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THE LESS FAMILIAR ELEMENTS IN THE ATOMIC ENERGY PROGRAM

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THE LESS FAMILIAR ELEMENTS IN THE ATOMIC ENERGY PROGRAM

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Many factors are responsible for the rapid growth of analytical chemistry during the past ten to fifteen years. One of the most important of these factors is the greatly increased significance to present day chemistry of many elements that in the recent past were principally chemical curiosities. Since the Atomic Energy program is concerned with many such elements, it is of interest to discuss the cause for this concern and the resulting effect on the field of analytical chemistry. A number of pertinent analytical research problems at Hanford will be cited as examples.

For the purpose of this discussion, the elements in the shaded areas of Figure 1

(Here Fig. 1)

were quite arbitrarily selected as the "unfamiliar" ones. Except for a large group in the rare earth and heavy metal regions, they are fairly randomly scattered in the periodic table. In considering the relation of the Atomic Energy program to these elements, it is most obvious that two of these elements, uranium and plutonium, are industrial products of the program. This, however, is a minor factor even with due consideration to the size of the overall project.

A much more significant consideration relates to the fission products. You will recognize that there are four principal mechanisms whereby nuclear transformations occur: spontaneous beta or alpha radioactivity, in which a transformation of one or two atomic numbers occurs; spallation, whereby a small part of a nucleus is dislodged under bombardment with high energy particles; fusion, whereby the nuclei of

[illegible]

FIGURE 1

150

of two light atoms combine to form a heavier one; and fission, in which a heavy nucleus splits into two more or less equal fragments.

Nuclear fission is, of course, a key phenomenon in the Atomic Energy program, since tremendous quantities of energy are liberated in the fission of nuclei. The dotted areas in Figure 2 show the distribution of fission products obtained by the fission

(Here Fig. 2)

of uranium or plutonium. Notice that there are two mass groupings slightly separated from each other and that these are spread across the middle of the periodic table and include the rare earth group and many of the other little known elements that occur in that region. The Atomic Energy program is concerned with the chemistry and the determination of these elements because they represent impurities that have been created by nuclear fission and which must be removed from the product with which they are associated. The fission product elements in nearly all cases have higher than normal atomic weights and, as a consequence, are strongly radioactive. As a result, minute traces present serious beta and gamma radiation hazards, and it is necessary that they be removed with almost absolute completeness. Thus, the analytical chemistry of these many fission products is of concern in the Atomic Energy program, even though the nuclear reaction that produces them is independent of the chemical properties of the fissioning element.

The entire subject of nuclear chemistry is a fascinating one that has grown tremendously over the past 15 years as a result of the discovery of nuclear fission and the development of many new techniques for studying the nucleus. The properties of atomic nuclei are being interpreted in terms of energy levels which are evaluated in part through a study of the energy spectrum of alpha, beta, and gamma emissions; the situation is analogous to the evaluation of energy levels of the atoms by means of the emission spectra obtained upon excitation. Correlations between the half-life, the

[illegible]
$$F_{10} = 2$$

energy of emitted radiation, and the atomic numbers are being established with sufficient certainty to allow prediction of the properties of yet undiscovered isotopes. There are even magic numbers associated with nuclear composition. Nuclei containing 50, 82, or 126 neutrons are known to be exceptionally stable with regard to nuclear properties. This situation bears a striking resemblance to its counterpart in the structure of the atom, whereby completed shells containing 2, 8, 18, etc., electrons are extremely stable chemically. In effect, there is being developed a new periodic table based entirely on nuclear properties and without consideration of chemical properties. The entire situation is strikingly reminiscent of the development of the atomic periodic table by Mendeleeff a little less than 100 years ago. It is the concern with these two periodic tables that tends so greatly to broaden the field of chemistry, because study in the nuclear field requires selection of materials on the basis of one set of properties, whereas the chemical handling of these materials must be carried out according to another set.

The second factor that tends to make the Atomic Energy program concerned with a large number of heretofore less familiar elements relates to the nuclear cross section. It would be well to digress for a moment to discuss the concept of cross section, since it promises to become increasingly important to the field of analytical chemistry.

