The microcrystalline test employing $[Co(NH_3)_6](NO_3)_3$ and sodium fluoride[9] is also given by both Sc^{III} and Th^{IV}.

The reagent suggested by Kuznetsov[14]—the Schiff base of *o*-aminophenylarsonic acid and salicylaldehyde—which gives a yellow color in the presence of Sc^{III} is just as satisfactory as a test for Th^{IV}.

With Morin[15] Sc^{III} gives a green fluorescence in ultraviolet light. Th^{IV} is also reported to give fluorescence, but weaker than that of Sc^{III}. Beck[15] also recommended sodium alizarin sulfonate as a reagent for Sc^{III} but did not mention Th^{IV} in this connection. Tests have shown that Th^{IV} gives the same crimson to violet color, and (on standing) violet precipitate, as does Sc^{III}.

With murexide[16] (ammonium purpurate) Sc^{III} gives a purple to yellow color change. Th^{IV} gives the same test, except in the presence of sulfate ion. However the addition of nitrilotriacetate, $N(CH_2COOH)_3$, to the Sc^{III} system regenerates the color of the reagent, indicating that the $Sc-N(CH_2COOH)_3$ complex is more stable than the murexide complex. The latter reaction does not occur for Th^{IV}.

Acknowledgment.—The work described herein was supported in part by a Frederick Gardner Cottrell Grant from the Research Corporation, whose financial assistance is gratefully acknowledged.

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(14) V. I. Kuznetsov, *J. Gen. Chem.* (*U. S. S. R.*), **14**, 897 (1944).

(15) G. Beck, *Mikrochim. Acta*, **2**, 9 (1937).

(16) G. Beck, *Anal. Chim. Acta*, **1**, 69 (1947).

CHICAGO 16, ILL.

RECEIVED APRIL 26, 1950

[CONTRIBUTION FROM THE OAK RIDGE NATIONAL LABORATORY, CHEMISTRY DIVISION]

Anion Exchange Studies. I. Separation of Zirconium and Niobium in HCl–HF Mixtures[1]

BY KURT A. KRAUS AND GEORGE E. MOORE

The specificity of anion exchange resins for negatively charged ions suggests their use for studies of negatively charged complexes of metal ions and for separations of metals which form such complexes. Anion exchange resins should be particularly useful with metal ions which would precipitate if not complexed (*e. g.*, 4th and 5th group elements), where cation exchange resins could not be used to full advantage. This paper, which deals with the separation of zirconium and niobium, is the first one of a series[2] dealing with these two groups of elements. Although the studies were restricted to HF–HCl mixtures it is expected that similarly effective separations could be attained with other complexing agents.

Experimental

Columns, Column Operation, Materials.—The adsorption columns were constructed of "Transflex" tubing with cross-sectional area *ca.* 0.03 sq. cm., as determined by weighing a column of water of known length. The resin used was Dowex-1, a "strong-base," quaternary amine polymer of mesh size 200–230 as obtained by wet screening. Only the chloride form of the resin was used. The eluent was contained in 50-ml. bakelite burets and the height of the liquid column was occasionally adjusted to maintain approximately the desired flow rate. It was not attempted to maintain constant flow rates. Instead, the volume of eluent which had passed was determined from the buret readings at frequent intervals.

In most cases the zirconium and niobium were added in a small volume to a column which had previously been equilibrated with the desired acid mixture. The added solution was of approximately the same composition as the solution in the column and the eluent. In a few cases the additions were carried out with the column liquid at a composition to cause strong adsorption, and the material was then eluted by changing the composition of the eluent.

The experiments were carried out with tracer zirconium (Zr^{95}, β-emitter $T_{1/2}$ = 65 d.)[3] and tracer niobium (Nb^{95}, β-emitter, $T_{1/2}$ = 35 d.),[3] which were obtained by neutron bombardment of spectroscopically pure zirconium metal[4] in the ORNL reactor.[5] The metal was dissolved in HF–HCl mixtures. Since Nb^{95} is the daughter of Zr^{95}, it was "carrier-free" while by necessity some inactive zirconium was present (*ca.* 0.5 mg. per experiment). The experiments were carried out in a thermostated room at 25 ± 1°. C. P. chemicals were used throughout.

Analytical Procedures. Direct Scanning of Columns.—The data were obtained through continuous monitoring of the activity of the effluent,[6] and through direct scanning of the columns with a detector to locate the activities (bands) and determine their elution rates. After elution identity of the tracers was established through standard radiochemical assays.

