counter in which the sample holder was at a positive potential. A slight correction to the counting yield, which was assumed to be 52%,[11] may be required due to the unknown counting efficiency for the actinon and recoil particles which escape from the sample plate.

Although five to six months after separation are required for the five alpha-emitting daughters of actinium to grow to equilibrium, fairly accurate estimates of the amount of actinium present may be made after one or two weeks by comparison of the experimental and theoretical growth curves.

Growth curves of the alpha activity of samples of the purified actinium have now been followed for periods up to one year. The coincidence of the experimental and theoretical curves indicates that the material was free of radioactive impurities. A typical growth curve is shown in Fig. 4.

The yield of actinium calculated from the equilibrium amount of alpha activity was 93 millicuries. Assuming the half-life to be 21.7 years this corresponds to 1.27 mg. of pure actinium.

A large amount of ThB activity was found in the TTA fraction from the original separation which contained the thorium isotopes, indicating the presence of RdTh (Th^{228}). The presence of these members of the thorium series might be ascribed to neutron capture by AcX, Ac^{227} or RdAc; however, since the radium bromide which was irradiated probably contained some $MsTh_1$ impurity, it is not possible to estimate the amount of such second order capture.

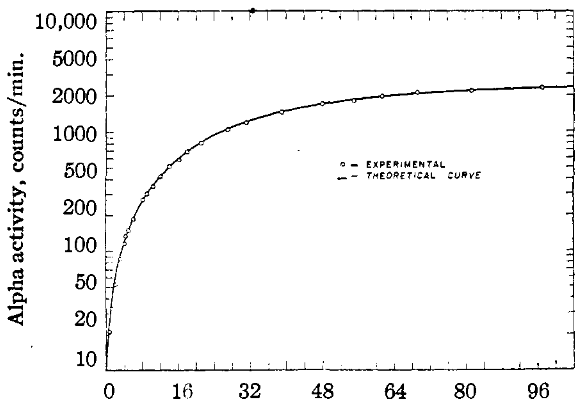

Fig. 4.—Typical curve of growth of alpha activity in purified actinium.

Summary

1. Milligram quantities of actinium, produced artificially by transmutation of radium with pile neutrons, have been isolated for the first time in the form of essentially pure compound.

2. Separation of actinium from radium and daughter elements was accomplished by extraction from aqueous solution at controlled hydrogen ion concentration with thenoyltrifluoroacetone–benzene solution.

3. The extraction of actinium, radium, thorium, polonium, lead, bismuth, thallium(I) and thallium(III) from aqueous solution by 0.25 *M* thenoyltrifluoroacetone in benzene as a function of the equilibrium *p*H of the aqueous phase has been investigated on a tracer scale.

CHICAGO 80, ILL. RECEIVED MAY 19, 1949

[CONTRIBUTION FROM THE CHEMISTRY DIVISION OF THE ARGONNE NATIONAL LABORATORY]

The Preparation and Identification of Some Pure Actinium Compounds[1]

BY SHERMAN FRIED, FRENCH HAGEMANN AND W. H. ZACHARIASEN

Previous preparations of actinium from natural sources have always contained gross amounts of the difficultly separable rare earths (in particular lanthanum), which occur in much greater concentrations than actinium in uranium ores. Therefore, direct observation of the characteristics of pure actinium compounds has been impossible, and the chemical properties of the element have necessarily been inferred entirely from its coprecipitation behavior with large amounts of inert carrier. Such tracer experiments have indicated that actinium is very similar to, and probably more basic than, lanthanum which is the most basic rare earth. For example, if an actiniferous solution of lanthanum is precipitated as the hydroxide, fluoride, phosphate, oxalate or carbonate, the activity due to actinium in each case is found almost quantitatively in the precipitate. Advantage has been taken of slight differences in solubility of actinium and lanthanum compounds in effecting some enrichment of actinium preparations, but isolation from natural sources has never been achieved.[1a]

Recently, milligram quantities of actinium, produced artificially by transmutation of radium in a chain-reacting pile, have been obtained in a pure state.[2] By applying microtechniques[3] which have been successfully employed in this Laboratory in the past with other heavy elements it has now been possible, for the first time, to prepare and identify several of the compounds of actinium.

