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**SEPARATION OF METAL IONS BY
ANION EXCHANGE IN MIXTURES OF
HYDROCHLORIC ACID AND HYDROFLUORIC ACID**

by

J. P. Faris

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS

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Analytical Chemistry Laboratory
Chemical Engineering Division

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SEPARATION OF METAL IONS BY ANION EXCHANGE IN MIXTURES OF HYDROCHLORIC ACID AND HYDROFLUORIC ACID

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ABSTRACT

Distribution coefficients were determined for the adsorption of more than 40 elements on anion-exchange resins from mixtures of HCl (0.1-12M) and HF (0.1-8M). Two resins, Dowex 1x10, 200-400 mesh and Dowex 1x4, 100-200 mesh, were used. Distribution coefficients were also determined for the adsorption of many elements on both resins from 0.1-12M HCl and 0.1-12M HF. Anion exchange in the presence of HF was found useful for separating impurities from various materials for their subsequent determination, and specific procedures used in our spectrochemical laboratory for this purpose are outlined. The results of a literature search on the use of anion exchange in hydrofluoric acid and fluoride-containing media are presented in an extensive bibliography.

I. ADSORPTION OF METAL IONS ON ANION-EXCHANGE RESINS FROM HCl-HF AND HF

A. Introduction

A considerable amount of work has been carried out in the past two or three decades to investigate the adsorption of metallic ions on anion-exchange resins from mixtures of hydrochloric and hydrofluoric acids. The early reports of Kraus, Moore, Nelson, and others at Oak Ridge National Laboratory, of Hague, Brown, Bright, and Machlan at the National Bureau of Standards and of Wish and others at the U.S. Naval Radiological Defense Laboratory contained significant information about the anion-exchange behavior of a number of elements in these acid mixtures. Numerous others, including Dixon and Headridge, de Gelis, Faris, Kalinin *et al.*, Nikitin, and Pakholkov *et al.*, have supplied additional data. Anion exchange in HCl-HF mixtures has been incorporated into separation schemes in many fields of interest, especially in analytical procedures for isolation of elements where the presence of hydrofluoric acid is desirable to prevent hydrolysis.

A common problem encountered in our analytical laboratory is the isolation of trace elements for their subsequent determination. This report describes the application of anion exchange in HCl-HF systems to the separation of trace elements from a wide variety of materials. An experimental program to measure distribution coefficients of metal ions was carried out in order to extend the available data on the anion-exchange behavior of elements from HCl-HF mixtures as fully as practical. The adsorption of over 40 elements on two anion-exchange resins, Dowex 1x10, 200-400 mesh and Dowex 1x4, 100-200 mesh, was determined from mixtures which were 0.1-12M in hydrochloric acid and 0.1-8M in hydrofluoric acid. The extent of adsorption of the elements from pure hydrochloric acid and from pure hydrofluoric acid was also determined using the same resins.

B. Experimental Method for Determination of Distribution Coefficients

The column-elution technique was employed, utilizing for the most part a series of 3/16 ID polyethylene columns. A typical column contained 0.5 g of anion-exchange resin in the chloride form (oven dried at $\sim 100^{\circ}\text{C}$) supported by a plug of plastic wool near the bottom tip. The experiments in pure hydrofluoric acid were carried out with the resin in the fluoride form. After a column was equilibrated with the proper acid or acid mixture, microgram amounts of a convenient number of elements, usually from 5 to 8, were placed in a small volume (~ 0.2 mL) of the appropriate solution and allowed to flow onto a column. Elution was continued and effluents were collected in fractions of 5 mL for analysis. A continuous elution procedure was used, adding a new group of elements as was convenient. Two sets of stock solutions of individual elements were prepared. One set contained the elements dissolved in HCl; one set contained the elements dissolved in HF. Portions of these solutions were combined to prepare appropriate stock solutions as needed. Particular care was taken to completely exclude all traces of chloride from the hydrofluoric acid solutions. The effluent fractions were spectrochemically analyzed by the copper spark method.^{1,2} An aliquot of each effluent fraction was evaporated on the flat ends of copper electrode pairs, sparked under standard conditions, and the spectra photographed in the appropriate wavelength regions. The amount of each element in an effluent fraction was visually estimated by comparing line intensities with those of a standard plate. Estimations could be made rapidly and with sufficient accuracy to permit close approximations of elution-curve maxima. Weight distribution coefficients were obtained using the relation $D = (V - i)/M$, where V is the volume in milliliters that had passed through a column when the elution curve maximum was reached, i is the first column volume of interstitial liquid, and M is the mass of resin in grams.

Determination of distribution coefficients of the more strongly adsorbed elements is impractical using the column-elution technique. Some of the values reported here were estimated by passing relatively large volumes through a column, but generally the elution was terminated after collecting 30-60 fractions, and elements that did not break through were reported as "adsorbed." Preliminary experiments³ demonstrated that quantitative spectrochemical analysis could be employed to estimate the adsorption of an element after batchwise equilibration. This procedure, however, did not offer any special advantage since radiochemical or other methods of analysis would generally be preferred for more accurate measurements of values for the more strongly adsorbed ions.

C. Results and Discussion

The experimental values for distribution coefficients are presented in Tables A-1 through A-7 in the Appendix. In Tables A-1 and A-2, weight distribution coefficients are given for the elements in mixtures of HCl (0.1-12M) and HF (0.1-8M) using Dowex 1x10, 200-400 mesh resin and Dowex 1x4, 100-200 mesh resin, respectively. Tables A-3 and A-4 give the respective results for the adsorption of elements in pure hydrochloric acid (0.1-12M) for the two resins, and Tables A-5 and A-6 give the respective results in pure hydrofluoric acid (0.1-12M) for the two resins. Additional experiments were run to determine the adsorption of some elements at lower HF concentrations, and these data are reported in Table A-7. The numbers marked with an asterisk are assigned values, estimated from curves drawn using experimental D values found at other acid

concentrations. The distribution coefficient of an element in any acid mixture within the experimental range may be similarly estimated.

The values presented in the tables were, as a rule, in agreement with literature values that are generally accepted as reliable. Elements tended to be more strongly adsorbed on the Dowex 1x10 resin than on the Dowex 1x4 resin, which has less cross-linkage.* Values are not given in the tables for the alkali and alkaline earth elements; they are not adsorbed from either acid and would not be expected to be adsorbed from a mixture.

The results originally published by this laboratory on the adsorption of elements from hydrofluoric acid⁴ have been refined for many elements and extended to 0.1M acidity. It was found that the strong adsorption of mercury(II) reported in the previous work was in error, probably due to traces of chloride in the stock solution.

II. ANALYTICAL APPLICATIONS OF ANION EXCHANGE IN THE PRESENCE OF HYDROFLUORIC ACID

A. Introduction

Anion exchange in fluoride-containing media has been applied to separations ranging from the macro level in the quantitative analysis of high-temperature alloys to the trace level after neutron activation of high-purity materials. Numerous separation schemes have been published which include anion exchange in the presence of hydrofluoric acid. An examination of the bibliography will illustrate the scope of applications.

The work of our spectrochemical laboratory in this area has been directed to the separation of trace elements for their determination in nuclear reactor materials and high-purity metals. Ion exchange is a convenient way to separate metal impurities for spectrochemical analysis since analytical determination may readily be performed at the parts-per-million level without the task of preparing standards for each type of matrix. Knowledge of the ion-exchange behavior of many elements permits the analyst to select an optimum procedure for efficient separation of the elements desired. For example, Huff⁵ used anion exchange in nitric-hydrofluoric acid to improve the procedure for determination of trace impurities in tantalum.

Some examples of procedures used in our spectrochemical laboratory are outlined below. Samples with known amounts of impurities added were frequently employed to test the efficiency of a procedure. High-purity reagents were generally used, and a control column was carried along to determine the "blank" level for the most common impurities. The nominal detection limits for elements (Table 1) were obtained using a Baird three-meter grating spectrograph.

* Distribution coefficients for elements on the Dowex 1x8 resin may be estimated from the values obtained on the Dowex 1x10 and Dowex 1x4 resins.