The determination of elution rates by scanning offers a number of advantages over the method of continuous monitoring of the effluent. Thus the elution of a previously identified activity can be determined rapidly and with relatively high precision, since the bands in general are quite sharp. Except in the most unfavorable cases a precision of better than 10% is attainable with the band moving through less than 1 cm. of column. Thus considerable time can be saved particularly when the elution rate of a material is unknown and when the optimum length of the column cannot be predicted. It is possible to measure elution rates of several activities at the same time and under identical conditions. Furthermore, the same band (or series of bands) can be studied under a variety of eluting conditions by changing the composition of the

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(1) This document is based on work performed for the Atomic Energy Commission at Oak Ridge National Laboratory.

(2) In two earlier communications (K. A. Kraus and G. E. Moore, THIS JOURNAL, **71**, 3263, 3855 (1949)) the method was shown to be successful for the separation of zirconium and hafnium, and niobium and tantalum. Since then the zirconium-hafnium separation was confirmed by E. H. Huffman and R. C. Lilly (*ibid.*, **71**, 4147 (1949)).

(3) Information from "Table of Isotopes," by G. T. Seaborg and I. Perlman, *Rev. Mod. Phys.*, **20**, 585 (1948).

(4) The only appreciable impurity was hafnium. It yields Hf^{181} (β-emitter, $T_{1/2}$ = 46 d.[3]) on bombardment. In most cases its behavior was very similar to zirconium and, as a first approximation, all results for zirconium also apply for hafnium. Details for their separation will be given in a later paper. Since in most cases discussed here relative enrichment of zirconium and hafnium was not determined, the uncertainty in the reported elution constants for Zr is relatively high.

(5) For some of the earlier work fission product Nb^{95} was used which was kindly furnished us by W. H. Baldwin and co-workers who isolated it from fission product mixtures.

(6) K. A. Kraus and G. E. Moore, THIS JOURNAL, **72**, 4293 (1950).

eluent as soon as elution rates are established with sufficient precision. Using the scanning technique columns of standard length (*e. g.*, 100 cm.) can be selected which are long enough to permit satisfactory measurement for rapidly moving bands. The method can furnish a promising check of adsorption theories, particularly changes of band characteristics with elution rate or distance.

For scanning a G–M tube was first mounted on a cathetometer stand. Collimation was achieved with a 0.25 inch lead shield containing a narrow slit. The tube was connected to a standard G–M scaler circuit. The shape and position of the bands was obtained from the counting rate as a function of column distance. In view of its usefulness the method was adapted for continuous operation. The automatic scanner is illustrated in Fig. 1. In this instrument the detector (G–M tube or scintillation counter) is driven by a guide screw and its output fed into a scaler with integrating device (count-rate meter) whose output in turn is recorded on a Brown Electronik Strip Chart recorder. The guide screw is driven by a synchronous motor through a gear box which permits scanning rates of 0.5, 1, 2, 4 and 8 in./min. The carriage also holds a reversing switch which is tripped by two adjustable tripping plates. This reversing switch also controls the power input of the recorder, so that it operates only during the downward travel of the carriage. The results of a typical scanning experiment are shown in Fig. 2 which is a reproduction of several scanning intervals taken from a continuous record. In Fig. 3 the band positions (maxima) in each consecutive interval are given together with the buret readings from which the elution constant E (see below) was calculated.

