

Production and Chemistry of Transactinide Elements

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Recent progress in production and chemistry of the transactinide elements, element 107 bohrium (Bh) and element 104 rutherfordium (Rf), is reviewed. First information on chemical properties of Bh was obtained in gas chromatographic experiments on an atom-at-a-time basis. Chemical separation and characterization of 6 atoms of ^{267}Bh produced in the bombardment of a ^{249}Bk target with ^{22}Ne beams are outlined. Aqueous chemistry of Rf being performed at JAERI (Japan Atomic Energy Research Institute) is briefly summarized. On-line anion-exchange experiments in acidic solutions on ^{261}Rf produced in the $^{248}\text{Cm}(^{18}\text{O},5n)$ reaction were conducted with a rapid ion-exchange separation apparatus. Characteristic anion-exchange behavior of Rf is discussed.

1. Introduction

According to the actinide concept,¹ the $5f$ electron series ends with element 103, lawrencium (Lr), and a new $6d$ electron transition series is predicted to begin with element 104, rutherfordium (Rf). The elements with atomic numbers $Z \geq 104$ are called transactinide elements or recently called superheavy elements.² The currently known transactinide elements, elements 104 through 112, are placed in the periodic table under their lighter homologues in the $5d$ electron series, Hf to Hg. Elements from 113 to 118 except for 117 synthesized by Oganessian et al.^{3,4} would be in the successive $7p$ electron series (see Figure 1).

Studies of chemical properties of the transactinide elements offer unique opportunities to obtain information about trends in the periodic table of the elements at the limits of nuclear stability and to assess the magnitude of the influence of relativistic effects on chemical properties. From calculations of electron configurations of heavier elements, it is predicted that sudden changes in the structure of electron shells may appear due to relativistic effects which originate from the increasingly strong Coulomb field of a highly charged atomic nucleus.⁵ Therefore, it is expected that heavier elements show a drastic rearrangement of electrons in their atomic ground states, and as electron configurations are responsible for chemical behavior of elements, such relativistic effects can lead to surprising chemical properties. Increasing deviations from the periodicity of chemical properties based on extrapolation from lighter homologues in the periodic table are consequently predicted.^{2,6-8} It would be no longer possible to deduce detailed chemical properties of the transactinide elements simply from the position of the periodic table.

1																	18
H																	He
3	4															10	
Li	Be															Ne	
11	12															18	
Na	Mg	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
55	56	57	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
87	88	89	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118
Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	112	113	114	115	116	117	118
Lanthanides																	
57	58	59	60	61	62	63	64	65	66	67	68	69	70	71			
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu			
Actinides																	
89	90	91	92	93	94	95	96	97	98	99	100	101	102	103			
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr			

Figure 1. Periodic table of the elements.

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Chemical experiments with the transactinide elements are generally divided into the following 4 basic steps; i) synthesis of transactinide nuclides, ii) rapid transport of the synthesized nuclides to chemical separation apparatuses, iii) fast chemical isolation of a desired nuclide and preparation of a sample suitable for nuclear spectroscopy, and iv) detection of nuclides through their characteristic decay properties. Due to short half-lives and low production rates of the transactinide nuclides, each atom produced decays before a new atom is synthesized. This means that any chemistry to be performed must be done on an "atom-at-a-time" basis.^{2,9,10} Thus, rapid and very efficient radiochemical procedures must be devised. Recent comprehensive reviews on the chemistry of the transactinide elements are found in References 2, 9, and 10.

The first successful chemical isolation and identification of Bh was accomplished in an experiment at the Philips cyclotron of PSI (Paul Sherrer Institute) by Eichler et al.¹¹ The result clearly indicated that the volatile bohrium oxychloride complex behaved like a typical member of group 7 of the periodic table of the elements. From recent systematic studies of the anion-exchange behavior of Rf, it was found that Rf is a member of the group 4 elements,¹² although unexpected chemical behavior of Rf was observed in the fluoride complex formation.¹³

In this paper, chemical studies on gas chromatographic behavior of Bh at PSI and aqueous chemistry of Rf at JAERI are outlined.