Table 1. Detection Limits^a for Ion Exchange-Copper Spark Method

Element	Detection Limit, ppm		Element	Detection Limit, ppm	
	100-mg Sample	1-g Sample		100-mg Sample	1-g Sample
Ag	2	0.2	Nb	4	0.4
Al	1 ^b	0.1 ^b	Nd	3	0.3
Au	4	0.4	Ni	2	0.2
Ba	1	0.1	P	50	5
Be	0.01	0.001	Pb	4	0.4
Bi	3	0.3	Pd	1	0.1
Ca	1 ^b	0.1 ^b	Pr	4	0.4
Cd	4	0.4	Pt	10	1
Ce	4	0.4	Rb	3	0.3
Co	2	0.2	Re	10	1
Cr	2	0.2	Rh	1	0.1
Cs	40	4	Ru	1	0.1
Cu ^c	5	0.5	Sb	40	4
Dy	2	0.2	Sc	0.1	0.01
Er	1	0.1	Sm	5	0.5
Eu	0.2	0.02	Sn	5	0.5
Fe	10	1	Sr	0.02	0.002
Ga	1	0.1	Ta	5	0.5
Gd	2	0.2	Tb	10	1
Hf	3	0.3	Te	50	5
Ho	2	0.2	Th	8	0.8
In	3	0.3	Ti	1	0.1
Ir	10	1	Tl	10	1
K	1	0.1	Tm	2	0.2
La	0.2	0.02	U	40	4
Li	0.01	0.001	V	1	0.1
Lu	0.1	0.01	W	5	0.5
Mg	1 ^b	0.1 ^b	Y	0.05	0.005
Mn	0.4	0.04	Yb	0.1	0.01
Mo	0.5	0.05	Zn	10	1
Na	1 ^b	0.1 ^b	Zr	1	0.1

^aImpurities in sample concentrated to 1 mL.^bLimited by amount in blank.^cGraphite spark method.⁶

B. Ion Exchange-Spectrochemical Procedures for Trace-Element Analysis Using Anion Exchange in the Presence of Hydrofluoric Acid

1. Analysis of Niobium for Trace Impurities
(J. P. Faris)

a. Experimental Procedure

Place a weighed 100-200 mg sample of niobium in a Teflon beaker and dissolve in HNO_3 -HF. Evaporate to dryness, add HF and repeat, then take up in 10-20 mL of 0.2M HF. Add sample solution to a preconditioned anion-exchange column containing resin in the fluoride form. A plastic column, 1/2 in. ID, filled with Dowex 1x8 to a height of about 4 in. is satisfactory. Elute impurities as outlined below.

- (1) Wash with about 50 mL of 0.2M HF to elute the non-adsorbed elements (Cd, Zn, Co, Ni, Cr, etc.). Wash with an additional 25 mL of 0.5M HF to elute Bi.
- (2) Wash with about 40 mL of 0.15M HCl-0.2M HF to ensure elution of Al, Be, Fe, In, V, U, Te, and Ga. This may be combined with eluate (1) above.
- (3) Wash with about 75 mL of 5M HCl-8M HF.* This elutes Mo, W, Zr, Hf, and Ti.
- (4) Wash with about 60 mL of 2M HCl-8M HF to elute Sn.†
- (5) Pour about 90 mL of 6M HCl-0.15M HF through column to elute Nb matrix. Discard.
- (6) Wash with about 75 mL of 12M HNO_3 -5M HF to elute Ta.‡
- (7) Evaporate the effluent solutions containing the impurities to a volume of 1 mL and determine the metals present by the copper spark method.^{1,2}

b. Alternative Procedure for Analysis of Niobium**
(E. A. Huff)

Dissolve sample as above and take up in a 7M HCl-4M HF acid mixture. Add sample to an anion-exchange column which has been preconditioned with this mixture.

- (1) Wash with about 60 mL of 7M HCl-4M HF. This elutes Al, Be, Ca, Cr, Hf, Mn, Ni, Pb, Ti, V, W, Zr, and other non-adsorbed elements.

* Acid mixtures are described by the actual, final concentration of each component in the mixture.

† Elution of Sn may be incomplete. This step may be omitted whenever the determination of Sn is not required.

‡ Evaporate to dryness to remove HNO_3 and take up in HCl-HF.

** This procedure is somewhat more convenient but does not allow for the analysis of Bi, Cd, Sn, or Ta.

- (2) Wash with about 40 mL of 3M HCl-8M HF. This elutes Co, Fe, Ga, Mo, and U.
- (3) Proceed as in steps 5, 6, and 7 in B.1.a above to obtain Ta and complete analysis.

2. Procedure for Determination of Niobium, Titanium, Zirconium, Tungsten, and Some Other Elements in Stainless Steel
(J. P. Faris)

Weigh 100 mg of sample and dissolve in HCl-HF mixture using a Teflon beaker. Evaporate to incipient dryness and take up in about 10 mL of 0.1M HCl-0.5M HF. Pour through a small preconditioned anion-exchange column. A Bio-Rad plastic column (0.7 × 10 cm) filled with Dowex 1×8, 100-200 mesh resin is satisfactory.

- (1) Wash well with the 0.1M HCl-0.5M HF mixture to remove Fe, Cr, Ni, Mn, etc. and discard washings. Under these conditions, Nb, Mo, W, Zr, Hf, Ti, Sn, and Ta remain on the column.
- (2) To elute the Zr, Hf, Ti, Nb, and W, wash with 6M HCl-0.15M HF until about 20 mL have been collected.
- (3) Evaporate to a small volume (1 mL) and determine the elements present by the copper spark method.^{1,2}

The Mo, Sn, and Ta remaining on the column may be recovered for analysis by washing the column with a strong nitric acid-hydrofluoric acid mixture. Elution of Mo and Sn may be completed with about 20 mL of 4M HNO₃-4M HF mixture; Ta may be eluted with a similar amount of 12M HNO₃-5M HF. Evaporate the solutions to dryness to remove any free nitric acid, take up in 1 mL of HCl-HF solution and determine the elements present by the copper spark method.

3. Analysis of Molybdenum for Trace Impurities Using Anion Exchange
(J. P. Faris)

The determination of trace impurities in molybdenum metal is not readily accomplished by direct spectrochemical analysis. The published methods are not practical for occasional samples because they may require installation of specialized equipment as well as the lengthy task of preparing adequate standards. Furthermore, no single method is applicable for the detection of every element that could likely be present in high-purity material. An anion-exchange separation procedure has been developed to isolate a number of impurities and permit their determination at the parts-per-million level by the copper spark method. Good agreement of results was obtained on one sample that was also analyzed by spark-source mass spectroscopy. The procedure is presented below:

a. Procedure for Fluoride-Soluble Elements

- (1) Weigh 100-300 mg sample, place in Teflon beaker and dissolve in distilled 7M nitric acid. Evaporate and convert to an HCl-HF medium by taking to dryness several times, adding

HCl plus a small amount of HF. Take up the final residue in ~15 mL of 6M HCl-0.1M HF mixture.

- (2) Add the sample solution to a preconditioned anion-exchange column. A plastic column, 1/2 in. ID, containing about 4 in. Dowex 1x4, 100-200 mesh resin, is suitable. Wash with 6M HCl-0.1M HF until about 100 mL of effluent is collected. This fraction (A) contains Nb, Zr, Hf, (W),* Pb, (In), V, As,[†] (Co), Al, Be, Cr, Mn, Ni, and other non-adsorbed elements.
- (3) Wash with ~25 mL 6M HCl-0.7M HF to ensure complete elution of W. This fraction (B) contains W, (Co), (In). It may be combined with fraction A for analysis.
- (4) Wash with 100 mL 1M HF. This fraction (C) contains Co, Bi, In, (Fe), Ga, Cd, Zn, (U).
- (5) Wash with 50 mL of 0.15M HCl-0.2M HF. This fraction (D) contains Te and any remaining U, Fe.
- (6) Remove molybdenum from column with about 200 mL 1M HCl.[†]
- (7) Wash with ~75 mL 12M HNO₃-5M HF. This fraction (E) contains Ta, Sn, Pd, Re.[†]
- (8) The various impurity fractions are evaporated to incipient dryness and examined visually. If no appreciable residue is observed,** fractions A and B and fractions C and D may be combined for analysis. Dissolve in HCl-HF and adjust volume of each sample to 1 mL. Determine metals present by the copper spark method.^{1,2}

b. Procedure for Determination of the Rare Earths and Thorium

Weigh a separate portion of sample and proceed as described in steps 1 and 2 of B.3.a above but use 6 to 7M HCl solution, omitting the HF. The effluent A will contain the rare-earth elements, Th, and other non-adsorbed metal ions. Evaporate to 1 mL and analyze by the copper spark method.^{1,2}

* Elements enclosed in parentheses may not be completely eluted.