Identification of the actinium compounds was

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(1) Presented at the San Francisco meeting of the American Chemical Society, March, 1949.

(1a) Gmelin, "Handbuch der anorganische Chemie," 8 Auflage, Actinium, System-Nummer 40, Berlin, 1942. This reference contains a summary of the literature on actinium to 1940.

(2) Hagemann, THIS JOURNAL, 72, 768 (1950).

(3) Fried and Davidson, *ibid.*, 70, 3539 (1948).

made by analysis of their X-ray diffraction patterns. Within the Manhattan Project during the war years, X-ray diffraction methods were extensively used as a means of determining the chemical identity of compounds of transuranium elements at a time when only microgram amounts of these elements were available. Satisfactory X-ray diffraction patterns were obtained with as little as two micrograms of material in a powder form. The interpretation of the X-ray data, together with the application of general crystal chemical principles and a knowledge of how the sample had been prepared, in many instances sufficed for conclusive chemical identification. The X-ray diffraction method was the only method of microchemical analysis used in the course of the actinium studies described in this paper.

Preparation of Actinium Compounds

In most cases the method of preparation was first tested and perfected using lanthanum as a "stand-in" for actinium. The choice of lanthanum for this purpose was obvious from a consideration of the relative positions of the two elements in the periodic system and their chemical similarity as indicated by the radiochemical behavior of actinium in tracer experiments. Frequent comparisons will be made between lanthanum and actinium chemistry throughout this paper.

Although approximately a milligram of actinium was available for experiment, the preparations were conducted on the ten-microgram scale. The reasons for such small-scale operations were twofold: (a) to minimize the radiation health hazards incident to handling the highly radioactive material, and (b) to reduce the fogging of the X-ray film by the gamma radiation emitted by the actinium samples. Actinium itself emits only weak radiation, but hard radiation due to the short-lived daughters rapidly grows into a sample after purification. In order to prevent complete masking of the X-ray pattern by background darkening of the film, it was found necessary to prepare and photograph the compound within twenty-four hours after separation from daughter activity. Excessive sample size, *i. e.*, greater than 5 to 10 micrograms, also resulted in fogged film even shortly after purification.

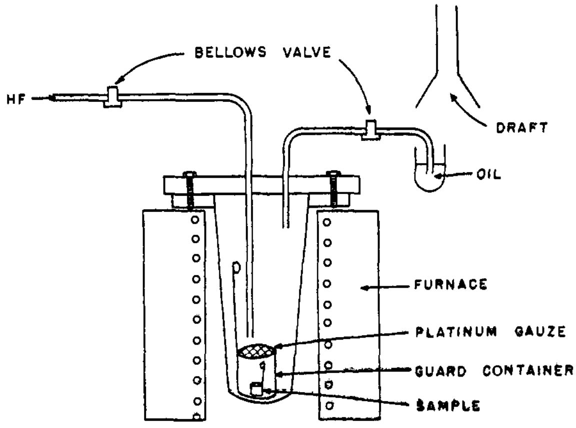

Fig. 1.—Platinum hydrofluorination apparatus.

In each experiment the actinium was purified immediately before starting the preparation by extraction with thenoyltrifluoroacetone.[2] Actinium, together with the thorium daughter, RdAc, was first extracted at a *p*H of 6 into a 0.25 *M* solution of thenoyltrifluoroacetone in benzene, the radium daughter, AcX, remaining in the aqueous phase. The actinium was then re-extracted into 0.1 *N* hydrochloric or nitric acid, leaving the RdAc in the benzene phase. Finally, any active deposit present was removed by coprecipitation with lead sulfide.

Where possible the compounds were formed *in situ* using the thin-walled glass or quartz X-ray sample capillary as the reaction vessel in order to reduce the time of preparation, to eliminate the necessity for transfer from one vessel to the other and to remove the possibility of air reacting with the compound.

Experimental

Actinium Trifluoride.—Ten micrograms of actinium was precipitated from 100 microliters of 0.1 *N* hydrochloric acid solution contained in a micro centrifuge cone by passing a stream of gaseous ammonia over the surface of the liquid. After centrifuging, the supernatant liquid was removed and the white gelatinous residue dried at 70° for two hours. The small fragments of the dried solid were then transferred to an all-platinum hydrofluorination apparatus (Fig. 1) and treated with hydrogen fluoride at 700° for fifteen minutes.