2. Production of Transactinide Elements

Longer-lived transactinide nuclides required in chemical experiments exist in the neutron-rich region of the chart of nuclides and they are produced in so-called hot fusion reactions of such beams as ^{18}O , ^{19}F , ^{22}Ne , and ^{26}Mg with actinides targets of ^{244}Pu , ^{248}Cm , ^{249}Bk , and so on. The produced transactinide nuclides must be identified by measurement of their decay or by that of their known daughter nuclei with unambiguous detection techniques.

A schematic representation of a typical experimental set-up for the production of transactinide nuclides is shown in Figure 2 where the target and recoil chamber for the production of ^{261}Rf at JAERI are depicted. A ^{248}Cm target of $610 \mu\text{g}/\text{cm}^2$ thickness prepared by electrodeposition of $\text{Cm}(\text{NO}_3)_3$ from isopropyl alcohol onto $2.4 \text{ mg}/\text{cm}^2$ thick beryllium backing foil is bombarded by ^{18}O beams delivered from the JAERI tandem accelerator. The $^{18}\text{O}^{6+}$ beam passes through $2.0\text{-mg}/\text{cm}^2$ HAVAR entrance window foil, $0.09 \text{ mg}/\text{cm}^2$ He cooling gas

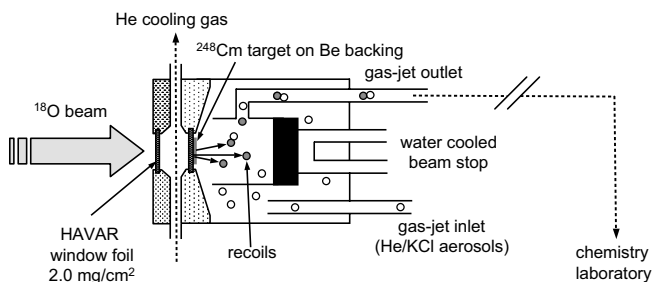


Figure 2. Schematic diagram of a target recoil chamber arrangement with a gas-jet system.

and the beryllium target backing, and it finally enters the ^{248}Cm target material. The reaction products recoiling out of the target are thermalized in a volume of He gas (≈ 1 bar) which is loaded with potassium chloride (KCl) aerosols generated by sublimation from the surface of KCl powder at 640°C . The products attached to the aerosols are swept out of the recoil chamber with the He gas flow (2.0 L/min) and are transported through a Teflon-capillary (length 20 m, inner diameter 2.0 mm) by a He/KCl gas-jet system to a chemistry laboratory.

The reactions and the expected production rates of the transactinide nuclides used for chemical studies are summarized in Table 1. Assuming the typical values of $800\ \mu\text{g}/\text{cm}^2$ for the target thickness and the beam intensity of $3 \times 10^{12}\ \text{s}^{-1}$, one can anticipate the production rates, for example, to be 4 atoms per minute for Rf and a 1 atom per hour for Bh.

3. Gas-phase Chemistry of Element 107, Bohrium (Bh)

Figure 3 shows the schematic of the experiment for the gas-phase chemistry of Bh.¹¹ A target of $670\ \mu\text{g}/\text{cm}^2$ ^{249}Bk covered with a layer of ^{159}Tb ($100\ \mu\text{g}/\text{cm}^2$) was bombarded for about 4 weeks with ^{22}Ne beams at a beam energy of 119 MeV to produce ^{267}Bh in the reaction $^{249}\text{Bk}(^{22}\text{Ne},4n)$. Reaction products recoiling from the target were adsorbed onto the surface of carbon particles in He gas and were then continuously transported with the gas-jet system to the chemistry apparatus OLG A III (On-Line Gas chromatography Apparatus).²⁰ The aerosol particles were collected on quartz wool inside the reaction oven kept at 1000°C . A mixture of HCl and O_2 as reactive gas was introduced to form volatile oxychlorides. The chromatographic separation took place downstream in the isothermal temperature section of the quartz column (length 1.5 m, inner diameter 1.5 mm). The retention times of compounds in the column depend mainly on their adsorption interaction with the surface of the column at the isothermal temperature and on the carrier gas velocity. Thus, volatile molecules with a nuclear lifetime longer than the retention time passed through the column, and were subsequently attached to CsCl aerosol particles in Ar gas and transported to the detection system ROMA (ROTating wheel Multidetector Analyzer). The CsCl particles deposited in vacuum on thin foils mounted on the circumference of a stepwise

