

RADIOACTIVE INDICATORS IN ANALYTICAL CHEMISTRY

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Radioactive elements used simply as a tool in chemical and physical research have been called radioactive indicators¹ (23). Since this term first appeared in the literature, innumerable applications of these indicators have been made to various problems of chemistry, physics, medicine, and biology (30), until at present a voluminous literature exists to bear testimony concerning their usefulness. Among the problems which have been attacked with their aid are many of analytical chemical significance; it is here proposed to summarize these contributions.

Radioactive indicators are not indicators in the usual analytical sense of the word, there being no question of a visual change. Their particular value lies rather in the ease and accuracy with which they are detectable in submicroscopic quantities. Their detection and measurement is simply effected by the use of suitable electroscopes and electrometers, or by means of the photographic plate, all of which are extremely sensitive to the radiations from radioelements. The type of electroscope employed depends of course on the nature of the element involved, i.e., on whether it emits alpha particles, beta particles or gamma radiation. Details of construction of instruments, and methods of obtaining and measuring radioactive preparations must be omitted here. They can be found by consulting standard texts on radioactivity (17, 28) and the scientific periodical literature (22). The Lind interchangeable head electroscopes (16) are very satisfactory for measuring alpha and gamma radiations. Convenient beta-ray electroscopes of simple construction are described in the literature (17, 22, 28).

The elements most frequently used in the past as indicators are included in table 1, together with their characteristic radiation (27). They have been chosen for the ease with which they are obtained and measured. The rate of disintegration of radioelements imposes a serious restriction on their general usefulness, for only elements which decay relatively slowly during the course of a series of experiments may be used. It will be seen that all

¹ This definition does not include the use of radioelements as sources of radiation in nuclear disintegration experiments.

elements listed save thoron, which is never used alone, have a convenient half-life.

Applications to analytical chemistry have been of two general types. On the one hand attempts have been made to use radioelements for strictly analytical purposes such as microanalyses and solubility determinations. More frequent, however, has been their use in studies relating to the nature of analytical precipitates and involving such puzzling phenomena as coprecipitation and aging. Such investigations are of interest to the analytical chemist and will be briefly described. For a more exhaustive discussion, the original papers must be studied.

TABLE 1
Common radioactive indicators

RADIOELEMENT	RADIATION	COMMON ELEMENT	HALF LIFE*
UX ₁	Beta	Th	24.5 days
Ra.....	Alpha	Ra	1590.0 years
Rn.....	Alpha	Rn	3.825 days
Ra D.....	Beta	Pb	22.0 years
Ra E.....	Beta	Bi	4.9 days
Po.....	Alpha	Po	140.0 days
Ms TH ₂	Beta	Ac	6.13 hours
Rd Th.....	Alpha	Th	1.90 years
Th X.....	Alpha	Ra	3.64 days
Th B.....	Beta	Pb	10.6 hours
Th C.....	Alpha, beta	Bi	60.5 minutes
Tn.....	Alpha	Rn	54.5 seconds

* Reference 27.

ANALYTICAL APPLICATIONS

Microanalysis

The strictly analytical applications will first be considered. Whenever a radioactive substance is added to a solution containing a given amount of inactive common element with which it is isotopic, the mixture is chemically inseparable; and the activity of the radioelement in arbitrary units thereafter represents the given weight of the common element in all of its chemical reactions. If now a certain fraction, say half, of the element is removed from solution by precipitation or electrodeposition, the same portion of the radioelement is simultaneously removed. The radioelement therefore acts as an indicator for the inactive element.

In this manner, G. von Hevesy and R. Hobbie (34) have taken advantage of the isotopic relationship between radium D and lead to place a radiometric control on a large series of microdeterminations of lead made in connection with a study (33) of the abundance of lead in rocks from various

sources. Their procedure involved the separation of traces of lead from the siliceous mass by the use of hydrofluoric acid, and the subsequent anodic deposition of PbO_2 , which was determined gravimetrically. They then performed the lead determination colorimetrically. The agreement being poor, a control on the completeness of recovery became imperative. This was done simply by the addition of a small quantity of radium D soon after removal of silica, and by subsequently determining the radioactivity of the deposited lead. Knowing both the total activity of the radium D added and the activity recovered by electrolysis, they calculated directly the percentage of total lead which succeeded in reaching the anode. Since this percentage corresponds to the weight of lead actually recovered, the total weight could easily be found by correcting for the deficiency in the radium D activity. Table 2 illustrates the data found and shows the agreement between the various methods of determining lead.