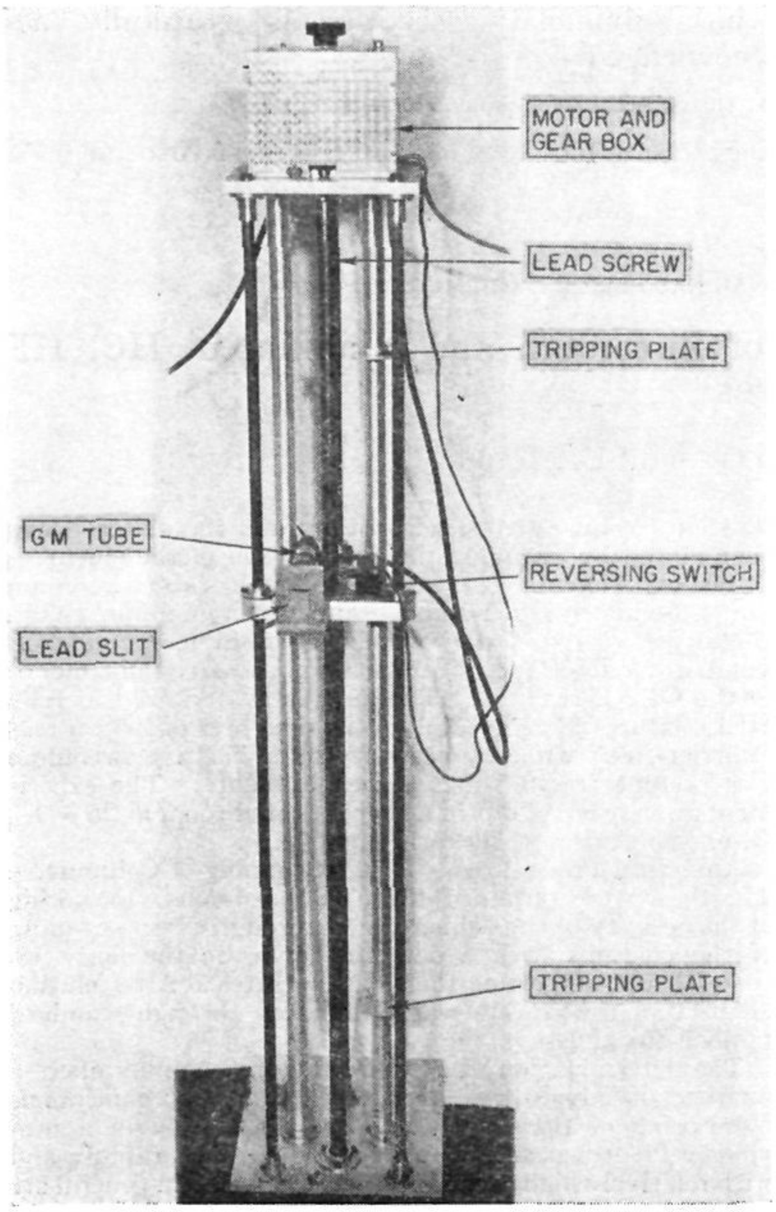

Fig. 1.—Automatic scanner.

Results and Discussion

Elution Constants of Zirconium(IV) and Niobium(V) in HF–HCl Mixtures.—The results of the experiments will be described in terms of the elution constant E, defined by the equation

$$E = dA/V \qquad (1)$$

where d is the distance in cm. a band maximum travels after V cc. of eluent have passed through a column of cross-sectional area A sq. cm.[7] The velocity with which an adsorption band moves through an adsorbent bed under equilibrium conditions is given by Ketelle and Boyd[8,9] as

$$d/t = v/(1 + D/i) \qquad (2)$$

where t is the time, v the linear flow rate of the solution, i the interstitial (fractional void) space and D the bed distribution coefficient (amount per cc. of bed/amount per cc. of solution).[10] Since

$$tv = V/iA \qquad (3)$$

combination of equations (1), (2) and (3) yields

$$E = 1/(i + D) \qquad (4)$$

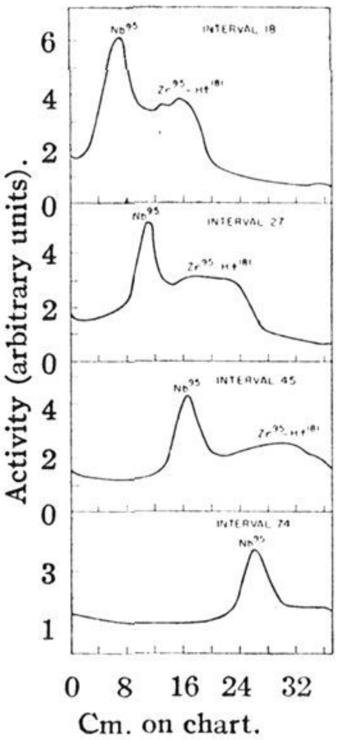

Fig. 2.—Typical intervals from continuous scanning record: Dowex-1 resin, 0.5 M HCl–0.5 M HF, motor speed 1 in./min., 1.00 cm. chart = 2.00 cm. column, flow rate = 0.26 ml. cm.$^{-2}$ min.$^{-1}$.