[†] Losses of As, Re, and possibly Sn may occur by volatilization. Recoveries of Sn, Pd, and Re have not been verified.

[†] This part of the procedure needs further work, because an appreciable amount of molybdenum sometimes remains on the column and is eluted with fraction E. A second pass through the column is then required.

** Any major impurity or breakthrough of matrix would greatly reduce detection limits.

4. Analysis of Uranium-Plutonium-Molybdenum Alloys for Trace Impurities
(E. A. Huff and J. P. Faris)

Analysis of uranium-plutonium-molybdenum alloys has previously been carried out by determining the impurities in the presence of molybdenum after separating the uranium and plutonium. The amount of molybdenum present (2-4%) limited the sample aliquot that could be taken for copper spark analysis. The previously described procedure which was developed for the analysis of molybdenum was subsequently applied to the analysis of these alloys to improve the detection limits for trace impurities as outlined below.

Procedure:

- (1) Dissolve a weighed sample, usually 100-300 mg, in 8M nitric acid and pass through a dual anion exchange-partition chromatography column to remove U and Pu, as described previously.^{7,7a} Plutonium is almost selectively retained by an anion exchange resin and uranium is extracted from solution by diethylhexyl-ethylhexyl phosphonate fixed on plastic powder.
- (2) Evaporate the effluent, which contains Mo and impurities, and convert to an HCl system by taking to dryness several times with HCl. Take up the final residue in 6M HCl and pass through a preconditioned anion-exchange column in the chloride form. A 1/2-in. plastic column containing approximately 5 in. of Dowex 1x4, 100-200 mesh is suitable. Wash with 60-80 mL 6M HCl and evaporate effluent to 1 mL for analysis. This fraction contains Na, Li, K, Rb, Cs, Ba, Sr, Ca, Mg, B, Ni, (Co), Cr, Mn, Cu, V, Al, As, Pb, Ti, (In), (Ag?), Am, and rare-earth elements.
- (3) Wash column with 80-100 mL 1M HF. This fraction contains Cd, Zn, Ga, Bi, (Fe) and any remaining Co and In.
- (4) Wash column with ~30 mL 0.15M HCl-0.2M HF to ensure complete elution of Fe and Te. Combine with previous HF fraction from step 3 and evaporate to a small volume for analysis by the copper-spark method.^{1,2}

5. Analysis of Cadmium Telluride Semiconductor for Trace Elements
(J. P. Faris)

Direct analysis of cadmium telluride samples using a direct current (dc) arc gave fairly good detection limits for many of the common elements. To determine the rare earths and to increase the sensitivity for a number of other elements, the anion-exchange procedure outlined below was found to be effective. Determination of many elements could be made in the fractional parts-per-million range.

Procedure:

- (1) Dissolve a weighed 100-500 mg sample in aqua regia, evaporate to dryness and take up in 3M HCl (~15 mL).

- (2) Add the sample solution to a preconditioned anion-exchange column. A plastic 1/2-in. column, containing about 4 in. Dowex 1x4, 100-200 mesh resin, is convenient. Wash with about 75 mL 3M HCl. The rare-earth elements and most common impurities are eluted while the matrix material (cadmium and tellurium) is retained on the resin.
- (3) Elute Te with 50-60 mL 1M HCl-0.1M HF mixture. Discard.
- (4) Elute Mo and Sn with ~100 mL of 2M HCl-8M HF mixture.
- (5) Elute Zn with ~50 mL of 0.1M HCl.
- (6) Evaporate the three impurity solutions to a small volume (1 mL) and determine the elements present by the copper spark method.^{1,2}

A control column should be carried along using the same procedure to determine the "blank" level for the most common impurities.

6. Separation Procedure for Trace-Element Analysis of Uranium-Niobium Alloys (J. P. Faris)

The carrier-distillation method⁸ is routinely used in the spectrochemical analysis of high-purity uranium-niobium alloys. This method is effective for many impurities but may not include some of the more refractory elements (*i.e.*, Ta, W, Zr). A two-step separation procedure designed primarily for the determination of this group is outlined below.* This procedure also allows the determination of a number the elements normally found by the dc arc technique and is useful for verification or cross-checking of results. The niobium content of the samples ranged up to 30%, but analysis of alloys of any uranium-niobium ratio could be made with only slight modification in the size of the ion-exchange column.

Procedure:

- (1) Place a weighed sample, ~200 mg, in a Teflon beaker and dissolve in 8M nitric acid plus a small amount of hydrofluoric acid. Remove uranium by partition chromatography, as described previously.^{7,7a} Diethylhexylethylhexyl phosphonate was used as the extractant because of its good stability in the presence of hydrofluoric acid.
- (2) Evaporate the effluent containing the niobium plus impurities to dryness several times after dissolving in HCl-HF. Finally dissolve in ~15 mL of 0.1M HCl-0.5M HF solution. Pass through a preconditioned anion-exchange column and wash the resin with ~60 mL of 0.1M HCl-0.5M HF. A plastic column, 1/2 in. ID containing about 4 in. Dowex 1x8, 100-200 mesh resin is suitable. The effluent contains the common impurities that are not adsorbed from this acid mixture.

* Alternative procedures, similar to those given for the analysis of niobium for trace impurities, may be applied after step 1.

- (3) Wash the resin column with ~50 mL of 6M HCl-6M HF solution. The effluent contains Zr, Hf, Ti, W, (Mo),* (Re) and any remaining V. Possibly some Pd, Be or Cd may break through.
- (4) Wash the column with ~75 mL of 7M HCl-0.2M HF to remove niobium. Discard.
- (5) Wash column with 60 mL of 12M HNO₃-5M HF to elute Ta, Sn, Be, Cd, Pd, Mo and Re. (Results for the latter four elements lacked good reproducibility; further checking is needed.)
- (6) Evaporate effluents obtained in steps 2, 3, and 5 and take each up in 1 mL of HCl-HF solution. Determine elements present by the copper spark method.^{1,2}

7. Analysis of UO₂-10% CeO₂ Alloys for Tungsten
(J. P. Faris)

A procedure used for the analysis of uranium oxide-10% cerium oxide is outlined below. Matrix elements (uranium and cerium) were removed by precipitation and anion exchange, after which tungsten was isolated for spectrochemical determination at the parts-per-million level. Other impurities which could also be determined using this procedure are Ti, Zr, Hf, (Nb), V, Al, Be, and Ni, as well as other elements that are soluble in HF and are not adsorbed by an anion-exchange resin under the conditions used.

Procedure:

- (1) Weigh sample, place in Teflon beaker, and dissolve in ~7M HNO₃ plus a few drops of HF. A sample weight of 200-400 mg is preferred.
- (2) After dissolution, add to 2-5 mL concentrated HF to precipitate CeF₃ and to dissolve the tungsten. Digest for one-half hour or longer, breaking up the larger particles with a plastic rod.
- (3) Evaporate to less than 10 mL, transfer to a plastic vial and adjust the volume to 10-15 mL with water. Mix, allow precipitate to settle, and decant an aliquot, usually about one-half of the sample solution, into a Teflon beaker. Evaporate to dryness and dissolve in 7.7M HCl-3M HF.
- (4) Prepare an anion-exchange column and precondition the resin with the 7.7M HCl-3M HF mixture. A plastic column, 1/2 in. ID, containing about 3 or 4 in. Dowex 1x8, 100-200 mesh is suitable. Add the sample solution to the column and wash with 7.7M HCl-3M HF collecting about 75 mL of the effluent. Evaporate to a small volume (1 mL) and analyze by the copper spark method.^{1,2}

Note: For some samples an additional 50 mL of wash was collected and analyzed to check for completeness of tungsten elution. Since none was detected in any of these fractions, it was assumed that the tungsten recovery was quantitative in the first fraction.

*Elements that may not be quantitatively eluted are enclosed in parentheses.

This procedure could also be modified to include determination of niobium by first washing the column with about 50 mL of 6M HCl-0.1M HF to elute Nb, then continuing as above.

8. Ion-Exchange Procedures for Determination of Trace Elements in High-Purity Aluminum

Direct spectrochemical analysis of aluminum metal for most trace impurities is readily accomplished using the dc arc. However, analysis for additional impurities may be carried out using ion-exchange procedures to separate the elements of interest for their determination. The procedures described below take advantage of the fact that aluminum is not retained by either anion or cation exchange resins from dilute HCl-HF solutions.