TABLE 2
Rock analyses with radiometric control

KIND OF ROCK	WEIGHT OF SAMPLE	Pb ON ANODE $\frac{\text{g. Pb}}{\text{g. Rock}}$	COLORIMETRIC DETERMINATION $\frac{\text{g. Pb}}{\text{g. Rock}}$	PER CENT OF Ra D RECOVERED	Pb CONTENT CORRECTED $\frac{\text{g. Pb}}{\text{g. Rock}}$
	<i>grams</i>				
Dunite.....	34.6180	32×10^{-6}	30×10^{-6}	71	42×10^{-6}
Lherzolite.....	24.6817	15×10^{-6}	13×10^{-6}	69	19×10^{-6}
Granite.....	48.0611	18×10^{-6}	16×10^{-6}	53	30×10^{-6}
Basalt.....	28.0984	4×10^{-6}	5×10^{-6}	100	4×10^{-6}
Kimberlite.....	33.0458	12×10^{-6}	11×10^{-6}	69	16×10^{-6}

A similar procedure is mentioned by A. von Grosse (31), who used uranium X_1 as an indicator for thorium in determining as little as 0.01 per cent in Katanga pitchblende. Such a procedure is equally applicable to the determinations of traces of bismuth and thallium, both of which possess radioactive isotopes.

R. Ehrenberg (2) has proposed numerous radiometric microanalytical procedures based upon a succession of reactions ending with an incomplete precipitation of radioactive lead nitrate (containing thorium B). All reactions are carried out in a microcentrifuge cup to permit the separation of solids as they are formed. Starting with a series of solutions of known ion concentration, Ehrenberg establishes an empirical "standardization" curve relating the initial concentration of a given ion to the quantity of the thorium B remaining in solution.² It should be emphasized that each ion requires an individual calibration curve, which will vary with the condi-

²The amount of thorium B is measured by the alpha- or beta-ray activity of thorium C after radioactive equilibrium is established.

tions prevailing during the analysis. The chloride-ion determination (3) will serve to illustrate the principle of the method. First a standardization curve was constructed in the region under investigation, say 1.8×10^{-3} to 5.4×10^{-3} mg. of chloride ion. To 0.15 cc. of chloride solutions of known concentrations were added successively 0.5 cc. of 10^{-3} *N* silver nitrate solution, 0.05 cc. of 10^{-2} *N* potassium chromate solution, and 1.0 cc. of 10^{-3} *N* radioactive lead nitrate solution, centrifuging after each precipitation; then 1.0 cc. of the supernatant liquid was removed for measurement in an electroscop. Tabulating the initial chloride content of the known solutions with the residual activities, expressed both in arbitrary units (discharge of electroscop in unit time) and as a percentage of a control solution containing no initial chloride, we obtain table 3. If now a solution containing an unknown chloride content were treated as described, and the residual activity amounted to 50 per cent of the control activity (i.e., 5000 arbitrary units), then by a simple interpolation it would be found that the unknown had contained 2.1×10^{-3} mg. of chloride ion.

TABLE 3
Chloride-ion standardization

Cl ⁻ CONCENTRATION IN MILLIGRAMS	ACTIVITY IN ARBITRARY UNITS	PER CENT ACTIVITY LEFT
0	10,000	100.0
1.8×10^{-3}	5,113	51.1
3.6×10^{-3}	4,533	45.3
5.4×10^{-3}	3,910	39.1

Similar micromethods have been presented for the determination of many anions and cations as well as for organic nitrogen and carbon; and a sugar determination has also been developed. Recently A. Polessitsky (26) has employed radium as an indicator for barium in a modification of Ehrenberg's method, and has determined several milligrams of barium in the presence of large amounts of lead. However, in all of these cases an empirical curve must first be established in the particular concentration range under investigation. Frequently linear relationships are obtained, though it is clear that these curves have no theoretical significance. As yet, no rigid test has been made of their accuracy or applicability to conditions usually encountered in the laboratory. It is uncertain whether such empirical methods will ever be of great utility except in isolated cases.