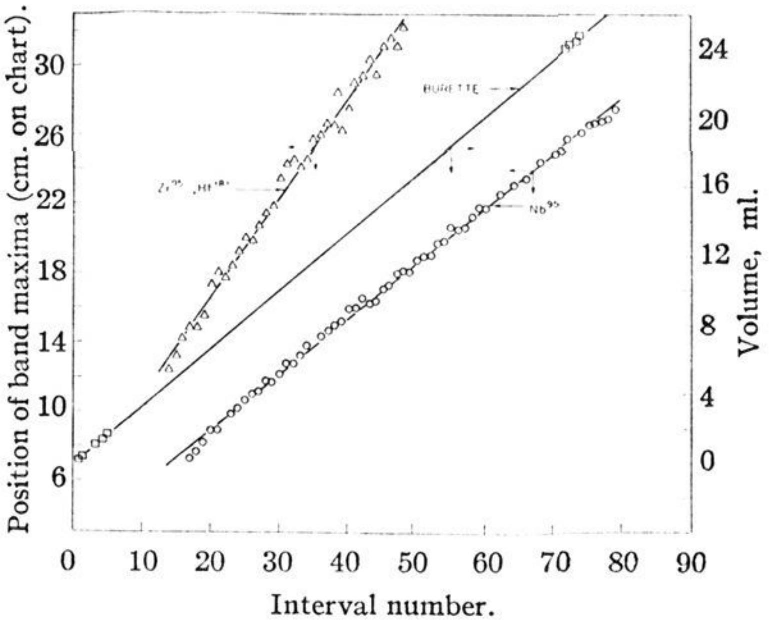

Fig. 3.—Determination of elution constant with automatic scanner: Dowex-1 resin, 0.5 M HCl–0.5 M HF, motor speed 1 in./min., average chart interval 36.86 cm., average column interval 73.69 cm., 1.00 cm. chart = 2.00 cm. column, flow rate 0.26 ml. cm.$^{-2}$ min.$^{-1}$.

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(7) E is closely related to the R value frequently used in chromatography which is defined as the ratio of the linear velocities of adsorption band to eluent (A. L. LeRosen, THIS JOURNAL, **64**, 1905 (1942); H. H. Strain, *Anal. Chem.*, **21**, 75 (1949); **22**, 41 (1950)).

(8) B. H. Ketelle and G. E. Boyd, THIS JOURNAL, **69**, 2800 (1947).

(9) This can also be derived from the theory of A. J. P. Martin and R. L. M. Synge, *Biochem. J.*, **35**, 1358 (1941).

(10) For column work D appears to be more useful than K_d, the distribution coefficient in the units amounts per gram oven-dried resin/amounts per cc. of solution. The relationship between these two distribution coefficients is $D = \rho K_d$ where ρ is the bed density (g. oven-dried resin/cc. of bed).

which for $D \gg i$ reduces to

$$E \approx E^* = 1/D \qquad (5)$$

Similarly as pointed out by Ketelle and Boyd,[8] i can be determined by measuring E for a non-adsorbed species (*e. g.*, a positively charged ion for an anion exchanger).

The elution constants of zirconium(IV) and niobium(V) (E_{Zr} and E_{Nb}) were determined for several series of HCl–HF mixtures. The results are summarized in Table I. Zirconium(IV) was found to be very weakly adsorbed under most conditions studied. In 3 and 9 *M* HCl, at all HF concentrations studied, it has an elution constant (E_{Zr}) which is within a factor of 2 or 3 of the maximum (*ca.* 3, since i = *ca.* $^1/_3$). At lower chloride concentrations zirconium becomes rapidly strongly adsorbed ($E_{Zr} = 8.5 \times 10^{-3}$ in 0.2 *M* HCl–0.5 *M* HF). Although the data are fragmentary, it appears that E_{Zr} is independent of HF concentration in the regions studied, suggesting that a single species predominates in these solutions.