Procedures:

Weigh two sample portions of 100-500 mg each, depending upon detection limits desired. Dissolve in distilled 6M HCl plus a small amount of HF, evaporate to incipient dryness and dissolve in 0.15M HCl-0.2M HF.

a. Anion Exchange

- (1) Pass the solution through a small preconditioned anion-exchange column. A plastic column, 0.7 cm x 4 cm (supplied by Bio Rad Laboratories) containing Dowex 1x4 or Dowex 1x8 is satisfactory.
- (2) Wash thoroughly with 0.15M HCl-0.2M HF and finally with several column volumes of 0.35M HCl-0.8M HF to ensure complete removal of the aluminum matrix.
- (3) Elute most of the retained elements (see Tables A-1 and A-2) with ~25 mL of 4M HNO₃-1M HF.
- (4) Elute tantalum with an additional ~25 mL of 12M HNO₃-5M HF. Collect effluents in separate plastic beakers, evaporate each to dryness and dissolve in 1 mL of HCl-HF solution. Determine elements present by the copper spark method.^{1,2}

b. Cation Exchange

- (1) Pass solution through a similar column containing cation-exchange resin. Dowex 50x8, 100-200 mesh, is satisfactory. Wash thoroughly with 0.15M HCl-0.2M HF and finally with a few mL of water.
- (2) Elute the retained impurities with ~25 mL of 4.5M HCl, evaporate to 1 mL, and determine the elements present by the copper spark method.^{1,2} A number of elements, including the rare earths, may be determined by this procedure.⁹⁻¹¹

Results for a previously analyzed sample to which some impurities were added to test the separation procedures are given in Table 2.

Table 2. Aluminum Metal Sample (Elution with 0.15M HCl-0.2M HF; visual comparison results, $\pm 50\%$)

Impurity	Amount Impurity Present, ppm		Amount Impurity Found, ppm	
	Reported	Added	By Anion X	By Cation X
Ag	<1	100	100	
Co	<5	50		40
Cd	<1	100		Very Low
Fe	2000	--		Very Low
Ga	200	--		300
Hf	<5	100	50	
Mn	40	--		40
Ti	60	--	60	
V	60	--		50
W	<10	100	100	
La	<0.5	50		40
Eu	<0.5	50		40
Sc	<0.5	50		0

9. Analysis of Aluminum-Uranium Alloys

A procedure similar to Procedure 8 has been used for the analysis of aluminum-uranium alloys for trace impurities. Uranium, like aluminum, is not retained by an anion-exchange resin from dilute HCl-HF nor by a cation-exchange resin from dilute HF. Samples are dissolved in HNO₃-HF, evaporated to dryness and taken up in an appropriate solution. The anion-exchange method is identical to that given for the analysis of aluminum metal. For cation-exchange separation, the sample aliquot is placed in $\sim 0.15M$ HF and passed through the column. After the column is washed well with 0.15M HF, the procedure is carried out in the same way as that described above.

10. Analysis of 80% Aluminum-20% Plutonium Alloy

Dissolve a weighed sample (~ 200 -300 mg) in HCl plus some HNO₃, take to incipient dryness and take up in 8M HNO₃. Pass sample solution through a column containing an anion-exchange resin in the nitrate form to remove the plutonium.¹² Wash thoroughly with 8M HNO₃. Divide the effluent into three equal portions and evaporate to dryness.

Portion A. Take up in dilute HCl-HF and pass through a small cation-exchange resin column. Proceed as described above for aluminum analysis.

Portion B. Take up in 0.1M HCl-0.5M HF and pass through a small preconditioned anion-exchange column. Wash thoroughly with 0.1M HCl-0.5M HF. Elute the adsorbed impurities with 4M HNO₃-1M HF and proceed as described for analysis of high-purity aluminum.

Portion C. Take up in 6M HCl and pass through a small preconditioned anion-exchange column. Wash thoroughly with 6M HCl, then elute the adsorbed elements (Fe, Ga, Te, Cd, U, Zn) with dilute HCl. Evaporate to a small volume and determine impurities present by the copper spark method.^{1,2}

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APPENDIX

**DISTRIBUTION COEFFICIENTS FOR ELEMENTS
IN HCl-HF MIXTURES, HCl, AND HF**

Table A-1. Distribution Coefficients on Dowex 1x10 Anion-Exchange Resin (200-400 mesh) as a Function of HCl and HF Concentrations (concentrations in molarity)

ads: adsorbed (): indefinite elution curve,
sl: slight tailing, poor reproducibility
~: not adsorbed ppt: precipitate
*: assigned value

Element	HF	HCl									
		0	0.1	0.2	0.5	1	3	6	9	10	12
Ti(IV)	0		-	-	-	-	-	-	-	~4	55
	0.1	ads	ads	295	42	15	4.1	-*	-	-*	sl?
	0.2	ads	ads	390	49	15	5.4	-	-	-*	-
	0.5	ads	ads	400	58	15	2.3	-*	-*	-*	-*
	1	ads	ads	360	60	15	1.4	-	-	-*	-
	4	320	290	158	44	14	-	-	-	-*	-
	8	72	65	45	25	10	-	-	-	-	-
Zr(IV)	0		-	-	-	-	-	-	sl-30	ads	ads
	0.1	ads	ads	310	39	7	-	-*	-	-*	-
	0.2	ads	ads	400	47	12	-	-	-	-*	-
	0.5	ads	ads	410	56	14.5	-	-*	-*	-*	-*
	1	ads	ads	340	58	15	-	-	-	-*	-
	4	320	280	140	43	14	-	-	-	-*	-
	8	60	58	45	24	7.5	-	-	-	-	-
Hf(IV)	0		-	-	-	-	-	-	~4	(75)	ads
	0.1	ads	ads	340	45	9	-	-*	-	-*	-
	0.2	ads	ads	400	52	13	-	-	-	-*	-
	0.5	ads	ads	410	57	14.5	-	-*	-*	-*	-*
	1	ads	ads	340	58	15	-	-	-	-*	-
	4	325	280	140	43	14	-	-	-	-*	-
	8	60	58	45	24	7.5	-	-	-	-	-
Nb(V)	0										
	0.1	ads	~475	150	61	35	13.5	4.8	14	35	121
	0.2	ads	~500	155	59	30	13.5	6	7	12*	42
	0.5	ads	~625	180	59	28	14	11	18	23*	34
	1	ads	~720	190	59	28	14	20	40	50	71
	4	~900	475	190	59	35	37	97	125	95	
	8	180	135	108	75	55	90	123	85		
Ta(V)	0										
	0.1	ads	ads	ads	ads	ads	ads	ads	ads	230	125
	0.2	ads	ads	ads	ads	ads	ads	ads	ads	390	122
	0.5	ads	ads	ads	ads	ads	ads	ads	ads	325	115
	1	ads	ads	ads	ads	ads	ads	ads	ads	275	102
	4	ads	ads	ads	ads	ads	ads	ads	ads	143	90
	8	410	~490	~440	~360	~295	175	104	65		
W(VI)	0										
	0.1	ads	~475	115	36	17.5	11	10	36	54	50
	0.2	ads	~750	158	45	22	13	8	26	36*	42
	0.5	ads	~1150	208	52	24	12.5	4.5	12	20*	29
	1	ads	~1400	230	53	25	11	sl	6	12	21
	4	ads	~560	185	52	22	5.2	-	sl		
	8	110	95	75	35	14	sl?	-	5		
Sn(IV)	0		ppt	(ppt)?	315	ads	ads	ads	ads	ads	
	0.1	ads	ads	340	58	~0	ads	ads	ads	ads*	ads
	0.2	ads	ads	380	62.5	~8	ads	ads	ads	ads*	ads
	0.5	ads	ads	390	66	26	165	ads	ads	ads*	ads*
	1	ads	ads	360	64	22	58	ads	ads	ads*	ads
	4	~340	~300	155	41	15	15	~280	ads	ads*	
	8	64	57	44	24	10.5	12	~205	~1000		
Mo(VI)	0		(~115)	(~38)	(~16)	(~9)	98	210	153	115	150-250
	0.1	ads	~160	68	31	24	35	170	130	~140	(~280)
	0.2	ads	~230	99	36	25	32	140	130	~130	(~275)
	0.5	ads	~300	112	42	25.5	23.5	72	120	~110	(~265)
	1	ads	~320	110	44	25	15.5	40	85	(~90)	(~250)
	4	195	173	86	34	17.5	7	14	(~88)		
	8	70	56	45	24	14	5.5	10	~85?		