Solubility determinations

Solubility determinations constituted one of the first uses of radioactive indicators. The basis of such determinations is again the chemical inseparability of isotopic mixtures. If a radioactive lead salt is precipitated

from a solution containing originally a given weight of lead associated with a known activity of radioactive isotope (RaD or ThB), then the solubility of the precipitate, in terms of lead ions, is given directly by the activity of the saturated solution. For example, F. Paneth and G. von Hevesy (23), using radium D, prepared a radioactive solution containing 9.69 mg. of lead chloride and possessing a beta-ray activity of 2030 arbitrary units, from which they precipitated active lead chromate by the addition of potassium chromate. Thereupon the insoluble salt was washed and a saturated solution prepared at 25°C. The beta-ray activity of the evaporation residue from 70 cc. of a saturated solution was 0.150 unit, indicating that $\frac{323.2}{278.1} \times 0.00969 \times \frac{1000}{70} \times \frac{0.150}{2030} = 1.2 \times 10^{-5}$ g. of lead chromate or 0.4×10^{-7} mole was dissolved per liter. Later experiments by G. von Hevesy and E. Róna (35), using thorium B, raised this low value to 2×10^{-7} mole per liter, in good agreement with the average of values obtained by other methods. Unfortunately this procedure, until fairly recently, has not been further developed. At present, however, experiments are planned in which the solubility of lead salts as a function of particle size will be measured by the indicator method.

Recently we have made use of the Paneth specific surface determination (20, 25) to permit estimates of solubilities in regions difficult to investigate. From a study of the thorium B adsorption, Paneth has calculated the surface extent of samples of lead sulfate to be

$$\text{Specific surface} = \frac{\text{Pb}_{\text{surf.}}}{1 \text{ g. PbSO}_4} = \frac{\text{ThB}_{\text{surf.}}}{\text{ThB}_{\text{soln.}}} \times \frac{\text{Pb}_{\text{soln.}}}{\text{wt. of PbSO}_4}$$

where $\text{Pb}_{\text{surf.}}$ is the weight (or number of ions) of lead present in the outermost crystal layer, $\text{Pb}_{\text{soln.}}$ is the amount of lead in the given volume of solution, and the ratio $\frac{\text{ThB}_{\text{surf.}}}{\text{ThB}_{\text{soln.}}}$ is the ratio of adsorbed thorium B to that left in solution. Using a specified sample of lead sulfate and determining the thorium B adsorption in increasing concentrations of lead nitrate, it was found that the ratio $\frac{\text{ThB}_{\text{surf.}}}{\text{ThB}_{\text{soln.}}}$ varied simply with $\text{Pb}_{\text{soln.}}$ calculated from the known lead concentrations, i.e., the specific surface remained constant (13, 20). Assuming this constancy to hold as well for $\text{Pb}_{\text{soln.}}$ values below that in a saturated water solution, I. M. Kolthoff and C. Rosenblum (13) studied the thorium B adsorption in increasing concentrations of sodium sulfate. In these cases the calculated $\text{Pb}_{\text{soln.}}$ values represented solubilities of lead sulfate in the corresponding sulfate-ion concentrations. Since the specific surface is known from experiments in the absence of excess lattice

ion, and the volume of solution and the weight of lead sulfate may be varied at will, then a simple measurement of the ratio $\frac{\text{ThB}_{\text{surf.}}}{\text{ThB}_{\text{soln.}}}$ permits the calculation of $\text{Pb}_{\text{soln.}}$ from the definition of specific surface. Solubilities so obtained were checked by direct analysis for lead in solutions as high as $1.5 \times 10^{-3} N$ in sodium sulfate. That the calculations are justifiable is seen from table 4.