TABLE I

ELUTION CONSTANTS OF ZIRCONIUM(IV) AND NIOBIUM(V) IN HF–HCl MIXTURES

		Elution constants		
M HCl	*M* HF	$E_{Zr} \times 10^3$	$E_{Nb} \times 10^3$	E_{Zr}/E_{Nb}
9	0.004	*ca.* 2000	3.0 ± 0.3^a	670
9	.01	.....	6.2	...
9	.02	.....	19	...
9	.05	*ca.* 3000	78	38
9	.1	.....	220	...
9	.18	.....	230	...
9	.5	2–3000	72 ± 2	35
9	1.0	.....	27	...
9	6.9	*ca.* 2000	18	110
6	0.5	*ca.* 1000	150 ± 10^a	7
3	.1	*ca.* 1500	190	8
3	.5	*ca.* 1000	180 ± 10^a	6
3	2.0	*ca.* 1000	110	9
3	5.0	*ca.* 1000	43	23
3	10.0	.....	21	...
3	17.3	.....	24	...
1	0.5	170	73	2.3
0.5	.05	.....	39	...
.5	.1	65 ± 15^a	40 ± 1^a	1.6
.5	.5	65 ± 15^a	45 ± 5^a	1.5
.2	.5	8.5	11.5 ± 1.5^a	0.74

[a] Averages of two or three determinations with maximum deviation given.

The elution constants of niobium (E_{Nb}) vary both with HF and HCl concentrations. Thus in 9 *M* HCl E_{Nb} is small at low fluoride concentrations (3.0×10^{-3} in 0.004 *M* HF), rises with increasing HF concentration, passes through a maximum near 0.1 *M* HF and then rapidly decreases (see Fig. 4). In 3 *M* HCl data have been obtained only at HF concentration equal to or larger than 0.1 *M* in which range E_{Nb} decreases up to 17 *M* HF. E_{Nb} is independent of HF concentration in 0.5 *M* HCl–0.1 to 0.5 *M* HF.

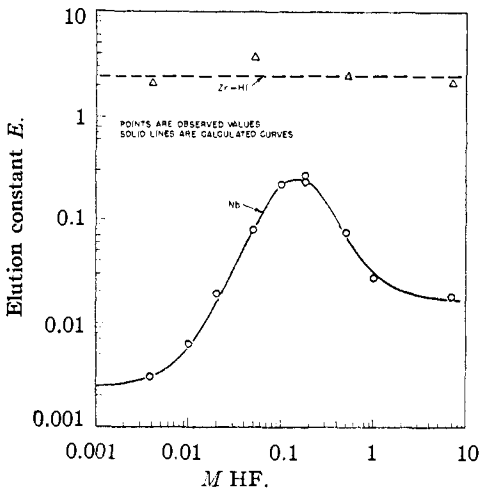

Fig. 4.—Elution constants as a function of HF concentration in 9 *M* HCl.

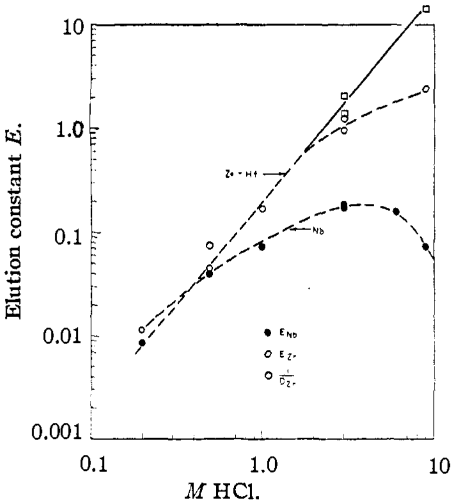

Fig. 5.—Elution constants as a function of HCl concentration in 0.5 *M* HF.

On the basis of these data separation of zirconium and niobium should be possible under a variety of conditions. Difficulties would occur only at low HCl concentration where E_{Zr} and E_{Nb} become approximately equal. In fact between 0.2 and 0.5 *M* HCl in 0.5 *M* HF there is an inversion of the two elution constants, E_{Zr} being larger in 0.5 *M* HCl and E_{Nb} in 0.2 *M* HCl (see Fig. 5). At an intermediate HCl concentration E_{Zr} is equal to E_{Nb} and separation would become impossible. Efficiency of separation of these elements is thus critically dependent on the composition of the eluent. It can be seen from Fig. 5 that in 0.5 *M* HF, E_{Zr} increases approximately with the square of the HCl concentration, while E_{Nb} increases at first approximately linearly, passes through a maximum and then sharply decreases. The ratio E_{Zr}/E_{Nb}, which determines the efficiency of separation, thus increases greatly with increasing HCl concentration. Furthermore until E_{Nb} passes through the maximum, the apparent paradox exists that separation becomes more effective as the elution rate of the slowest component increases. Typical separations are illustrated in Fig. 6 which shows elution curves transcribed from automatic records of the activity of the effluent. It may be noticed that under some conditions the separations are sufficiently good as to permit the use of extremely short columns, make batch separations very effective and permit economical operation of columns under break-through conditions.