(contd)

Table A-1. (contd)

Element	HF	HCl									
		0	0.1	0.2	0.5	1	3	6	9	10	12
Re(VII)	0			ads	ads	ads	380	81	22.5	16.5	26
	0.1	ads	ads	ads	ads	ads	ads	75	21	16	23
	0.2	ads	ads	ads	ads	ads	ads	75	21	16*	25
	0.5	ads	ads	ads	ads	ads	280	62	16.5	16*	24
	1	ads	ads	ads	ads	ads	180	47	15	16	27
	4	ads	~770	550*	340	215	72	21	14	16	
	8	175	185	176	128	85	32	14	(15-80)		
Hg(II)	0		ads	ads	ads	ads	ads	ads	~200	~120	58
	0.1	sl	ads	ads	ads	ads*	ads*	ads*	165	~110	58
	0.2	sl	ads	ads	ads	ads*	ads*	ads*	164	~108*	58
	0.5	sl?	ads	ads	ads	ads	ads*	ads*	140	~92*	48
	1	-	ads	ads	ads	ads*	ads*	ads*	116	76	45
	4	-	ads	ads	ads*	ads	ads	~255	69	45	
	8	-	ads	ads	ads*	ads	~410	~100	37		
Pd(II)	0		ads	ads	ads	ads	~500	120	55	38	28
	0.1		ads	ads	ads	~2300	570	119	45	~35	26
	0.2		ads	ads	ads	~2200	500	112	45	35*	26
	0.5	ads	ads	ads	ads	~1900	450	102	43	33*	25
	1	ads	ads	ads*	ads	~1500	385	91	40	30	25
	4	ads	ads	ads*	ads	~850	190	54	27	22	
	8	ads	ads	ads	~1400	490	95	33	17		
Bi(III)	0		ppt	ads	ads	ads	ads	202	74	57	43
	0.1	33	ads	ads	ads	ads	ads	200	66	50	43
	0.2	14	ads	ads	ads	ads	ads	198	65	50*	43
	0.5	-	~580	ads	ads	ads	ads	178	64	50*	41
	1	-	106	~2500	ads	ads	~1400	156	59	50	38
	4	-	16	225	ads	ads	~500	85	40	34	
	8	-	10.5	145	ads	ads	~220	48	28		
Pb(II)	0		-	11	19	24	15	~3	-	-	-
	0.1	-	-	(10)	18	22	14	-	~*	~*	-
	0.2	-	-	(10)	17.5	22	14	-	~*	~*	-
	0.5	-	(4)	(9)	16	21	14	-	~*	~*	~*
	1	-	(4.5)	(8.5)	15	20	14	-	-	~*	-
	4	-	(5)	(8)	12	15	6.5	-	-	-	
	8	-	(4)	(5)	10	14	5.5	-	-	-	
In(III)	0		-	sl?	sl	10	20	18	13	11	7.5
	0.1	50	-	-	sl	8	18	16.5	12	11	7
	0.2	23	-	-	sl	6	17	16	12	11*	7
	0.5	sl	-	-	sl?	6	16	15	12	10*	6.5
	1	-	-	-	-	4.4	14.5	13	10	8.5	6
	4	-	-	-	-	sl	13.5	11	6.5	5	
	8	-	-	-	-	sl	10	7.5	5.5		
V	0		-	-	-	-	-	-	-	~*	-
	0.1	(>400)	(6)	-	-	-	-	-	-	~*	-
	0.2	(>200)	(14)	-	-	-	-	-	-	~*	-
	0.5	(10-250)	(35)	(10)	-	-	-	-	-	~*	~*
	1	(10-180)	(60)	(22)	(8)	-	-	-	-	~*	-
	4	(55)	(42)	(21)	(11)	10	-	-	-	~*	-
	8	(23)	(18)	14	10	-	-	-	-	-	-
U(VI)	0		-	-	-	~1.2	35	ads	ads	ads	ads
	0.1	ads	-	-	-	-	18	ads	ads	ads*	ads*
	0.2	ads*	sl	-	-	-	16	ads	ads	ads*	ads*
	0.5	ads*	8	-	-	-	6	~260	ads	ads*	ads*
	1	~690	15.5	9	-	-	sl	120	~480	ads*	ads*
	4	72	21	14.5	-	-	-	33	150	~480	
	8	23	15	11	sl	-	-	26	210		
Te(IV)	0		-	-	-	-	730	ads	ads	ads	ads
	0.1	155	-	-	-	-	ads	ads	ads	ads*	ads
	0.2	170	-	-	-	-	ads	ads	ads	ads*	ads
	0.5	172	12	sl?	-	-	~270	ads	ads	ads*	ads*
	1	158	31	14.5	sl	8.5	120	ads	ads	ads*	ads
	4	68	36	47	25	14	30	ads	ads	ads	
	8	36	35	34	23	14	32	ads	ads		

(contd)

Table A-1. (contd)

		HCl									
Element	HF	0	0.1	0.2	0.5	1	3	6	9	10	12
Fe(III)	0		-	-	-	~1.5	(45)	ads	ads	ads	ads
	0.1	200	-	-	-	-	(~50)	ads	ads*	ads*	ads*
	0.2	80	-	-	-	-	(~35)	ads	ads	ads*	ads*
	0.5	24	-	-	-	-	12	ads*	ads*	ads*	ads*
	1	10	-	-*	-	-	4	ads*	ads	ads*	ads*
	4	-	-	-*	-	-	-	ads	ads	ads*	ads*
	8	-	-	-*	-	-	-	ads	ads	ads	ads
Co(II)	0		-	-	-	-	-	9	71	64	45
	0.1	-	-	-	-	-	-	8	63	59	45
	0.2	-	-	-	-	-	-	8	63	59*	44
	0.5	-	-	-	-	-	-	9	61	56*	42
	1	-	-	-	-	-	-	12	60	53	37
	4	-	-	-	-	-	-	17	46	36	36
	8	-	-	-	-	-	-	41	33		
Ga(III)	0		-	-	-	~2	(sl-90)	ads	ads	ads	ads
	0.1	45	-	-	-	-	(sl-60)	ads	ads	ads*	ads
	0.2	20	-	-	-	-	(sl-50)	ads	ads	ads*	ads
	0.5	-	-	-	-	-	(sl-35)	ads	ads	ads*	ads*
	1	-	-	-	-	-	(sl-15)	ads	ads	ads*	ads*
	4	-	-	-	-	-	sl	ads	ads	ads	ads
	8	-	-	-	-	-	sl	ads	ads		
Cd(II)	0	-	400	ads	ads	ads	ads	~370	68	36	15
	0.1	-	~380	ads	ads	ads	ads	~315	60	34	15
	0.2	-	~355	~560	ads	ads	ads	310	59	33*	15
	0.5	-	~300	~550	ads	ads	ads	287	54	29*	15
	1	-	~225	~325	ads	ads	~950	220	48	25	15
	4	-	~50	180	ads	ads	ads	92	26	15	
	8	-	25	105	ads	ads	~210	44	15		
Zn(II)	0		9	25	92	ads	ads	245	90	68	45
	0.1	-	~5	21	92	ads	ads	240	84	65	45
	0.2	-	~5	21	92	ads	ads	240	82	63*	43
	0.5	-	~5	17	92	ads	ads	210	80	59*	39
	1	-	~5	16	92	ads	ads	180	75	52	36
	4	-	-	15	88	ads	~300	100	51	35	
	8	-	-	16	158	290	165	57	30		
As(V)	0		-	-	-	-	-	-	-	-	-
	0.1	160	-	-	-	-	-	-	-	-	sl?
	0.2	59	-	-	-	-	-	-	-	-*	-
	0.5	(40-140)	sl	-	-	-	-	sl	-	-	7
	1	(170-260)	~30	6	sl	sl?	(5-15)	15	14	14	14
	4	(230)	(190-240)	~170	~100	65	28	16.5	12	11	
	8	95	92	88	65	46	22	14	10		
B(III)	0		-	-	-	-	-	-	-	-	-
	0.1	ads	ads	~570	280	143	45	19	10	9	5
	0.2	ads	ads	~540	250	130	43	17	7.5	7*	~4
	0.5	ads	ads	~510	220	112	38	15	5.5	5*	3*
	1	ads	~800	410	188	94	32	14.5	5	~5	sl
	4	200	~190	142	75	48	19	10	7	~6	
	8	56	65	55	38	26	12	7.5	6.5		
Al(III)	0.1-8		-	-	-	-	-	-	-	-	-
Be(II)	0.1-8		sl	-	-	-	-	-	-	-	-
Cr(III)	0.1-8		-	-	-	-	-	-	-	-	-
P(V)	0.1-8		-	-	-	-	-	-	-	-	-
Au(III)	0.1-8		ads	ads	ads	ads	ads	ads	ads	ads	ads
Pt(IV)	0.1-8		ads	ads	ads	ads	ads	ads	ads	ads	ads
Sb(V)	0.1-8		ads	ads	ads	ads	ads	ads	ads	ads	ads
Tl(III)	0.1-8		ads	ads	ads	ads	ads	ads	ads	ads	ads