TABLE 4
Solubility of lead sulfate in sodium sulfate solutions

SODIUM SULFATE CONCENTRATION	SOLUBILITIES (23°C.)	
	Radiometric	Analytical
	<i>N</i>	<i>N</i>
0	(2.8×10^{-4})	2.8×10^{-4}
5.05×10^{-4}	1.27×10^{-4}	1.34×10^{-4}
7.50×10^{-4}	0.99×10^{-4}	1.04×10^{-4}
10.0×10^{-4}	0.81×10^{-4}	0.90×10^{-4}
25.0×10^{-4}	0.56×10^{-4}	(0.60×10^{-4})
50.0×10^{-4}	0.44×10^{-4}	
75.0×10^{-4}	0.34×10^{-4}	
100.0×10^{-4}	0.31×10^{-4}	

TABLE 5
Solubility of lead sulfate in alcohol-water mixtures

WEIGHT PER CENT OF ETHYL ALCOHOL	SOLUBILITIES (25.5°C.)	
	Mg. of PbSO_4 per liter	Molarity
(Water solution)	45.1	1.49×10^{-4}
13.51	10.1	3.33×10^{-5}
29.9	1.46	0.48×10^{-5}
49.5	0.32	0.107×10^{-5}

Solubilities of lead sulfate in several aqueous alcohol solutions have also been determined in this manner. Preliminary determinations yield the data included in table 5. No claims of great accuracy are made. Rather is the method of an orienting nature, permitting close approximations to the correct solubilities in regions which are not readily susceptible to customary analytical procedures.

The validity of the mass action law at great dilutions

The sensitivity of radiometric procedures is admirably illustrated by recent tests of the validity of the mass action law as applied to the insoluble

compound radium sulfate. O. Erbacher and B. Nikitin (5, 18), and more recently B. Nikitin and P. Tolmatscheff (19), have found that the activity product of radium sulfate remains constant in sodium sulfate and sulfuric acid concentrations as high as 0.1 *N*. Table 6 illustrates some of the findings from the latter paper.

The validity of the Nernst electromotive force equation at great dilutions

Another fundamental expression which had been tested in extreme dilutions is the so-called Nernst electromotive force equation, from which it follows that for every ten-power difference in concentration of a given ion, there is a corresponding difference in electrode potential of $2.303 \frac{RT}{nF}$ volts. Since it is extremely difficult to measure the potential of an electrode of the first order containing ion concentrations as small as those involved in the handling of radioelements (10^{-8} to 10^{-14} *N*), F. Paneth and G. von Hevesy

TABLE 6
Validity of the mass action law

COMPOSITION OF SOLUTION	$\alpha_{\text{Ra}^{++}} \times \alpha_{\text{SO}_4^-}$
H ₂ O.....	4.17×10^{-11}
$10^{-4}N$ Na ₂ SO ₄	4.31×10^{-11}
$10^{-3}N$ Na ₂ SO ₄	4.08×10^{-11}
$10^{-2}N$ Na ₂ SO ₄	4.45×10^{-11}
Average.....	4.25×10^{-11}

(24) resorted to approximate measurements of deposition potentials of polonium, thorium B, and radium E, which, in the absence of polarization effects, equal in magnitude their electrode potentials. However, there is an insufficient concentration of radioactive ions to carry a measurable current; therefore, instead of measuring current, they determined the quantity of radioelement depositing on a noble metal at various potentials. At a voltage corresponding to the deposition potential, there should occur an abrupt increase in the amount of radioelement deposited. Their results are illustrated in figure 1, in which are found plotted the percentage deposition of radium E from two 0.1 *N* nitric acid solutions which are 10^{-9} *N* in bismuth (radium E containing no added inactive bismuth) and 10^{-4} *N* in bismuth (containing inactive bismuth nitrate), respectively, as a function of applied potential. The voltages which produce the sudden increase in deposition of radioelement are presumably the deposition potentials. From figure 1 we see that these voltages are 0.14 and 0.24 for the 10^{-4} *N* and the 10^{-9} *N* bismuth solutions, respectively, indicating that a differ-

ence in potential of 100 millivolts should be set up between two half cells containing the above amounts of bismuth. Applying the Nernst equation to the trivalent bismuth ion at the temperature of the experiment, a difference of potential equivalent to 90 millivolts was predicted. Considering the extreme difference in concentration involved, and that this work was done before the methods of purification of many radioelements had been developed, the agreement is exceedingly gratifying.