In a nuclear pile, neutrons are liberated upon fission of an atom of plutonium or uranium-235. These liberated particles form, in effect, a neutron atmosphere in the pile. They move in a manner similar to gas molecules and are eventually captured by collision with nuclei. The chain reaction requires that sufficient neutrons be captured by uranium-235 to cause fission and thus yield a continuing supply of neutrons; other neutrons are captured by a nucleus, thereby causing a unit increase in mass number.

Neutrons liberated during fission have extremely high energies. Capture by an atomic nucleus will occur only if the neutron makes a direct hit, and in this case, the

6732-6

reactive cross section of the nucleus is almost equal to the actual physical cross section of the nucleus, which is very small. The majority of the neutrons, however, lose their excess kinetic energy and are reduced to velocities corresponding to the normal Maxwellian distribution at the ambient temperature. In this case there is a strong tendency for the neutrons to be captured by a nucleus, and as a consequence, the effective cross section of the nucleus is many times bigger. In special cases in which the energy of the neutron is in resonance with the nucleus, this effect may be extremely large.

The unit for nuclear cross section is the "barn", which corresponds on the C.G.S. scale to 10^{-24} square centimeters. The story goes that during the early days of the project several physicists at Los Alamos who were engaged in the measurements of cross sections found one of exceptionally high value and commented, in effect, that it was as big as a barn. The uniqueness of the term and the need for coded terms at that time has resulted in the acceptance of the term. The circles in Figure 3 show the variation in

(Here Fig. 3)

cross section for slow neutrons. Many values are less than one barn, but in other cases, they are tens of thousands of barns. In specific cases where resonance capture occurs, the cross section will be many times larger and in a few cases may approach an area as big as one square Angstrom, which corresponds to atomic dimensions. In terms of our solar system, this would correspond to a situation in which a moving mass passing the solar system tangent to the orbit of Pluto would be absorbed by the sun.

The importance of cross section considerations in the Atomic Energy program is extremely great in that it is necessary to employ materials in a pile that will not consume neutrons by virtue of large cross sections. Thus, the selection of structural materials, containers, and cooling media must be based principally on this consideration. Similarly, the impurities in these materials and in the uranium fuel must be

6732-7

SLOW NEUTRON CROSS - SECTIONS

CROSS-SECTION FOR
SLOW NEUTRONS

○ — < 1
○ — 10
○ — 50
○ — 10²
○ — 10³
○ — 10⁴

¹ H									² He
³ Li	⁴ Be	⁵ B	⁶ C	⁷ N	⁸ O	⁹ F		¹⁰ Ne	
¹¹ Na	¹² Mg	¹³ Al	¹⁴ Si	¹⁵ P	¹⁶ S	¹⁷ Cl		¹⁸ Ar	
¹⁹ K	²⁰ Ca	²¹ Sc	²² Ti	²³ V	²⁴ Cr	²⁵ Mn		²⁶ Fe	²⁷ Co
²⁹ Cu	³⁰ Zn	³¹ Ga	³² Ge	³³ As	³⁴ Se	³⁵ Br		³⁶ Kr	
³⁷ Rb	³⁸ Sr	³⁹ Y	⁴⁰ Zr	⁴¹ Nb	⁴² Mo	⁴³ Tc		⁴⁴ Ru	⁴⁵ Rh
⁴⁷ Ag	⁴⁸ Cd	⁴⁹ In	⁵⁰ Sn	⁵¹ Sb	⁵² Te	⁵³ I		⁵⁴ Xe	
⁵⁵ Cs	⁵⁶ Ba	⁵⁷ La	⁵⁸ Ce	⁵⁹ Pr	⁶⁰ Nd	⁶¹ Pm	⁶² Sm	⁶³ Eu	⁶⁴ Gd
			⁷² Hf	⁷³ Ta	⁷⁴ W	⁷⁵ Re	⁷⁶ Os	⁷⁷ Ir	⁷⁸ Pt
⁷⁹ Au	⁸⁰ Hg	⁸¹ Tl	⁸² Pb	⁸³ Bi	⁸⁴ Po	⁸⁵ At		⁸⁶ Rn	
⁸⁷ Fo	⁸⁸ Fr	⁸⁹ Ac	⁹⁰ Th	⁹¹ Pa	⁹² U	⁹³ Np	⁹⁴ Pu	⁹⁵ Am	⁹⁶ Cm
								⁹⁷ Bk	⁹⁸ Cf