Table A-2. Distribution Coefficients on Dowex 1x4 Anion-Exchange Resin (100-200 mesh) as a Function of HCl and HF Concentrations (concentrations in molarity)

ads: adsorbed (): indefinite elution curve,
sl: slight tailing, poor reproducibility
-: not adsorbed ppt: precipitate
*: assigned value

Element	HF	HCl									
		0	0.1	0.2	0.5	1	3	6	9	10	12
Ti(IV)	0		-*	-*	-*	-*	-*	-	-	-	31
	0.1	ads	ads*	~260*	47*	17.5	~4*	-	-	-*	sl
	0.2	ads*	ads*	325	55	17.5	sl	-	-	-*	sl
	0.5	ads*	ads*	350*	63*	17.5	-*	-*	-*	-*	-*
	1	ads	ads*	330	64	19	-	-*	-	-*	-
	4	370	210	128	45	17	-	-	-	-*	-
	8	67	55	45	25	13	-	-*	-*	-	-
Zr(IV)	0		-*	-*	-*	-*	-*	-	(10-30)	~210	ads
	0.1	ads	ads*	~265*	44*	12	-	-	sl?	-	-
	0.2	ads*	ads*	325	52	14.5	-	-	-	-*	-
	0.5	ads*	ads*	360*	62*	16.5	-*	-*	-*	-*	-*
	1	ads	ads*	315	64	18	-	-*	-	-*	-
	4	380	200	118	41	15	-	-	-	-*	-
	8	60	50	44	24	11.5	-	-*	-*	-	-
Hf(IV)	0		-*	-*	-*	-*	-*	-	sl	~35	ads
	0.1	ads	ads*	~280*	47*	13.5	-	-	-	-*	-
	0.2	ads*	ads*	330	54	15.5	-	-	-	-*	-
	0.5	ads*	ads*	360*	62*	17	-*	-*	-*	-*	-*
	1	ads	ads*	315	64	18	-	-*	-	-*	-
	4	380	200	118	41	15	-	-	-	-*	-
	8	60	50	44	24	11.5	-	-*	-*	-	-
Nb(V)	0										
	0.1	ads	ads*	150	57*	30	12*	7.5	11	22*	59
	0.2	ads*	ads*	135	55	27	12	5.5	5.5	13*	50
	0.5	ads*	ads*	162	53*	27	12*	7.7*	9*	14*	35*
	1	ads	ads*	172	53	25	12	10.5	17	19.5*	26
	4	~820	~315*	140	51	25.5	18	44	48	-	-
	8	125	80	72	47	30.5	40	60	35	-	-
Ta(V)	0										
	0.1	ads	ads*	ads*	ads*	ads*	ads*	ads*	78*		41
	0.2	ads*	ads*	ads*	ads*	ads*	ads*		75		39
	0.5	ads*	ads*	ads*	ads*	ads*	ads*		70*		38*
	1	ads	ads*	ads*	ads*	ads*	ads*		65		36
	4	ads	ads*	~312*	267	222*	145	86	45		-
	8	200	170	153	132*	112	77	50*	31		-
W(VI)	0										
	0.1	ads	ads*	97	40*	20	11*	11.3	30	33*	35
	0.2	ads*	ads*	127	45	24	13	10	22	29*	35
	0.5	ads*	ads*	185*	48*	25*	13*	7.2*	13*	18*	24*
	1	ads	ads*	208	49	24	12	4.5	8	11*	16
	4	ads	ads*	144	50	21	6	-	-	-	-
	8	88	68	60	34	17	-	-	-	-	-
Sn(IV)	0		ppt	(sl-50)	140	ads*	ads*	ads*	ads*	ads*	ads*
	0.1	ads	ads*	ads*	ads*	54	ads*	ads*	ads*	ads*	ads*
	0.2	ads*	ads*	300	60	34	265	ads*	ads*	ads*	ads*
	0.5	ads*	ads*	355*	59*	25	91*	ads*	ads*	ads*	ads*
	1	ads	ads*	350	55	24	43	ads*	ads*	ads*	ads*
	4	~440	220	134	43	16	14	157	ads	-	-
	8	64	55	46	24	14	11.5	135	-	-	-
Mo(VI)	0		(20-100)	(sl-30)	(10-35)	(5-70)	53	125	86	140	(70-150)
	0.1	ads	138	53	30*	24	29*	95	76		(80-ads)
	0.2	ads*	178	75	34	24	26	83	75		(~150)
	0.5	ads*	~240	100	38*	24	20.5*	47*	71*		-
	1	ads	250	106	39	24	16	29	64		(70-150)
	4	225	112	71	33	18	10	16	39		-
	8	57	47	42	24	15	6	12	(32-100)		-

(contd)

Table A-2. (contd)

Element	HF	HCl									
		0	0.1	0.2	0.5	1	3	6	9	10	12
Re(VII)	0			ads*	ads*	ads*	107	29	12	14	21
	0.1	ads	ads*	ads*	ads*	ads*		26	13		21
	0.2	ads	ads*	ads*	ads*	ads*	107	26	13		24
	0.5	ads*	ads*	ads*	ads*	ads*	95*	23*	13*		23*
	1	ads	ads*	ads*	ads*	ads*	77	20	13		23
	4	ads	340	292	170	100	35	14	13		
	8	112	105	~105	82*	54	22	10			
Hg(II)	0			ads*	ads*	ads*	ads*	~295	75	54	30
	0.1	s1+	ads	ads*	ads*	ads*	ads*		77	55*	30
	0.2	s1+	ads	ads*	ads*	ads*	ads*	~300	76	54*	30
	0.5	-	ads*	ads*	ads*	ads*	ads*	~280*	74*	54*	30*
	1	-	ads*	ads*	ads*	ads*	ads*	~260*	70	53*	30
	4	-	ads	ads	ads*	ads*	ads*	120	41		
	8	-		ads	ads*	ads*	250	~67*	31		
Pd(II)	0		ads*	ads*	ads*	ads*	225	71	35	30	24
	0.1		ads	ads*	ads*	ads*		70	35	30*	24
	0.2		ads	ads*	ads*	ads*	225	68	35	30*	24
	0.5	ads*	ads*	ads*	ads*	ads*	212*	65*	33*	29*	23*
	1	ads*	ads*	ads*	ads*	ads*	190	62	31	28*	21
	4	ads*	ads*	ads*	ads*	ads*	106	43	24		
	8	ads*	ads*	ads*	ads*	ads*	71	30	18		
Bi(III)	0		ppt	ads*	ads*	ads*	550	125	55	46	39
	0.1	42	adv*	ads	ads*	ads*	ads*	110	54	45*	38
	0.2	24	ads*	ads*	ads*	ads*	ads*	110	54	45*	38
	0.5	12	~220	ads*	ads*	ads*	ads*	108*	51*	44*	37*
	1	-	62	ads*	ads*	ads*	ads*	105	49	42*	35
	4	-	12	98	ads	ads*	~270	68	36		
	8	-	5.4	71		ads*	148	45	36		
Pb(II)	0		-	5.5	14	17.5	13	-	-	-*	-
	0.1	-	-	(5)?	14*	16	12*	-	-	-*	-*
	0.2	-	-	5.5	14	16	11.5	-	-	-*	-*
	0.5	-	s1?	7	14*	16	11*	-*	-*	-*	-*
	1	-	s1?	4	14	15	10.5	-*	-	-*	-*
	4	-*	s1?	s1?	14	15	6.5	-	-	-*	-*
	8	-*	s1?	(7)?	13	14	s1?	-*	-	-*	-*
In(III)	0		-	-	6	11.5	18	16	12	10.5	8
	0.1	60	-	-	5.5*	11.5	17.5*	16	12	10.5*	8
	0.2	31	-	-*	5	11.5	17	15.5	12	10.5*	8
	0.5	13	-	-	-	10.5	16.5*	15*	11.5*	10*	8*
	1	-	-	-	-	7.5	15.5	14	11		8
	4	-	-	-	-	6.5	14	12	7.5		
	8	-	-	-	-	7.5	12	7.5	5.5		
V	0		-	-	-	-	-	-	-	-*	-
	0.1	(>220)	7	-	-*	-	-*	-	-	-*	-
	0.2	(>250)	24	s1	-	-	-	-	-	-*	-
	0.5	(45-230)	(38)	15	-	-	-*	-*	-*	-*	-*
	1	(40-300)	(60)	(30)	12	-	-	-*	-	-*	-
	4	(62)	(40)	(34)	15	13	-	-	-	-*	-
	8	(24)	(22)	16	13.5	9	-	-*	-	-*	-
U(VI)	0		-	-	-	-	13	142	ads	ads	ads
	0.1	ads	-	-	-*	-	-	~125	ads*	ads*	ads*
	0.2	ads*	-	-*	-	-	7	~121	ads*	ads*	ads*
	0.5	ads*	13.5	-	-*	-	-	85*	ads*	ads*	ads*
	1	~57*	25	-	-	-	-	52	~225	ads*	ads*
	4	61	21	13	-	-	-	18	129		
	8	20	14	11	-	-	-	16	90		
Te(IV)	0		-	-	-	-	125	ads	ads	ads*	ads
	0.1	95	-	-	-*	-	-	ads	ads*	ads*	ads*
	0.2	107	-	-	-	-	93	ads	ads*	ads*	ads*
	0.5	115	10	-	-*	-	58*	ads	ads*	ads*	ads*
	1	108	25	14	-	-	35	ads*	ads*	ads*	ads*
	4	56	43	32	18	12	16	ads*	ads	ads*	
	8	29	27	26	16	14	14		ads*		