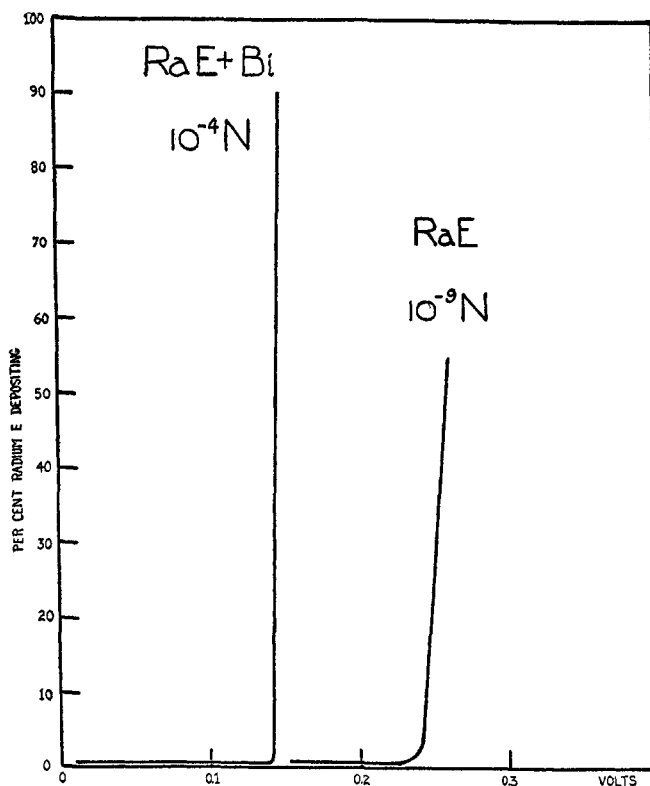


FIG. 1. Separation of radium E from 0.1 *N* nitric acid

An interesting application of radioactive indicators could be made in a study of the quantitative electrolytic deposition of individual metals and separation of metals. Paneth (21) emphasizes the potentialities of such electrochemical studies; and his belief seems justified by experiments (4, 32) on the mechanism of the conduction of electricity at the electrode-solution interface.

THE NATURE OF ANALYTICAL PRECIPITATES

Aged precipitates

Many studies have been made of the adsorption and coprecipitation of radioactive substances. Aside from the direct measurement of the adsorption of radioelements themselves, K. Fajans and T. Erdey-Grúz (6) have proposed a modification intended to permit the determination of the order of adsorbability of foreign ions in general. For example, when 0.4 g. of

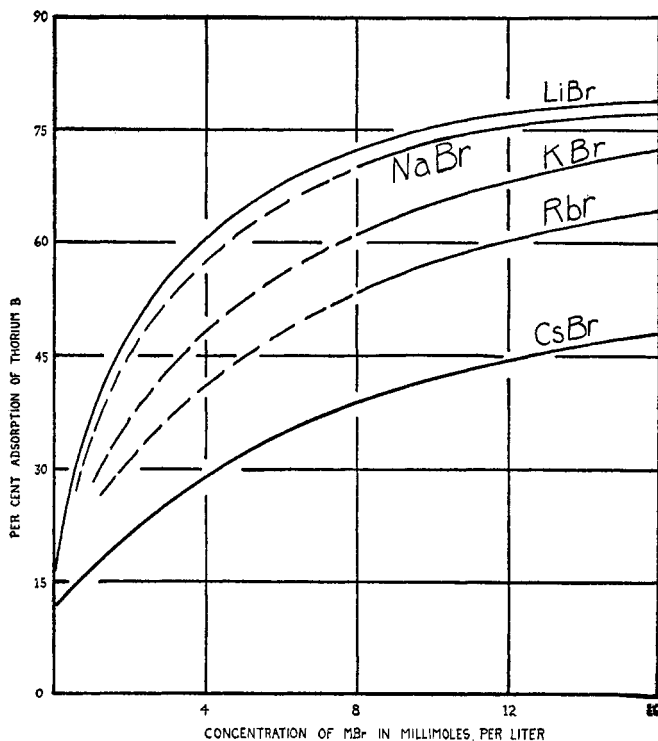


FIG. 2. Adsorption of thorium B on silver bromide in the presence of alkali bromides