On cooling, the hydrogen fluoride was pumped off, dry nitrogen admitted, and the resulting white product mounted in an X-ray capillary. X-Ray diffraction analysis showed the compound to be actinium trifluoride, AcF_3.

It was also shown by X-Ray diffraction that the same compound was obtained by the addition of hydrofluoric acid to a solution of actinium and drying the precipitate at 70°.

Actinium Oxyfluoride.—This compound was prepared by partial hydrolysis of the trifluoride at elevated temperatures.

A ten-microgram sample of actinium fluoride was precipitated in a *quartz* micro cone from 0.1 *N* hydrochloric acid solution with dilute hydrofluoric acid. The precipitate was centrifuged and dried at 70°. The cone was then placed inside a *quartz* tube (fitted with a ground joint) and attached to a vacuum line. The system was evacuated and the vapors from an ammonium hydroxide solution were allowed to act on the actinium trifluoride at 900–1000° for ten minutes. The progress of the hydrolysis was followed by observing the sublimate of ammonium fluoride formed in the reaction

$$AcF_3 + 2NH_3 + H_2O \longrightarrow AcOF + 2NH_4F$$

The reaction product was transferred to an X-ray capillary and shown by analysis to be actinium oxyfluoride, AcOF.

Ignition of lanthanum trifluoride in *air* at 800° for one hour was sufficient to convert it to the oxyfluoride, LaOF, but prolonged ignition of actinium trifluoride in *air* at temperatures up to 1000° had no effect beyond melting the fluoride.

Actinium Trichloride.—Ten micrograms of dried actinium hydroxide (or oxalate) was treated with carbon tetrachloride vapor at elevated temperatures in a *quartz* X-ray capillary in a manner analogous to that used in preparing the chlorides of neptunium and plutonium.[3] The capillary was alternately evacuated and filled with carbon tetrachloride vapors while continuously raising the temperature of the furnace. No material was observed to sublime until the temperature had reached 960°, when a white zone formed in the cool portion of the capillary.

After no more material could be collected, the capillary was sealed off and subjected to X-ray analysis. The white sublimate proved to be actinium trichloride, $AcCl_3$.

Actinium Oxychloride.—This compound was first prepared in an attempt to effect complete hydrolysis of actinium trichloride to the oxide. A sample of actinium trichloride which had been sublimed on the walls of a *quartz* X-ray capillary was allowed to react with the vapors from an ammonium hydroxide solution at 1000° for several minutes. The resulting compound was shown by X-ray diffraction analysis to be the oxychloride, AcOCl.

The same compound also was formed when a sample of actinium which had been precipitated from 0.1 *N* hydrochloric acid solution with ammonia was ignited in air at 1000°. This unexpected result may be explained in one of two ways: either a small amount of ammonium chloride impurity was carried with the precipitate which on subsequent heating reacted to form the oxychloride, or actinium oxychloride was precipitated from the hydrochloric acid solution directly when it was neutralized by the ammonium hydroxide. A reaction analogous to the latter is reported to occur with lanthanum.[4]

Actinium Tribromide.—The preparation of this compound was accomplished by the action of aluminum bromide on the oxide of actinium.

$$Ac_2O_3 + 2AlBr_3 \longrightarrow 2AcBr_3 + Al_2O_3$$

A ten-microgram sample of actinium hydroxide (or oxalate) was introduced into a *quartz* X-ray capillary which was heated to 500° in vacuum, when it presumably was converted to the oxide. Approximately 50 micrograms of aluminum metal was then introduced on top of the oxide, and the mixture was treated with chlorine-free bromine at 350°. As the aluminum metal reacted with the bromine, aluminum bromide sublimed as a white solid in the zone just outside the heating block. This procedure eliminated the difficult transfer of small amounts of the exceedingly hygroscopic aluminum bromide from a reagent bottle into the bore of the fine capillary.

The tube was evacuated and sealed off so that its length was approximately five inches, and placed in a heavy-walled *quartz* jacket which was sealed at both ends. The heavy tube containing the X-ray capillary was heated to 750° for two hours and then allowed to cool.