(contd)

Table A-2. (contd)

Element	HF	HCl									
		0	0.1	0.2	0.5	1	3	6	9	10	12
Fe(III)	0		-*	-*	-*	-*	8	ads	ads	ads*	ads*
	0.1	265	-	-*	-*	-		ads	ads*	ads*	ads*
	0.2	123	-	-*	-*	-*	5	~295	ads*	ads*	ads*
	0.5	44	-*	-	-*	-*		~290*	ads*	ads*	ads*
	1	20	-*	-	-*	-*		~280*	ads*	ads*	ads*
	4	-	-	-*	-*	-*		~230	ads		
	8	-	-	-*	-*	-*					
Co(II)	0		-	-	-	-		4.5	40	41	30
	0.1	-	-*	-	-*	-*		4.5	40	41*	30
	0.2	-	-*	-*	-	-		4.5	40	41*	30
	0.5	-	-*	-	-*	-*		4.5*	38*	40*	29*
	1	-*	-*	-	-*	-		4.5	36		28
	4	-*	-	-	-	-		15.5	31		
	8	-*	-	-	-*	-		28	26		
Ca(III)	0		-	-	-	-	(9-30)	ads	ads	ads*	ads
	0.1	68	-	-	-*	-		ads*	ads*	ads*	ads*
	0.2	37	-	-*	-*	-	(5-10)	ads	ads*	ads*	ads*
	0.5	14.5	-	-	-*	-	s1*	ads*	ads*	ads*	ads*
	1	5.5	-	-	-*	-		ads*	ads*	ads*	ads*
	4	-	-	-	-	-		ads	ads*		
	8	-	-	-	-*	-		ads*	ads*		
Cd(II)	0	-	143	260	470	ads	465	122	31	21	12
	0.1	-	120	~190	ads*	ads*		117	31	21*	12
	0.2	-	108	185	ads	ads*	450	112	31	21*	12
	0.5	-	94	160	ads*	ads*	425*	108*	30*	20*	12*
	1	-	71	130	ads*	ads*	~400	105	28		12
	4	-	21	72	230	~320	195	58	19		
	8	-	14	60	~210	280	105	34	13		
Zn(II)	0		4.2	11	47	175*	320	118	51	40	29
	0.1	-	-	9	46*	172*	310	113	50	39*	29
	0.2	-	s1?	9	46	170	308	108	50	39*	29
	0.5	-	-	9	45*	162*	303*	106*	49*	38*	29*
	1	-	-	9	40	151	288	105	47		27.5
	4	-	-	6	57	135	151	65	34		
	8	-	-	13	98	205	99	42	25		
As(V)	0		-	-	-	-	-*	-	-	-*	s1?
	0.1	200	-	-	-*	-	-*	-	-	-*	-
	0.2	80	-	-	-	-	-	-	-	-*	-
	0.5	55	4.5	-	-*	-	-	-	-	-	-
	1	(150-240)	19	~8	-	-	(s1-20)	11	12		10.5
	4	(~185)	(~110)	(80-140)	(14,65)*	(14,53)*	21	16	12		
	8	80	60	~64	53	38	18	13	10		
B(III)	0		-	-	-	-	-	-*	-	-*	-
	0.1	ads	ads*	ads*	-	70	21*	9	8		7.5
	0.2	ads	ads*	~235	~125	70	21	9	7		7.5
	0.5	ads	ads*	~220*	115*	67*	19.5*	9*	7*		7*
	1	ads	ads*	200	~100	60	18	9	7		4.2
	4	162	110	85	46	34	14	9	~7		
	8	46	45	45	34	22	12.5	8.5	6		
Be(II)	0		-	-	-	-	-	-	-	-*	-
	0.1	ads	s1?	s1?	-	-	-*	-	-*	-*	-*
	0.2	ads	s1	s1*	s1?	-	-	-	-*	-*	-*
	0.5	420	7	~4	-	-	-*	-*	-*	-*	-*
	1	150	10	~4	-	-*	-	-*	-*	-*	-*
	4	20	11	6	s1?	-	-	-	-	-*	-*
	8	7	s1	s1	-	-*	-	-*	-*	-*	-*

*Two numbers represent two distinct peaks.

Table A-3. Distribution Coefficients on Dowex 1x10 Anion-Exchange Resin (200-400 mesh, chloride form) as a Function of HCl Concentration (concentrations in molarity)