silver bromide was shaken with a weakly acid solution of thorium B, only 1.8 per cent of the thorium B was adsorbed. When, however, the bromide-ion concentration was increased by the addition of potassium bromide, there occurred an increase in thorium B adsorption, until at 16 millimoles of potassium bromide per liter, 63.9 per cent of thorium B was adsorbed. Repeating these experiments with potassium chloride, an effect similar to that with bromide ion was obtained, except that the thorium B adsorption was less. Thus only 21.5 per cent of thorium B was adsorbed from a solu-

tion containing 16 millimoles of potassium chloride per liter. Fajans interprets this as indicating a smaller adsorption of chloride ion by silver bromide than of bromide ion. Adopting this procedure J. F. King and collaborators (11, 12) have determined the adsorption of various ions by thallos halides and silver bromide. Some of these findings are included in figure 2, showing the effect of alkali bromides on the thorium B adsorption by silver bromide. From the diminution in thorium B adsorption, it may be concluded that the adsorption of alkali ions is in the order $Cs > Rb > K > Na > Li$.

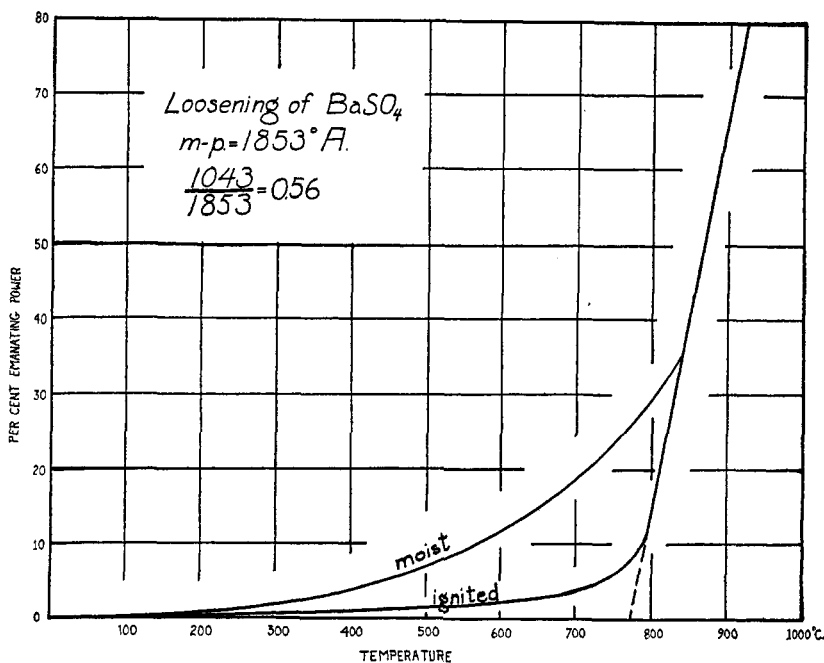


FIG. 3. Effect of heat on the emanating power of barium sulfate

O. Hahn has developed the emanation method (10) as a valuable means of investigating the mechanical properties of crystals. The procedure requires a precipitation or recrystallization in the presence of a radioelement which precedes the gaseous emanation in one of the radioactive families. Emanators most frequently used in the past have been radium and radiothorium. The quantity measured is the amount of emanation which succeeds in escaping from the crystals, expressed as per cent of the total emanation formed from the emanator. By this method, the aging of metal oxide gels has been intensively studied under various conditions

(1, 7, 8, 9). Quite illuminating is the effect of temperature on certain barium salts impregnated with a trace of radium. The effect of heat on the emanating power of barium sulfate taken from experiments of F. Strassmann (29) is shown in figure 3. The behavior of two similarly prepared samples of barium sulfate is represented. One sample was heated to 600–700°C. for three hours after precipitation, whereas the second was simply air-dried and therefore retained a considerable quantity of occluded water. Evidently the initial emanating power of the ignited precipitate is negligible and remains practically unaffected by heat until a temperature of 780°C. is attained. At this point there occurs an abrupt increase in emanating power, indicating a loosening of the crystal lattice which permits the escape of radon. The air-dried precipitate behaves somewhat differently, showing from the very beginning a gradual increase in emanating power with rise in temperature, owing to a slow distillation of occluded water which sweeps with it appreciable quantities of radon. However here, too, there is evident a sudden liberation of emanation at about 800°C., indicating that barium sulfate possesses a characteristic relaxation temperature. From present indication it would appear that such a relaxation temperature may be characteristic of many crystal powders, and occurs at an absolute temperature equal approximately to 0.5–0.6 of the absolute melting point of the substance (29).