After removal from its protecting jacket, the capillary was connected to the vacuum line as shown in Fig. 2 and the tip broken by rotating the stopcock. The contents were then subjected to fractional sublimation in vacuum. The excess aluminum bromide was distilled first at temperatures below 400° and on raising the temperature to 800° a white material finally sublimed. This was shown by X-ray diffraction analysis to be actinium tribromide, $AcBr_3$.

Actinium Oxybromide.—When a sample of actinium tribromide was treated with the vapors from an ammonium hydroxide solution at 500° for five minutes in the manner described above for the hydrolysis of actinium trichloride, the white oxybromide of actinium was formed. This was identified by X-ray diffraction as AcOBr.

Actinium Sesquioxide.—The oxide was prepared by ignition of actinium oxalate (precipitated from a nitric acid solution) in a *quartz* X-ray capillary in an atmosphere of dry oxygen (0.5 atm.) at 1100°. X-Ray diffraction analysis showed it to be actinium sesquioxide, Ac_2O_3. The oxides of actinium, lanthanum, cerium and praseodymium are isomorphous.

As described above, ignition of actinium hydroxide precipitated by ammonia gas from hydrochloric acid solution, or hydrolysis of actinium chloride at high temperature yielded the oxychloride rather than the oxide.

Actinium Phosphate Hemihydrate.—Fifty microliters of a 1.0 *N* solution of sodium dihydrogen phosphate was added to an equal volume of solution containing 10 micrograms of actinium in 0.1 *N* hydrochloric acid. The white solid which precipitated was centrifuged, washed, and dried at 70°. X-Ray analysis showed this compound to be actinium phosphate hemihydrate, $AcPO_4 \cdot 0.5H_2O$.

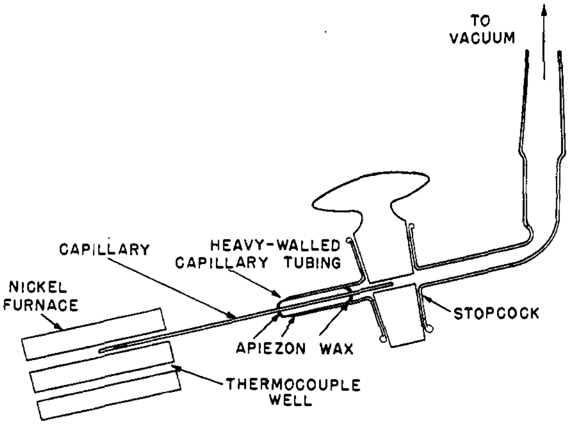

Fig. 2.—Apparatus for the sublimation of actinium iodide and bromide.

Actinium Sesquisulfide.—Ten micrograms of actinium as the oxalate was placed in a graphite crucible, surrounded by a *quartz* tube, and heated to 1400° by means of an induction furnace for six minutes. During the heating, a stream of hydrogen sulfide which had been bubbled through carbon disulfide was passed through the system. The black product was removed from the crucible and subjected to X-ray analysis. It was shown to be the sesquisulfide, Ac_2S_3, isomorphous with the corresponding lanthanum compound.

Treatment of actinium oxide for one hour at 1000° with a mixture of hydrogen sulfide and carbon disulfide yielded a black product which, however, gave an X-ray diffraction pattern too faint to be interpreted. Under similar conditions lanthanum sesquisulfide was formed at 1400 but not at 1000°.

Unidentified Compounds

The following actinium preparations were carried out under conditions which yielded the corresponding lanthanum compounds; however, no positive identification of the products could be made by the X-ray diffraction method. Either no diffraction pattern was obtained or the pattern produced was too complex to permit interpretation.

Actinium Iodide.—When the oxide of lanthanum or of the heavy elements neptunium, plutonium or americium is treated with a mixture of aluminum metal and iodine in a closed system at 600 to 700° for a few hours, the iodide of the element in question is obtained. The reactions may be represented either by

$$X_2O_3 + AlI_3 \longrightarrow XI_3 + Al_2O_3$$

or

$$6XO_2 + 8AlI_3 \longrightarrow 6XI_3 + 4Al_2O_3 + 3I_2$$

depending upon the formula of the oxide.