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ads: adsorbed          ( ): indefinite elution curve,
sl: slight             tailing, poor reproducibility
-: not adsorbed        ppt: precipitate
*: assigned value

```

[illegible]

Table A-4. Distribution Coefficients on Dowex 1x4 Anion-Exchange Resin (100-200 mesh, chloride form) as a Function of HCl Concentration (concentration in molarity)

ads: adsorbed (): indefinite elution curve,
sl: slight tailing, poor reproducibility
-: not adsorbed ppt: precipitate
*: assigned value

Element	HCl								
	0.1	0.2	0.5	1	3	6	9	10	12
Tl(IV)	-	-	*	*	*	-	-	-	31
Zr(IV)	-	-	*	*	*	-	(10-30)	~210	ads
Hf(IV)	-	-	*	*	*	-	sl	~35	ads
Sn(IV)	ppt	(sl-50)	140	ads*	ads*	ads*	ads*	ads*	ads*
Mo(VI)	(20-100)	(sl-30)	(10-35)	(5-70)	53	125	86	140	(70-150)
Re(VII)	-	ads*	ads*	ads*	107	29	12	14	21
Hg(II)	-	-	ads*	ads*	ads*	~295	75	54	30
Pd(II)	ads*	ads*	ads*	ads*	225	71	35	30	24
Bi(III)	ppt	ads*	ads*	ads*	550	125	55	36	49
Pb(II)	-	5.5	14	17.5	13	-	-	*	-
In(III)	-	-	6	11.5	18	16	12	10.5	8
V	-	-	-	-	-	-	-	*	-
U(VI)	-	-	-	-	13	142	ads	ads	ads
Te(IV)	-	-	-	-	125	ads	ads	ads*	ads
Te(VI)	-	-	-	-	-	-	-	-	-
Fe(III)	*	*	*	*	8	ads	ads	ads*	ads
Co(II)	-	-	-	-	-	4.5	40	41	30
Ga(III)	-	-	-	-	(9-30)	ads	ads	ads*	ads
Cd(II)	143	260	470	ads	465	122	31	21	12
Zn(II)	4.2	11	47	175*	320	118	51	40	29
As(V)	-	-	-	-	-	-	-	-	sl?
B(III)	-	-	-	-	-	*	-	*	-
Al(III)	-	-	-	-	-	-	-	-	-
Be(II)	-	-	-	-	-	-	-	-	-
Sc(III)	-	-	-	*	*	*	*	*	-
P(V)	-	-	-	*	*	*	*	*	-
Cr(III)	-	-	*	*	*	*	*	*	*
Mn(II)	-	-	-	-	-	sl?	~3	*	~4
Mg(II)	-	-	*	*	*	*	*	*	*
Sr(II)	-	-	*	*	*	*	*	*	*
Ba(II)	-	-	*	*	*	*	*	*	*
Ni(II)	-	-	*	*	*	*	*	-	*

Table A-5. Distribution Coefficients on Dowex 1x10 Anion-Exchange Resin (100-200 mesh, fluoride form) as a Function of HF Concentration (concentrations in molarity)

ads: adsorbed (): indefinite elution curve,
sl: slight tailing, poor reproducibility
-: not adsorbed ppt: precipitate
*: assigned value

Element	HF							
	0.1	0.2	0.5	1	2	4	8	12
Ti(IV)	ads	ads	ads	ads		320	72	28
Zr(IV)	ads	ads	ads	ads		320	60	23
Hf(IV)	ads	ads	ads	ads		325	60	23
Nb(V)	ads	ads	ads	ads		~900	180	~150
Ta(V)	ads	ads	ads	ads		ads	410	~235
W(VI)	ads	ads	ads	ads		ads	110	~50
Sn(IV)	ads	ads	ads	ads		~340	64	~27
Mo(VI)	ads	ads	ads	ads		195	70	~37
Re(VII)	ads	ads	ads	ads		ads	175	~90
Hg(II)	sl	sl	sl?	-	-	-	-	-
Pd(II)	ads	ads	ads	ads		ads	ads	ads
Bi(III)	33	14	-	-	-	-	-	-
Pb(II)	-	-	-	-	-	-	-	-
In(III)	50	23	sl	-	-	-	-	-
V	>400	>200	(10-250)	(10-180)	(sl-180)	(55)	(23)	
U(VI)	ads	ads*	ads*	~690		72	23	11
Te(IV)	155	170	172	158	118	68	36	24
Te(VI)	44	24	9.5	-	-	-	-	-
Fe(III)	200	80	24	10	-	-	-	-
Co(II)	-	-	-	-	-	-	-	-
Ga(III)	45	20	-	-	-	-	-	-
Cd(II)	-	-	-	-	-	-	-	-
Zn(II)	-	-	-	-	-	-	-	-
As(V)	160	59	(40-140)	(170-260)	(255)	(230)	95	~75
As(III)	-	-	-	-	-	-	-	-
B(III)	ads	ads	ads	ads	~800	200	56	~28
Al(III)	ads	ads*	112	41	15	5.5	-	-
Be(II)	ads	~1000	260	95	34	12.5	4.4	2.5
Sb(V)			ads	ads	ads	ads	ads	ads
Sb(III)	ads	340	112	49	21	8	-	-
Sc(III)	ads	ads*	ads*	315	105	36	12	6
P(V)	~210	75	27.5	13.5	6.5	-	-	-
Cr(III)	(some)	(some)	(8-30)	(sl-6)	sl	sl	-	-
Mn(II)	(some)	(sl+)	sl	sl?	sl?	sl	-	-
Tl(I)	-	sl?	sl?	sl?	-	-	-	-
Tl(III)	(40)?	(50)?	sl?	sl?	sl	(sl-50)	(sl-50)	-
Mg(II)	-	-	-	-	-	-	-	-
Cu(II)	-	-	-	-	-	-	-	-
Ag(I)	sl+?	sl?	sl?	-	-*	-	-	-
Sr(II)	-	-	-	-	-	-	-	-
Ba(II)	-	-	-	-	-	-	-	-
Ni(II)	-	-	-	-	-	-	-	-

Table A-6. Distribution Coefficients on Dowex 1x4 Anion-Exchange Resin (100-200 mesh, fluoride form) as a Function of HF Concentration (concentrations in molarity)

ads: adsorbed (): indefinite elution curve,
sl: slight tailing, poor reproducibility
-: not adsorbed ppt: precipitate
*: assigned value

Element	HF							
	0.1	0.2	0.5	1	2	4	8	12
Ti(IV)	ads	ads*	ads*	ads		370	67	21*
Zr(IV)	ads	ads*	ads*	ads		380	60	18*
Hf(IV)	ads	ads*	ads*	ads		380	60	18*
Nb(V)	ads	ads*	ads*	ads		~820	125	
Ta(V)	ads	ads*	ads*	ads		ads	200	
W(VI)	ads	ads*	ads*	ads		ads	88	
Sn(IV)	ads	ads*	ads*	ads		~440	64	
Mo(VI)	ads	ads*	ads*	ads		225	57	26*
Re(VII)	ads	ads	ads*	ads		ads	112	
Hg(II)	sl+	sl+	-	-	-	-	-	-*
Pd(II)			ads*	ads*		ads*	ads*	
Bi(III)	42	24	12	-	-	-	-	-*
Pb(II)	-	-	-	-	-	-*	-*	-*
In(III)	60	31	13	-	-	-	-	-*
V	>220	>250	(45-230)	(40-300)	(sl-200)	(62)	(24)	
U(VI)	ads	ads*	ads*	~575	190	61	20	10*
Te(IV)	95	107	115	108	89	56	29	19*
Te(VI)	38	~23	~11	~5.5	-	-	-	-*
Fe(III)	265	123	44	20	9	-	-	-*
Co(II)	-	-	-	-*	-*	-*	-*	-*
Ga(III)	68	37	14.5	5.5	-	-	-	-*
Cd(II)	-	-	-	-	-*	-	-	-*
Zn(II)	-	-	-	-	-*	-	-	-*
As(V)	200	80	55	(150-240)	(~210)	(~185)	80	
As(III)	-	-	-	-	-	-	-	-
B(III)	ads	ads	ads	ads	~580	162	46	-*
Al(III)	ads	ads	195	77	31	12	-	-*
Be(II)	ads	ads	420	150	55	20	7	4*
Sb(V)								
Sb(III)	ads	420	145	66	29.5	13	~5	
Sc(III)	ads	ads	ads*	425	142	47	16	8*
P(V)	235	108	42	20	10	4.5	-	-*
Cr(III)	(some)	(some)	(some)	sl+	sl+	sl	-	-*
Mn(II)	sl	sl	sl	sl	sl	sl	sl	-*
Tl(I)	-	-	-*	-	-*	-*	-*	-*
Tl(III)	(~35)?	(~35)?	sl?		sl+	(sl-35)	(sl-55)	
Hg(II)	-	-	-*	-*	-*	-*	-*	-*
Cu(II)	-	-	-*	-*	-*	-*	-*	-*
Ag(I)	sl+	sl?	sl?					
Sr(II)	-	-	-*	-*	-*	-*	-*	-*
Ba(II)	-	-	-*	-*	-*	-*	-*	-*
Mi(II)	-	-	-*	-*	-*	-*	-*	-*

Table A-7. Distribution Coefficients for Several Elements in Dilute Hydrofluoric Acid (Dowex anion-exchange resin, 200-400 mesh and 100-200 mesh, fluoride form; HF concentrations in molarity)

Element	Distribution Coefficient at Indicated HF Concentration							
	0.1	0.15	0.2	0.3	0.4	0.5	0.6	1.0
<u>Dowex 1×10, 200-400 Mesh</u>								
Bi(III)	33	17	14	10.8	-	-		-
Ga(III)	45	26	20	13	~6	-		-
In(III)	50	32	23	14	~9	sl		-
Fe(III)	200	112	80	46	32	24		10
P(V)	~210	103	75	48	34	27.5		13.5
<u>Dowex 1×4, 100-200 Mesh</u>								
Bi(III)	42	31	24	17	14	12	10.7	-
Ga(III)	68	49	37	25	18	14.5	12.5	5.5
In(III)	60	42	31	21	16	13	10.5	-
Fe(III)	265	185	123	80	57	44	35	20
P(V)	235	155	108	68	51	42	34	20

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