Fresh precipitates

Considerable progress has likewise been made in understanding the nature of fresh precipitates and the processes occurring on aging at room temperature. Recent experiments (14) with the adsorption of thorium B by lead sulfate precipitates of increasing age have indicated that the fresh crystals, though apparently well formed when viewed under the microscope, are in reality spongy masses of exceedingly minute amicronic particles. These conclusions were drawn from the fact that the specific surfaces (20, 25) calculated from thorium B adsorptions were extremely large for fresh lead sulfate. Our latest experiments (15) show that fresh lead sulfate is permeable by lead ions; and even a fifty minute old sample permits a free mobility of lead ions throughout the crystal mass. This was demonstrated by adding 0.1 *N* sodium sulfate solution to a slight excess of 0.1 *N* lead nitrate solution. After a noted time (age of precipitate), thorium B was added and the whole shaken for a given time before measuring the adsorption of the radioelement. It was found that for fresh precipitates and sufficiently long shaking periods the ratio $\frac{\text{ThB}_{\text{surf.}}}{\text{ThB}_{\text{soln.}}}$ equalled the ratio $\frac{\text{Pb}_{\text{ppt.}}}{\text{Pb}_{\text{soln.}}}$. Clearly the precipitate has allowed thorium B ions, and

therefore lead ions, to diffuse freely throughout the crystals from which it is composed. A number of such experiments are graphically represented in figure 4. Here the term $Pb_{\text{exchanged}}$ is used in preference to $Pb_{\text{surf.}}$, since lead ions from crystal layers other than the outermost are involved. In the figure the $Pb_{\text{exchanged}}$ is expressed as per cent of the maximum. Repeating these experiments using radioactive lead sulfate samples yields similar curves, showing the exchange to be a reversible process. From these data it is clear that the drastic aging process occurs within the microscopic crystal, and evidently involves a growth of the primary amionic particles.

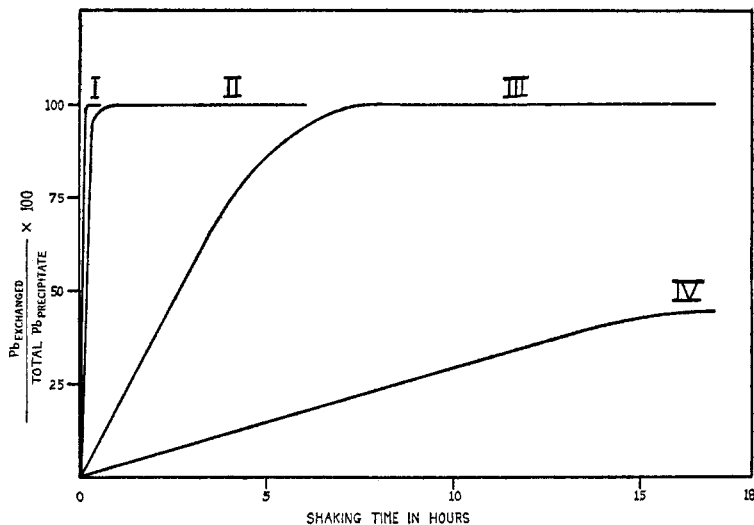


FIG. 4. Aging of lead sulfate at room temperature. I, precipitate 50 seconds old; II, precipitate 8.5-9 minutes old; III, precipitate 50-51 minutes old; IV, precipitate 3 hours old.

SUMMARY

1. Applications of radioactive indicators to microanalysis were summarized.
2. Solubility determinations made with the aid of radioelements were described.
3. The sensitivity of the methods of measuring radioactive substances permitted the examination of fundamental laws concerning the separation of ions in extreme dilutions.
4. It was found possible to study the relative adsorbability of various ions by their effect on the adsorption of thorium B.
5. The emanation method was shown to be a convenient means of studying properties of analytical precipitates.

6. The mechanism of aging processes in fresh precipitates may be studied with the help of radioelements. The example of lead sulfate aging was briefly considered.

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