FIGURE 3

FIG. 3

67302-8

controlled on this same basis. As a result, the elements of concern and of utility are determined on the basis of nuclear properties -- a situation which brings many unfamiliar elements into prominence. We have seen that the existence of fission products presents a parallel situation.

Turning, then, from the cause for concern with these less common elements, it is of interest to consider the effect. First and foremost is the emphasis that has been placed on analytical chemistry, an emphasis that was necessary to develop the tools required to advance the project. Unfamiliar elements appeared in great number, in unfamiliar combinations, and frequently, in extremely low concentrations. The high activity levels and attendant radiation hazard of many materials imposed severe limitations in analysis and thus placed a premium on new techniques that were more rapid and that allowed remote operation.

The result has been the establishment of strong analytical research and development programs within the project and the employment of many highly capable chemists who continue to apply their abilities in the field of analytical chemistry. This has had a decided influence on the colleges and universities of the country, as evidenced by the establishment and strengthening of analytical departments in so many cases. The availability of analytical chemists in greater number, the availability of new techniques that they have developed, and the realization of the need and place of analytical chemistry has generated a momentum that has been more and more recognized by all industries. The strength and position of analytical research groups in industry and the tremendous growth of the Analytical Division of the ACS are ample evidences of this situation.

The second effect of the project on the field of analytical chemistry is the introduction of radiochemistry as a major analytical technique. The availability of

radioactive tracers in great abundance and variety and the development of techniques and equipment for measuring activity promise to revolutionize research and development approaches in many fields of science. By the first of 1953 the Atomic Energy Commission had made over 32,000 shipments of radioactive isotopes to various institutions and laboratories. The greatest use is in the fields of biology and organic chemistry, where tracer technique provides a unique method for establishing chemical and physiological mechanisms. Tracer techniques in other fields have been picked up more slowly but are increasing rapidly in number. Many unique examples might be cited, such as study of the diffusion of atoms within a solid, measurement of vapor pressure of metals, or the evaluation of engine wear by determining the quantity of radioactive isotopes worn from the cylinders and introduced into the oil. The main value of tracer technique is, of course, that a radioisotope is chemically identical with the stable isotope and that techniques for determining the former are so extremely sensitive; with a short-lived radioisotope such as sodium-24, it is possible to detect as little as 10^{-19} grams.

Another promising aspect of radiochemistry is that of activation analysis whereby a material is exposed to a neutron flux to induce activation of some molecules as a result of neutron absorption. By this means extremely minute traces may be partially activated so that counting techniques may be employed to detect their presence.

A third manner in which the Atomic Energy project has greatly influenced analytical chemistry is through the development of analytical instruments in great variety and in great number. The need for determining many elements, the emphasis on micro analysis, the need for remote handling, and the many requirements for isotopic analysis lead directly and naturally to the employment of physical techniques for analytical purposes. The conventional wet methods are slow and in most cases require macro samples; in addition, they are subject to many interferences which all too frequently in the past have been neglected or ignored. On the other hand, the availability of a

6732-10

wide variety of analytical techniques based on physical properties affords the greatest opportunity for effecting rapid analyses and avoiding the slow and cumbersome chemical separation steps.

We have spoken of the relation between the Atomic Energy program and the field of analytical chemistry and the influence of the project on the latter. It will be of interest to cite some examples of recent developments from our analytical research effort at Hanford. The investigation and application of coulometric titrations is one such item. This technique is one in which an electric current serves, in effect, as the reagent in the titration of a solution. It is necessary that 100% current efficiency be obtained in the electrode reaction if the stoichiometric relationship between the Faraday and the chemical equivalent is to be applied. A specific example is that of the titration of U(IV) to U(VI). The reactions involved are summarized in Figure 4.