When 10 micrograms of actinium oxide was treated with about 50 micrograms of aluminum metal and an approximately equal amount of iodine at 700° for two hours in a manner analogous to the preparation of actinium tribromide, a white compound volatile at 800° was obtained. However, the complex X-ray diffraction pattern obtained from this material, which was different from that of lanthanum iodide, has not yet been interpreted.

Since ammonium iodide had been found to convert lanthanum oxide to lanthanum triiodide, this reaction was tried with actinium. An excess of ammonium iodide was added to 10 micrograms of actinium oxalate in a *quartz* X-ray capillary. The capillary was sealed after having

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[4] Kolthoff and Elmquist, THIS JOURNAL, 53, 1225 (1931).

been evacuated and then placed in a heavy-walled *quartz* jacket which was also sealed, and the assembly heated in a furnace for one-half hour at 500 to 525°. The capillary was removed from its jacket, opened by means of the "breaker" shown in Fig. 2, and the contents subjected to fractional sublimation in vacuum. The fraction subliming at 700 to 800° produced the same X-ray diffraction pattern as the previous sample.

It is, of course, possible that the crystal structure of actinium iodide differs from that of the rare earth iodides. The identity of the products obtained by the two methods of preparation, which under similar conditions both yielded lanthanum triiodide, strongly suggests that the compound formed is actinium triiodide, but that it is not isomorphous with lanthanum iodide.

Actinium Oxyiodide.—A sample of "actinium iodide" was hydrolyzed at 700° for five minutes in the same manner as was described for actinium bromide. The resulting compound produced an, as yet, uninterpreted X-ray diffraction pattern, different from either that of "AcI_3" or Ac_2O_3. It is probable that the product of this reaction was the oxyiodide, AcOI, although if so, it is not isomorphous with lanthanum oxyiodide, LaOI.

Actinium Hydroxide.—By passing a stream of gaseous ammonia over a solution of actinium in 0.1 *N* hydrochloric or nitric acid until the solution was alkaline, a white gelatinous precipitate, presumably the hydroxide, was formed. After centrifuging, washing, and drying at 70° for one hour this material produced an X-ray diffraction pattern so diffuse that it could not be interpreted.

Actinium Oxalate.—Addition of 50 microliters of 1 *N* ammonium oxalate solution to 10 micrograms of actinium in an equal volume of 0.1 *N* hydrochloric acid solution produced a white dense precipitate. However, when 100 microliters of saturated oxalic acid solution was added to 10 micrograms of actinium in 50 microliters of 0.1 *N* hydrochloric acid, no visible precipitate was formed. Apparently the oxalate is fairly soluble in mineral acids and requires the buffering action of the ammonium oxalate to reduce the hydrogen ion concentration to a point where precipitation can take place. Although the actinium oxalate appeared crystalline under the microscope, no X-ray diffraction pattern could be obtained from this material. This appears to be a common experience with highly radioactive cations in combination with organic anions.

Anhydrous Actinium Phosphate.—Heating lanthanum phosphate hemihydrate produces the anhydrous form. A sample of the corresponding actinium compound was heated to 700° in vacuum. The resulting material, which was black (probably because of a small amount of impurity), gave an X-ray diffraction pattern which is different from that of $LaPO_4$ and which has not been interpreted. Although the hemihydrate of actinium phosphate is isomorphous with the corresponding lanthanum compound, the anhydrous phosphate apparently is not.

Actinium Potassium Sulfate.—Ten micrograms of actinium was precipitated from 0.1 *N* hydrochloric acid solution with gaseous ammonia. The precipitate was centrifuged and dissolved in 50 microliters of 0.1 *N* sulfuric acid. Upon addition of 100 microliters of saturated potassium sulfate solution, a white crystalline precipitate, probably a double salt of actinium and potassium sulfates, was formed. The X-ray diffraction pattern has not yet been interpreted.

X-Ray Diffraction Studies

As mentioned above, fogging of the X-ray film from gamma radiation emitted by the short-lived daughters of actinium was troublesome. This difficulty was overcome by the use of intense X-ray beams, by taking the diffraction data within a period of twelve hours after the initial separation of actinium from its daughter elements, and by using very small samples to minimize X-ray absorption. The weight of the samples varied from 2 to 10 micrograms. The diffraction patterns were obtained using exposure times of two and one-half hours and copper K-radiation filtered through nickel foil.