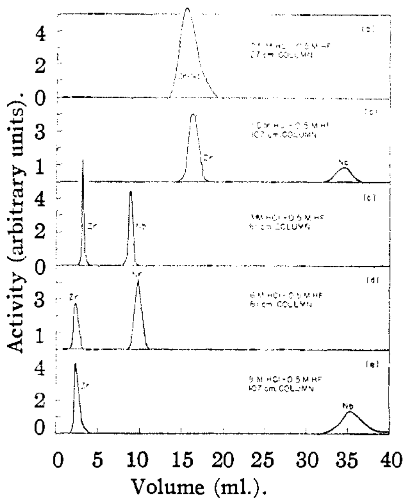

Fig. 6.—Separation of Zr and Nb with Dowex-1 anion exchange columns: average flow rate *ca.* 0.3 ml./sq. cm./min.; cross-section of column: 0.0226 sq. cm.

Ionic Species.—Assuming that the adsorption equilibrium of an ion A^{-n} can be represented by the equation[11]

$$n\mathrm{RCl} + \mathrm{A}^{-n} \rightleftharpoons \mathrm{R}_n\mathrm{A} + n\mathrm{Cl}^- \qquad (6)$$

(where R represents the resin) one obtains the mass action constant

$$K_{\mathrm{A}^{-n}} = \frac{[\mathrm{R}_n\mathrm{A}]}{[\mathrm{RCl}]^n}\,\frac{(\mathrm{Cl}^-)^n}{(\mathrm{A}^{-n})}\,\frac{\gamma^n_{\mathrm{Cl}^-}}{\gamma_{\mathrm{A}^{-n}}} \qquad (7)$$

where brackets indicate activities, parentheses concentrations and where γ is the activity coefficient of the ion given as subscript. For tracer solutions the simplifications

$$\begin{aligned}[\mathrm{R}_n\mathrm{A}] &= \alpha(\mathrm{A}^{-n})_\mathrm{r} \text{ and}\\ [\mathrm{RCl}]^n &= \text{constant}\end{aligned} \qquad (8)$$

will be assumed where α is a proportionality constant and $(\mathrm{A}^{-n})_\mathrm{r}$ the concentration of A^{-n} in the resin. Combining equations (4), (5), (7) and (8) and remembering

$$D_{\mathrm{A}^{-n}} = \frac{(\mathrm{A}^{-n})_\mathrm{r}}{(\mathrm{A}^{-n})} \qquad (9)$$

one obtains

$$D_{\mathrm{A}^{-n}} = K^*_{\mathrm{A}^{-n}}\,\frac{\gamma_{\mathrm{A}^{-n}}}{\gamma^n_{\mathrm{Cl}^-}}\,\frac{1}{(\mathrm{Cl}^-)^n} = \frac{1 - Ei}{E} = \frac{1}{E^*} \approx \frac{1}{E} \qquad (10)$$

where $K^*_{\mathrm{A}^{-n}} = K_{\mathrm{A}^{-n}}[\mathrm{RCl}]^n/\alpha$ is a proportionality constant. Thus, neglecting the activity coefficient ratio, a plot of log E^* (or log E for small values of E) *vs.* log (Cl^-) for a single (tracer) species has a slope equal to the charge n of the ion. For two ions of different charge two straight lines will result which cross at some hydrochloric acid concentration P. The order of elution will depend on the relative magnitude of the chloride concentration of the eluent and of P. Thus for $(\mathrm{HCl}) < P$ the more highly charged ion elutes last (is more strongly adsorbed) and for $(\mathrm{HCl}) > P$ it elutes first.

On the basis of the following observations it appears probable that P is considerably smaller than 1 M and possibly smaller than 0.1 M for the type of complexes studied here and that hence in strong hydrochloric acid the more strongly adsorbed ions carry the smaller negative charge and most probably have a charge of minus one. Thus P for the very similar pair of ions $\mathrm{HSO_4^-}$–$\mathrm{SO_4^=}$ was found to be small (*ca.* 0.05).[12] A tantalum complex in HF–HCl solutions for which log E *vs.* log (HCl) was approximately unity, suggesting a charge of minus one, had a very small E (*ca.* 5×10^{-3}) in 9 M HCl.[13] Conversely the zirconium complex studied here for which log E *vs.* log (HCl) was considerably larger than unity, suggesting a charge more negative than minus one, had a large value of E (E^* = *ca.* 10 in 9 M HCl).[14] Addition of hydrogen fluoride to concentrated hydrochloric acid solutions of some ions which are generally considered to be strongly complexed by fluoride ions, causes a marked increase of E.[15] It appears reasonable to assume that increased complexing is usually paralleled by an increase in negative charge.

