monitor of the α -particle beam. The cake was then dissolved in sulfuric acid (200 ml), and the fluoride was distilled

Table 3. Analysis of lyophilized blood sera by computer-coupled activation analysis. [From Fite, Steele, and Wainerdi (2)]

Sample No.	Sodium (%)	Chlorine (%)	Magnesium (%)
1	2.67	4.08	0.38
1	2.91	4.36	0.78
2	2.63	4.34	0.51
2	2.16	3.80	0.24
3	2.62	4.32	0.48
3	2.88	4.40	0.43
4	2.06	3.86	0.20
4	2.94	4.39	0.48
6	2.21	4.19	0.24
6	2.91	4.25	0.33
6	3.04	4.20	0.26

at 150° to 160°C; the distillate was caught by aqueous ammonia. The fluoride was precipitated as calcium fluoride, together with calcium carbonate, then filtered off, washed, and dried. The radioactivity was then measured. The time required for the separation was about 2 hours, and the overall recovery of fluoride was about 80 percent.

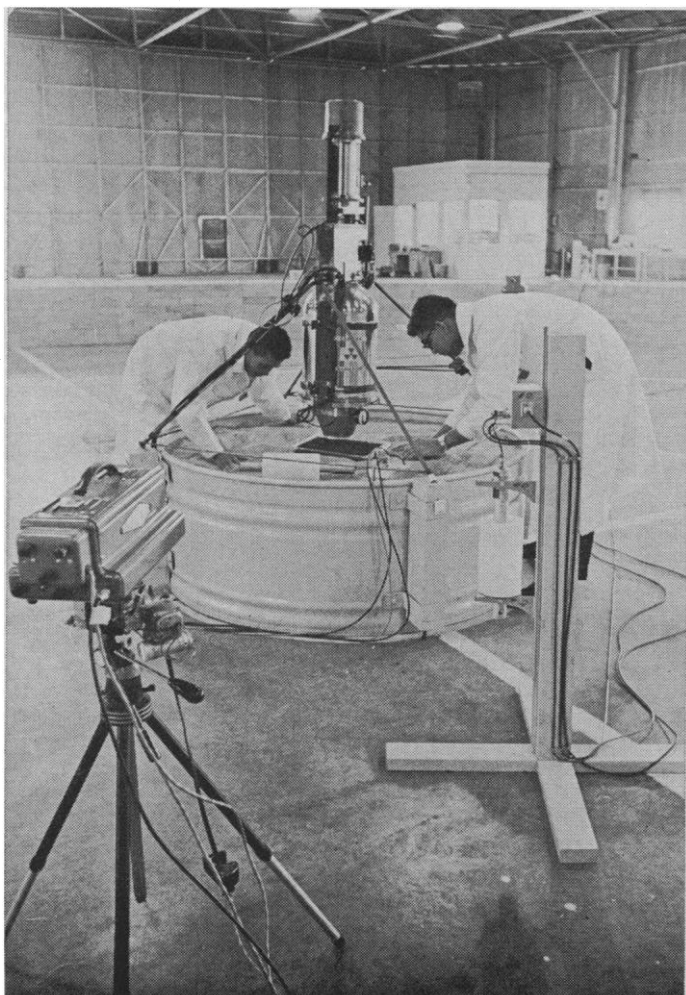


Fig. 5 (left). A tray of material such as that postulated for the surface of the moon is placed in position for neutron bombardment and subsequent gamma-ray spectral analysis. Fig. 6 (right). The gamma-ray spectra resulting from bombardment of the "moon material" of Fig. 5 with neutrons of 14-Mev energy is recorded remotely for subsequent data reduction by computer.