The X-ray diffraction studies of the various actinium preparations led to conclusive identification of nine different compounds of actinium. The experimental data and the details of the structural analysis will be given in subsequent papers dealing with the crystal structure of these compounds. All the definitely identified substances proved to be compounds of trivalent actinium, and they are all isostructural with the analogous compounds of lanthanum. The unit cell dimensions are consistently larger for the actinium compounds than for the corresponding lanthanum compounds.

The results of the crystal structure studies of the actinium preparations are summarized in Table I. The unit cell dimensions of the isostructural lanthanum compounds have been included in order to illustrate the close crystal chemical similarity between actinium and lanthanum.

Complete crystal structures have been deduced for all the compounds listed in Table I. It has thus been possible to deduce the crystal radius of the trivalent actinium ion. The value found for the radius of Ac^{+3} is 1.11 Å., which should be compared with the radius of 1.04 Å. for La^{+3}. These two ions are the two largest trivalent cations in existence. Since both ions have electronic configurations corresponding to inert gases, and since the crystal radii differ by only 0.07 Å., it is to be expected that the crystal chemical properties of actinium and lanthanum will show a corresponding similarity.

Table II gives a survey of the crystal chemical relationship of actinium to other trivalent elements. A cross indicates that the compound is isostructural with the corresponding actinium compound, a circle that it is not. No entry indicates either that the compound does not exist, or that it has not been investigated.

The crystal structure studies provide proof of the correctness of the prediction made by Niels Bohr[5] twenty-five years ago that a 5f-series of elements begins near the end of the periodic system. The 5f-elements, to the extent they are now known, show prominent valence states of four and three. Thorium is the prototypic element for the tetravalent, actinium for the trivalent series. The crystal radii of the 5f- and of the 4f-series of elements are shown in Table III as taken from a recent publication.[6] The filling of the f-shells results in a monotonic decrease in crystal radius with increasing atomic number. Table III demonstrates this "lanthanide" contraction (first drawn attention to by V. M. Goldschmidt[7]) and the corresponding "thoride" and "actinide" contractions of the 5-f series of elements.

(5) Bohr, *Nature*, **112**, 29 (1923).

(6) Zachariasen, *Phys. Rev.*, **73**, 1104 (1948).

(7) Goldschmidt, "Geochemische Verteilungsgesetze," Oslo, 1925.

TABLE I

CRYSTAL STRUCTURE DATA FOR ACTINIUM AND LANTHANUM COMPOUNDS

Compound	Symmetry	Structure type	Unit cell dimensions a₁, Å.	a₂, Å.	Calculated density
AcF_3	Hexagonal	LaF_3	4.27 ± 0.01	7.53 ± 0.02	7.88
LaF_3	Hexagonal	LaF_3	4.140 ± .001	7.336 ± .001	5.93
$AcCl_3$	Hexagonal	UCl_3[a]	7.62 ± .02	4.55 ± .02	4.81
$LaCl_3$	Hexagonal	UCl_3	7.468 ± .003	4.366 ± .003	3.84
$AcBr_3$	Hexagonal	UCl_3	8.06 ± .04	4.68 ± .02	5.85
$LaBr_3$	Hexagonal	UCl_3	7.951 ± .003	4.501 ± .003	5.07
AcOF	Cubic	CaF_2	5.931 ± .002		8.28
LaOF	Cubic	CaF_2	5.76 ± .02		6.00
AcOCl	Tetragonal	PbFCl	4.24 ± .02	7.07 ± .03	7.23
LaOCl	Tetragonal	PbFCl	4.113 ± .003	6.871 ± .009	5.41
AcOBr	Tetragonal	PbFCl	4.27 ± .02	7.40 ± .03	7.89
LaOBr	Tetragonal	PbFCl	4.147 ± .003	7.376 ± .012	6.11
Ac_2O_3	Hexagonal	La_2O_3	4.07 ± .01	6.29 ± .02	9.19
La_2O_3	Hexagonal	La_2O_3	3.93 ± .01	6.12 ± .02	6.57
Ac_2S_3	Cubic	Ce_2S_3[b]	8.97 ± .01		6.75
La_2S_3	Cubic	Ce_2S_3	8.706 ± .001		5.01
$AcPO_4 \cdot {}^1/_2H_2O$	Hexagonal	$LaPO_4 \cdot {}^1/_2H_2O$[c]	7.21 ± .02	6.64 ± .03	5.48
$LaPO_4 \cdot {}^1/_2H_2O$	Hexagonal	$LaPO_4 \cdot {}^1/_2H_2O$	7.081 ± .005	6.468 ± .008	4.28