The complicated hydrogen fluoride dependence of E_{Nb} necessitates assumption of at least three species. Species I would exist (in 9 M HCl) only at very low fluoride concentrations and may not contain fluoride ions. Formation of species II would account for the increase of E with increasing fluoride concentration. The subsequent decrease of E at still higher fluoride concentrations would be due to the formation of species III. On the basis of the above considerations one could tentatively assign a charge of minus one to species I and III and a greater negative charge to species II.[16]

The probable general equation for the formation of species II from species I is

$$\mathrm{NbX_6^-} + n\mathrm{HF} + j\mathrm{H_2O} \rightleftharpoons \mathrm{H}_{n-j}\mathrm{NbX}_{6-m}\mathrm{F}_n{}^{-1-j+m} + m\mathrm{X}^- + j\mathrm{H_3O^+} \qquad (11)$$

with the (molarity) equilibrium constant

$$K^*_{12} = (2)(\mathrm{X}^-)^m(\mathrm{H_3O^+})^j/(1)(\mathrm{HF})^n \qquad (12)$$

where (1) and (2) represent the concentration of species I and II. At constant hydrochloric acid concentration equation (12) reduces to

$$K_{12} = K^*_{12}/(\mathrm{X}^-)^m(\mathrm{H_3O^+})^j = (2)/(1)(\mathrm{HF})^n \qquad (13)$$

For the formation of species III from species II the analogous expression for the equilibrium constant is

$$K_{23} = K^*{}_{23}/(\mathrm{X}^-)^{m'}(\mathrm{H_3O^+})^{j'} = (3)/(2)(\mathrm{HF})^{n'} \qquad (14)$$

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(11) A. P. Vanselow, This Journal, **54**, 1307 (1932).

(12) K. A. Kraus and E. Rona, unpublished results.

(13) K. A. Kraus and G. E. Moore, This Journal, **73**, 13 (1951).

(14) log E^* *vs.* log (HCl) has a slope of approximately two for zirconium. The conclusion that the charge of the complex is minus two (*i. e.*, $\mathrm{ZrX_6^=}$ or $\mathrm{HZrX_7^=}$), however, does not appear warranted since the change of the activity coefficient ratio $\gamma_{\mathrm{A}^{-n}}/\gamma^n_{\mathrm{Cl}^-}$ is not known. A charge of minus three appears equally probable (*i. e.*, $\mathrm{ZrX_7^{\equiv}}$).

(15) This is the case with tantalum (ref. 13) and protactinium (K. A. Kraus and G. E. Moore, unpublished results) as well as with niobium at low M HF, but apparently not with niobium at high M HF as will be discussed below.

(16) It should be noted, however, that assumption of specific charges is unnecessary for the evaluation of the fluoride dependence.

Defining

$$D_1 = (1)_r/(1);\ D_2 = (2)_r/(2);\ D_3 = (3)_r/(3) \text{ and } D_0 = T_r/T \quad (15)$$

where D_0 is the observed distribution coefficient, D_1, D_2 and D_3 those of the "pure" species and T_r and T the concentrations of niobium in the resin and solution, respectively, setting (HF) = f and combining equations (13), (14) and (15) one obtains

$$D_0 = (D_1 + D_2K_{12}f^n + D_3K_{12}K_{23}f^{n+n'})/(1 + K_{12}f^n + K_{12}K_{23}f^{n+n'}) \quad (16)$$