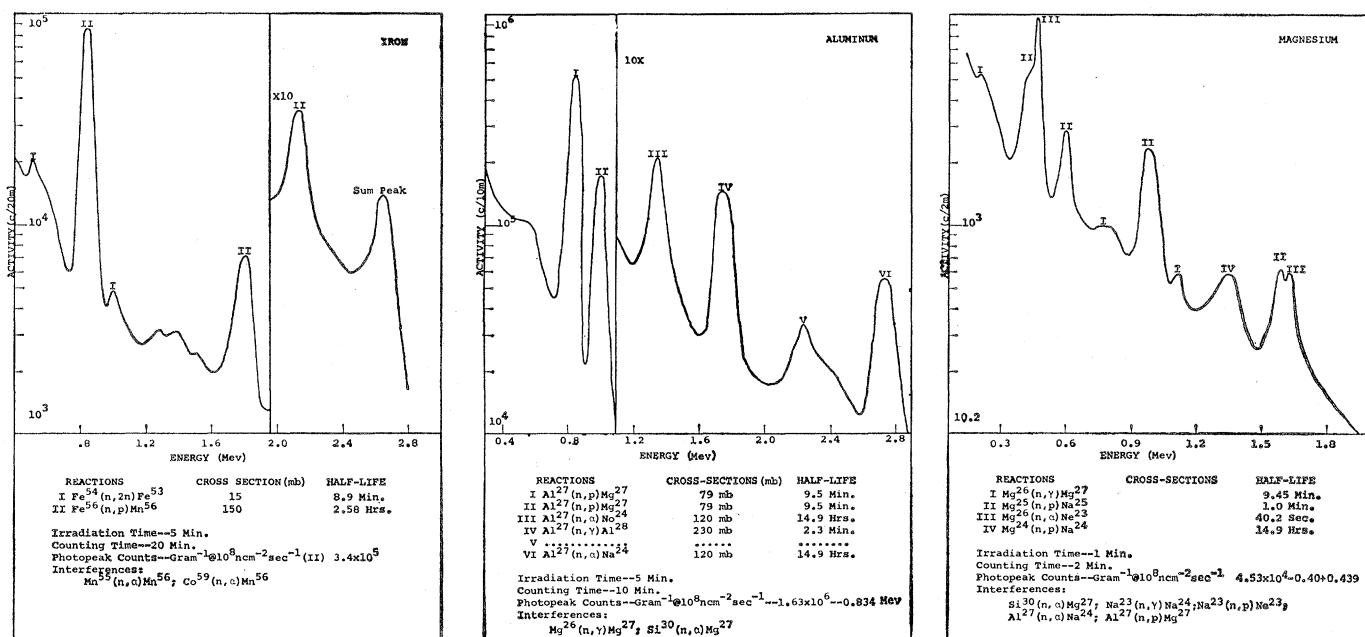


Fig. 7. The gamma-ray spectrum of iron (left), aluminum (middle), and magnesium (right) which results from bombardment with neutrons of 14-Mev energy.

Kaiser and Meinke have shown (31) that trace cobalt, present in microgram amounts from vitamin B₁₂, can be analyzed by rapid activation analysis in rat kidney as well as in vitamin preparations. A 15-minute radiochemical separation procedure, coupled with gamma-ray spectrometry, permitted utilization of the Co^{60m} isotope, with a half life of 10 minutes. With their procedure, the lower limit of detection at a thermal-neutron flux of 10¹² neutrons per square centimeter per second was about 5×10^{-8} gram of cobalt.

Fukai and Meinke (32) demonstrated the analysis of marine organisms for vanadium, arsenic, molybdenum, tungsten, rhenium, and gold by nuclear activation analysis. Radiochemical procedures coupled with gamma-ray measurements resulted in unambiguous determination of submicrogram amounts of these elements in the ashes of several different types of marine organisms. Table 1 (adapted from their work) shows a comparison of approximate experimental and calculated sensitivities and the mode of chemical separations they employed.

Schindewolf and Wahlgren (33, 34) applied nuclear activation analysis to determine the rhodium, silver, and indium content of five chondritic meteorites. The averages were 0.186 and 0.094 part per million for rhodium and silver, respectively, and the upper limit for the indium content was 0.001 part per million. The values for rho-

dium and silver were in substantial agreement with the estimates of Suess and Urey (35), but the values for indium were lower by a factor of 100 or more than had been postulated.

In a similar matrix, Morris and Killick (36) determined the silver and thallium content of rocks by neutron activation analysis. Utilizing the Harwell pile BEPO as an activating source and radiochemical separations based on precipitation, electrodeposition, and solvent extraction, they analyzed samples

containing as little as 0.03 part of silver and 0.04 part of thallium per million. The rocks they examined in this instance included two international standards, G1 and W1, and samples from the Inch Mass, Aberdeenshire, Scotland. The same authors used nuclear activation analysis to determine ultra-micro quantities of silver in platinum sponge (37). The platinum sponge was irradiated in the Harwell pile BEPO for a month; the assay was made through measurement of the radioactive isotopes