[a] W. H. Zachariasen, *J. Chem. Phys.*, **16**, 254 (1948). [b] W. H. Zachariasen, *Acta Crystall.*, **2**, 57 (1949). [c] R. C. L. Mooney *J. Chem. Phys.*, **16**, 1003 (1948).

TABLE II

SURVEY OF CRYSTAL CHEMICAL RELATIONSHIPS OF ACTINIUM AND OTHER TRIVALENT IONS

Compound	Ac	U	Np	Pu	Am	La	Ce	Pr	Nd
XF_3	x	x	x	x	x	x	x	x	x
XCl_3	x	x	x	x	x	x	x	x	x
XBr_3	x	x	x	0	0	x	x	x	0
XOF	x			x		x			
XOCl	x			x		x		x	x
XOBr	x			x		x		x	x
X_2O_3	x					x	x	x	x
X_2S_3	x	0	0	x	x	x	x		
$XPO_4 \cdot 0.5H_2O$	x					x	x	x	x

TABLE III

CRYSTAL RADII, Å.

Number of 4f or 5f electrons	Thoride series	Actinide series	Lanthanide series
0	Th^{+4} 0.95	Ac^{+3} 1.11	La^{+3} 1.04
1	Pa^{+4} (0.91)	(Th^{+3} 1.08)	Ce^{+3} 1.02
2	U^{+4} .89	(Pa^{+3} 1.06)	Pr^{+3} 1.00
3	Np^{+4} .88	U^{+3} 1.04	Nd^{+3} 0.99
4	Pu^{+4} .86	Np^{+3} 1.02	61^{+3} (0.98)
5	Am^{+4} .85	Pu^{+3} 1.01	Sm^{+3} 0.97
6		Am^{+3} 1.00	Eu^{+3} 0.97

Discussion

Direct observation of the chemical behavior of the pure actinium compounds emphasizes the similarity between actinium and lanthanum. Qualitatively, actinium is apparently more basic, since its compounds are more difficult to hydrolyze than the corresponding lanthanum compounds. The volatilities of the various actinium halides seem to be of about the same order of magnitude as those of the lanthanum halides and it probably will be found that the melting points will also be similar. In all cases where the compounds could be identified they were found to be isomorphous with the corresponding lanthanum compounds and no indication of other than the trivalent state was observed. The results of these experiments are entirely in agreement with the conclusions which are drawn from tracer studies, namely, that actinium is a higher and more basic homolog of lanthanum.

Acknowledgment.—The authors wish to take this opportunity to thank Miss Anne Plettinger, who made all the X-ray exposures, for working continuously on each set of compounds until the pictures were completed, obviating the necessity for repeating many of the purifications.

Summary

1. Nine compounds of actinium, AcF_3, $AcCl_3$, $AcBr_3$, AcOF, AcOCl, AcOBr, Ac_2S_3, Ac_2O_3 and $AcPO_4 \cdot 0.5H_2O$ have been prepared on the microgram scale and identified by analysis of their X-ray diffraction patterns.
2. Attempts to prove the identity of the compounds thought to be the iodide, hydroxide, oxalate, oxyiodide and double salt with potassium sulfate, failed, either in that no X-ray diffraction pattern was obtained or that interpretation has not yet proved possible.
3. The compounds that have been identified are all isomorphous with the corresponding lanthanum compounds and exhibit only the trivalent state.
4. The lattice dimensions, crystal radii and the crystal systems of nine identified actinium compounds are given along with a survey of the crystal chemical relationships among the heavy elements and rare earths.

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RECEIVED MAY 19, 1949