In the limits, *i. e.*, when the two reactions do not overlap, equation (16) reduces to

$$D_0 = (D_1 + D_2K_{12}f^n)/(1 + K_{12}f^n)$$

and

$$D_0 = (D_2 + D_3K_{23}f^{n'})/(1 + K_{23}f^{n'}) \quad (17)$$

The 9 M HCl data could not be fitted to equation (16) taking $n = 1$ and $n' = 1$. Satisfactory agreement was obtained using $n = n' = 2$ as shown by the solid line Fig. 4 which represents the calculated values. The constants $D_1 = 415$, $D_2 = 1.0$, $D_3 = 60$, $K_{12} = 1.5 \times 10^4$ and $K_{23} = 1.25$ were evaluated by trial and error. They can only be considered rough approximations in view of the limited number of data available. An analogous calculation for the 3 M HCl data was not attempted since the slight increase in E at very high HF concentration suggests the formation of a fourth species. However, it is apparent that K_{23} must be at least a factor of 10 smaller in 3 M HCl than in 9 M HCl. Since niobium is known to form oxygenated ions (of the type $NbOX_n^{+3-n}$)[17] it appears reasonable to assume that species I and II contain one coördinated oxide ion. This would explain the great difference in anion exchange behavior of niobium and tantalum.[2,13,18] In species III the oxide ion has probably been replaced by fluoride ions. This would make plausible the great decrease in K_{23} with decreasing HCl concentration and the decrease in E as species III is formed since it would give a mechanism for a decrease in negative charge on addition of fluoride ions.

Acknowledgment.—It is a pleasure to acknowledge the valuable technical assistance of Mrs. Hazel Templeton and to thank Messrs. J. East, F. W. Manning, P. P. Williams and the Instrument maintenance group under R. E. Toucey for the assembly and the maintenance of the electronic equipment, Mr. F. King for the construction of the automatic scanner, and Dr. G. E. Boyd for many valuable discussions.

Summary

1. The behavior of zirconium(IV) and niobium(V) on the anion exchange resin Dowex-1 was studied in a number of HF–HCl mixtures.

2. Separation of the two elements is excellent under a number of conditions. Although zirconium is usually eluted first, the elements can be eluted in reverse order under some conditions.

3. Attempts have been made to interpret the data in terms of ionic species and their equilibria. In the case of niobium at least three species were observed in 9 M HCl differing by two fluoride ions each. The first two species, existing at low fluoride concentration, are probably oxygenated.

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[17] A. F. Wells, "Structural Inorganic Chemistry," Clarendon Press, Oxford, 1945, p. 424.

[18] In ref. (2) this difference was attributed to a difference in charge between the niobium and tantalum complexes and it was assumed that the more strongly adsorbed tantalum carries a greater negative charge. This was equivalent to the assumption that the cross-over point P would occur at chloride concentrations so high as to be practically unattainable in aqueous solutions. As pointed out above, this assumption now appears very unlikely.

OAK RIDGE, TENN. RECEIVED MAY 15, 1950

[CONTRIBUTION FROM THE OAK RIDGE NATIONAL LABORATORY, CHEMISTRY DIVISION]

Anion Exchange Studies. II. Tantalum in Some HF–HCl Mixtures[1]

BY KURT A. KRAUS AND GEORGE E. MOORE

In the previous paper[2] results on the anion exchange behavior of niobium and zirconium in HCl–HF mixtures were given. This paper will deal with a similar study of tantalum and the subsequent one will deal with protactinium and a general discussion of the separation of the triad niobium, tantalum and protactinium.[3]

Experimental

Details of the general experimental procedure were given earlier.[2] Suffice it to repeat that the studies were carried out with Dowex-1 columns of *ca.* 0.023 sq. cm. cross-section at 25 ± 1°. Ta^{182} tracer (β-emitter, $T_{1/2} = 117$ d.)[4] was used, obtained by slow neutron bombardment of spectroscopically pure tantalum metal. Identity of the tracer was checked by standard radiochemical techniques. Elution rates were determined by monitoring the effluent and by direct scanning of the columns.

Results and Discussion

The results of the experiments are summarized in Table I where the molarities of HCl and HF of the eluents are given and the observed values of the elution constant E_{Ta} defined[2] as

$$E_{Ta} = dA/V \quad (1)$$

where d is the distance (cm.) the band maximum moves after passage of V ml. of eluent through a

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[1] This document is based on work performed for the Atomic Energy Commission at Oak Ridge National Laboratory.

[2] K. A. Kraus and G. E. Moore, THIS JOURNAL, **73**, 9 (1951).

[3] For a preliminary communication on the separation of niobium and tantalum see K. A. Kraus and G. E. Moore, *ibid.*, **71**, 3855 (1949).

[4] Information from "Table of Isotopes," by G. T. Seaborg and I. Perlman, *Rev. Mod. Phys.*, **20**, 585 (1948).