(Here Fig. 4)

It is not possible to conduct the titration by direct anodic oxidation of the U(IV); as the reaction approaches the endpoint, the concentration of the tetravalent uranium becomes so small that 100% current efficiency cannot be expected. As a result, it is necessary to add bromide in high concentration to the sample solution. One hundred percent current efficiency is obtained in the anodic oxidation of bromide to free bromine. Since the latter does not effectively oxidize U(IV), it is necessary to employ an electron carrier in the system. Ferrous iron is effective for this purpose, and the overall reactions are as indicated: bromine oxidizes ferrous iron, and the ferric iron so obtained oxidizes the uranium. The net effect is that the Faraday relationship holds for the oxidation of U(IV) to (VI).

The endpoint may be detected by any convenient method. The potentiometric technique, employing a separate pair of indicator electrodes, is most applicable. Figure 5 indicates situations that may exist in the potentiometric endpoint detection.

(Here Fig. 5)

6732-11

COULOMETRIC URANIUM REACTIONS

FIGURE 4

6732-12

FIGURE 2

6732-13

If we consider the central solid curve to represent the equilibrium potential during the course of the titration, and if we consider the upper dotted line to represent the instantaneous measured potential, it is apparent that the measured value leads the true potential. In this case the endpoint is indicated before it has truly been attained, and there is thus no danger of overshooting the endpoint. This situation prevails in the above titration of uranium. In other cases, however, the measured potential may lag the equilibrium value, and the danger of overshoot is present. A convenient method for handling this situation has been developed by Carson (1) and is depicted in Figure 6.

(Here Fig. 6)

A sidearm attachment to the titration cell serves to hold back a small part of the sample solution while the major portion is titrated to a first endpoint, which is beyond the equivalence point. The withheld portion is then mixed with the bulk of the solution, and the process is repeated. In this manner the endpoint is approached stepwise so that the true equivalence point is attained.

Coulometric titrations have several inherent advantages. Currents may be measured in small quantities and with extreme precision so that highly precise micro determinations are indicated; the method is absolute in that it is standardized by electrical means and requires no standard liquid titrants; no dilution of the sample occurs during the process of titration; and remote automatic control is quite feasible. Our research effort at Hanford has led to the development of a completely automatic unit that performs the titration, anticipates the endpoint, and shuts off the instrument at the close of the titration (2). Techniques have been developed and described by Carson to allow the determination of uranium (3), plutonium, acid (4), and oxine (5), and to standardize various oxidizing and reducing agents. The quality of analyses obtained is indicated by the following data concerning the coulometric titration of uranium: in 2 ml of solution, 10 mg of uranium may be determined with ± 0.03 mg, and 5 ug of uranium may be determined within ± 0.3 ug.

6732 14

FIGURE 6

6932-15

A second technique, and one that promises to be of extreme value in the field of radio-chemistry, is that of gamma-ray scintillation spectrometry. The technique is in many ways analogous to that of emission spectrometry, the essential difference being that the latter breaks up visible and ultraviolet radiations into its spectrum, whereas the former breaks up gamma radiation into its spectrum. The approach in the two cases is somewhat different in that the gamma rays are not physically separated into a spectrum. The instrument employs a scintillation crystal of sodium iodide which has been activated with thallium, and upon which the gamma photons are allowed to fall. The gamma energy is absorbed and partly converted to visible radiation, which is picked up by a photomultiplier tube and amplified to yield an electrical pulse. Since there is a direct relationship between the gamma energy and the intensity of the visible scintillation, it follows that the amplitude of the pulse is in direct relation to the gamma energy. The pulses are segregated according to amplitude, thus providing a measure of the original gamma energy distribution. Figure 7 is an example of such a gamma energy distribution.

(Here Fig. 7)

It is apparent that the technique permits the detection and determination of a single gamma emitter in a mixture without resort to time consuming chemical separations. The subject is treated in detail by Connally and Leboeuf (8).