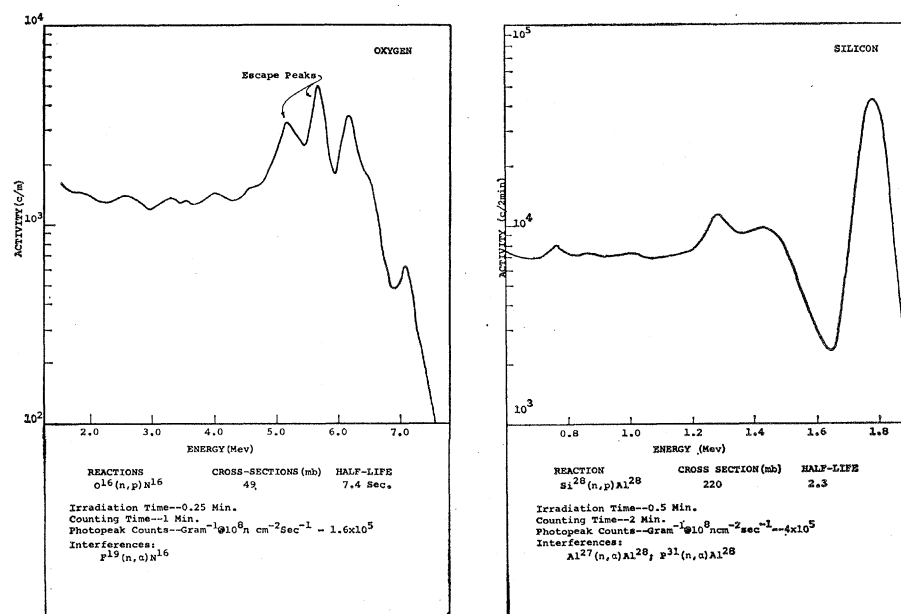


Fig. 8. The gamma-ray spectrum of oxygen (left) and silicon (right) which results from bombardment with neutrons of 14-Mev energy.

$\text{Ag}^{110\text{m}}$ and Ag^{110} . Radiochemical separation after the addition of a carrier was based mainly on precipitations of silver as chloride, sulfide, oxide, and iodate, and on electrolytic deposition. A gamma-ray scintillation counter was used. The precision of the analyses was better than ± 10 percent. This procedure is applicable for platinum containing as little as 0.02 part of silver per million.

Hoste, Bouten, and Adams (38) demonstrated the applicability of instrumental activation analysis for determining minor constituents (0.001 to 1 percent) in steels. The results they obtained for the analyses of magnesium in high-alloy steels are presented in Table 2.

Fite, Steele, and I recently demonstrated (2) the use of a computer-coupled automatic activation analysis system for the nondestructive determination of sodium, chlorine, and magnesium in lyophilized blood sera. The results of these analyses are presented in Table 3. Irradiation of duplicate samples was carried out in the pneumatic tube of the Texas A. & M. research reactor for 3 and 10 minutes, respectively. The neutron flux was measured as 4×10^{10} neutrons per square centimeter per second, and the samples were counted immediately after irradiation (20 seconds later). The analytical results of Table 3 were obtained by computer unsorting of the complex gamma-ray spectra resulting from radioactivation, with all of the necessary times, measurements, and basic nuclear data supplied automatically by, or to, a pre-programmed IBM 709 computer.

The feasibility of using nuclear activation analysis for determining the composition of the surface of the moon is now being studied in several laboratories (2, 39). It is postulated that the following elements occur on the surface of the moon (40): hydrogen, oxygen, magnesium, aluminum, silicon, carbon, nitrogen, sodium, potassium, calcium, iron, nickel, phosphorus, sulfur, chlorine, vanadium, barium, strontium, cobalt, and lithium. It is, of course, desirable to analyze the surface of the moon for all these materials, but this would

probably require a greater variety of activating particles than could be produced by machines transported to the moon in the near future. The use of 14-Mev and thermalized neutrons which could be produced by a relatively small moon probe (a prototype is shown in Figs. 5 and 6) offers considerable promise for determining the presence and amounts of iron, aluminum, magnesium, oxygen, and silicon—probably the main constituents of the lunar crust—as well as other elements of interest. Figures 7 and 8 show the gamma spectra which result from bombardment of iron, aluminum, magnesium, oxygen, and silicon with neutrons of 14-Mev energy. A bombardment of 5×10^5 neutrons per square centimeter per second for 5 minutes, followed by a 30-minute period of counting with a sodium iodide scintillation detector (3 by 3 in.), will make possible qualitative and quantitative analyses for at least the five probable macro-constituents and will probably yield considerably more information than that (2).

Conclusion

Nuclear activation analysis—whether with instrumental or radiochemical procedures, manual or automatic systems—seems destined for an important place in analytic chemistry. Experience will determine the best choice of activating particles and the best detectors, or it may even lead to the development of entirely new techniques. But it seems evident that nuclear activation analysis will increasingly satisfy the need for precise, sensitive, and rapid elementary analysis, and will do so at relatively low cost.

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