There is one principal difficulty that interferes with application of the technique, especially in the lower energy regions. This is evidenced by the increasingly greater background at lower energies in Figure 7. This interference results from Compton interaction between the gamma photon and the crystal, and can be explained by consideration of Figure 8.

(Here Fig. 8)

6035.16

FIGURE 7

Fig. 7

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FIGURE 8

GAMMA INTERACTIONS IN SCINTILLATION CRYSTALS

FIG. 8

6737 18

The sketch designated A in that figure describes a total interaction between the gamma ray and the crystal. In this case the former is completely destroyed and its energy is transmitted to a fast moving electron which is slowed down by interaction with atoms of the crystal. These atoms, upon returning to the normal energy states, emit the visible radiation which is picked up by the photocathode. In Sketch B the Compton interaction is described. In this case the gamma photon loses only part of its energy to an electron, and the remaining part continues on an altered path and may emerge from the crystal. Since the excited electron has not acquired the full energy of the gamma photon, the visible emission resulting from its travel is indicative of lower energy and yields an electrical pulse of low amplitude. One method under investigation for reducing this effect is that of employing a guard crystal. This is described in Sketch C. In this case the secondary gamma ray must travel through the guard crystal, where it has a high probability of being absorbed. When this situation does occur, scintillations occur simultaneously in the central and in the guard crystals. The photomultiplier tubes, viewing each crystal separately, both receive an impulse, and the effect is cancelled because they are connected by an anti-coincidence circuit. The net effect of the use of a preliminary model of this type is illustrated in Figure 9, which shows the spectrum of a sample having a single gamma emission.

(Here Fig. 9)

The upper curve shows the high Compton background in the absence of a guard crystal, and the lower curve shows the benefits gained by its use. The subject is described more completely by Connally (7).

A natural sequel to consideration and effort on the subject of instrumental analyses is that of continuous automatic monitoring methods. A number of techniques are being considered in this connection. Some are particularly convenient, either by nature of the measurement or the simplicity of the required equipment. One such technique is

REDUCTION OF COMPTON COUNTING
RATE USING TWO CRYSTALS

FIGURE 9

Fig. 9

6732-20

that of gamma monitoring which is simplified by virtue of the fact that the sample material need not come in physical contact with the measuring device. A simple and convenient device for such monitoring is shown in Figure 10.

(Here Fig. 10)

The sample consists of a liquid flowing through the sampling tube and is viewed through an opening in a circular metal disc by a total gamma scintillation counter. The circular disc may be rotated automatically so as to place in sequence under the counter head a blank area to allow determination of background, a standard gamma source to allow calibration, and the sample. The technique thus allows continuous recording of the gamma activity of the sample with intermittent calibration by means of background and standard measurements.

Another very simple continuous monitoring device is that for turbidity with a unit such as shown in Figure 11.

(Here Fig. 11)

The sealed envelope contains a light source in an appendage to the cell and a phototube that picks up the light scattered from solid matter in a suspension. Such a unit need not be more than several inches in length and is thus conveniently immersed in a suspension to provide a continuous record of the turbidity level.

A third monitoring device that might be mentioned is that for determining alpha activity. In this case it is necessary to bring the sample into close proximity with the counter head and to recognize that liquids or moderate-to-large quantities of salt attenuate the alpha activity and thus make quantitative counting difficult. One device that is applicable for the purpose is shown in Figure 12.

(Here Fig. 12)

The sample is allowed to flow continuously through the bore of the lower stopcock. On an intermittent cycle this stopcock is mechanically turned 90° to segregate a previously

6732-21

AUTOMATIC GAMMA COUNTER

Fig. 10

FIGURE 10

6732.2.2

TURBIDIMETER PROBE

FIGURE 11

6732.23

FIGURE 12

Fig-12

6732.24

calibrated volume of sample. The upper stopcock is then likewise turned 90° to segregate a volume of flush solution, which washes the sample into a hollow on a strip tape located below the delivery tip. A heater under the tape dries the sample, and the tape is then displaced to carry the sample under an alpha scintillation counter, at which point the alpha activity is counted.

Glass electrodes, of course, have been used for many years as a continuous monitoring device. One consideration that required attention before such application to highly radioactive solutions is the effect of activity on the behavior and life of the electrodes. Tests made with extremely high levels of alpha, beta, and gamma activity showed no deleterious effect in either respect (6), even though the glass was strongly discolored by the radiation.

Another monitoring device that might be cited by way of example is that employed for photometric measurement. In application to highly radioactive materials, it was necessary to overcome the difficulties associated with discoloration of glass cell windows. This problem is overcome in part in a system such as that shown in Figure 13.

(Here Fig. 13)

Liquid in a sample line is intermittently drawn by application of a vacuum into the separate glass photometric cell where its optical density is determined through application of the constant light source and phototube pick-up before being returned to the sample line. By this means continuous contact between the active solution and the glass cell is avoided and, in addition, it becomes convenient to draw a standard solution into the cell from an external source in order to recalibrate the equipment.

Similarly, polarography lends itself to remote, automatic analyses and has been successfully applied in this manner. Intermittent introduction of sample is readily accomplished; provisions can be made to trap out mercury; and temperature control can be afforded by a temperature bath or by automatic correction. It is more convenient

6732-25

PHOTOMETER CELL UNIT

FIGURE 13

6732-26

to plot the derivative polarogram rather than the wave because the chart record then consists of a series of pips, the height of each representing the concentration of a given sample.

By way of example we have discussed coulometric titration, gamma-ray spectrometry, and several automatic analytical monitoring devices. The final technique that might be discussed is that of x-ray photometry. The essential features of an x-ray photometer are shown in Figure 14.

(Here Fig. 14)

They consist of the source of x-rays, the sample and reference cells, and the x-ray pick-up. The principle of the technique is identical with that of visible photometry in that it involves measurement of the quantity of radiation absorbed by the sample. In this case, however, the extent of x-ray absorption is related to the atomic number of the atoms in the path of the radiation. Thus, the technique is most applicable to the determination of heavy metal atoms in a light solvent. A commercial instrument was developed by the General Electric Company⁽⁹⁾ for specific application to the determination of lead in gasoline; it is equally applicable for the determination of uranium or plutonium in solutions, as shown in Table I which lists the relative absorption of x-rays by a randomly selected group of elements.

TABLE I

RELATIVE X-RAY ABSORPTION FOR VARIOUS ELEMENTS
(Aqueous Solutions -- 12 mm path length -- 42 kvp.)

<u>Element</u>	<u>Atomic Number</u>	<u>Absorption per g/l (in equiv. mils Al)</u>	<u>Relative Absorption</u>
U	92	6.76	100
Pb	82	4.93	73
La	57	2.10	31
Ag	47	3.36	50
Br	35	2.64	39
Cu	29	1.96	29
Fe	26	1.42	21
Cl	17	0.39	5.8
Al	13	0.31	4.6
Na	11	0.14	2.1

6732-27

FIGURE 14

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Several modifications of the commercial instrument were desirable in order to make it most efficient for handling radioactive solutions. The most specific need was to reduce the volume of the sample solution in order to reduce exposure hazard. This was accomplished by reducing the cross section of the x-ray beam and by employing small sample cells such as those shown in Figure 15.

(Here Fig. 15)

The cells are of fixed path length so that there is no need for volume measurement of the solution added. Two size cells have been employed, one with a path length of 30 mm that holds 8 ml of solution, and one with a path length of 12 mm, holding 2.7 ml.

The technique finds its greatest application to relatively high concentrations of heavy metal salts, the optimum concentration in such cases being 40 g/l. High concentrations of extraneous salts constitute an interference, particularly if the salts contain atoms of relatively heavy mass. The precision of a single measurement of an unknown against a reference standard is $\pm 0.3\%$. If a second measurement is made with the standard and reference cells interchanged, the precision of the average measurement is reduced to $\pm 0.15\%$, which is exceptionally good for a photometric method of analysis.

The author acknowledges his debt to the analytical research chemists of the Hanford Operations whose work he has discussed and to the Atomic Energy Commission for sponsorship of this work through its prime contract with the General Electric Company.

6732-29

Unclassified

FIGURE 15

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6732-31

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