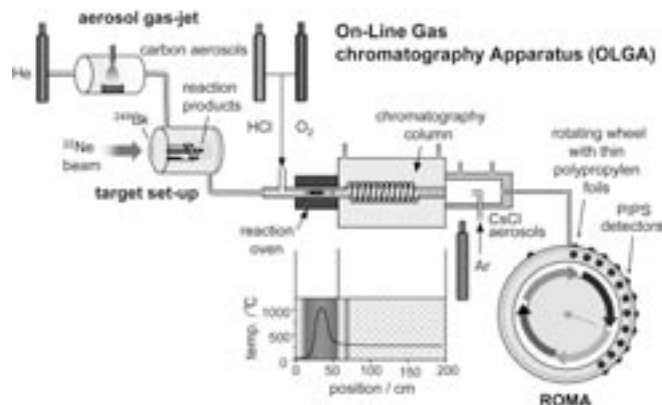


Figure 3. Schematic view of the Bh gas-chromatographic experiment. Courtesy of H. W. Gäggeler.

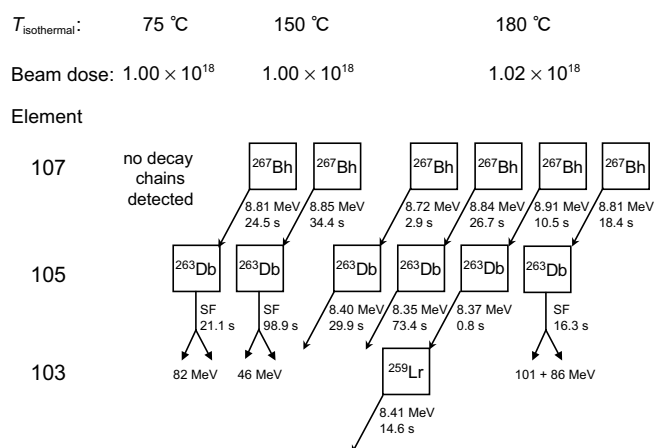


Figure 4. Decay chains attributed to the decay of ^{267}Bh . The observed α -particle and spontaneous fission decay energies and the lifetimes are given. Adapted from Reference 11.

rotating wheel. Every 10 s the collected samples were successively moved between a series of 12 pairs of PIPS (passivated ion-implanted planar silicon) detectors to measure the energy of α -particles and spontaneous fission decay. Measurements were performed at three different isothermal temperatures. Rhenium-186 was simultaneously produced in the reaction $^{159}\text{Tb}(^{22}\text{Ne},4n)$, and was served as a yield monitor for the chemical separation process. The overall yield, including transportation of the products with the gas-jet system, chemical yields, and sample preparation in the detection system, was on the average about 16% for ^{186}Re .¹¹ The yield of ^{186}Re was measured with an HPGe γ -ray detector.

The nuclide ^{267}Bh decays by α -particle emission to ^{263}Db , an isotope of element 105, which decays either by spontaneous fission or α -particle emission to ^{259}Lr . About 180,000 samples were measured throughout the experiment. As shown in Figure 4, four genetically linked decay chains attributed to the decay of

TABLE 1: Typical Nuclides Used in Chemical Studies of the Transactinide Elements

Atomic number	Nuclide	Half-life	Nuclear reaction	Production cross section	Production rate*	Reference
104	^{261}Rf	78 s	$^{248}\text{Cm}(^{18}\text{O},5n)$	13 nb	$4\ \text{min}^{-1}$	14
105	^{262}Db	34 s	$^{248}\text{Cm}(^{19}\text{F},5n)$	1.3 nb	$0.5\ \text{min}^{-1}$	14
105	^{262}Db	34 s	$^{249}\text{Bk}(^{18}\text{O},5n)$	6 nb	$2\ \text{min}^{-1}$	15
106	^{265}Sg	7.4 s	$^{248}\text{Cm}(^{22}\text{Ne},5n)$	0.24 nb	$5\ \text{h}^{-1}$	16
107	^{267}Bh	17 s	$^{249}\text{Bk}(^{22}\text{Ne},4n)$	60 pb	$1\ \text{h}^{-1}$	17
108	^{269}Hs	14 s	$^{248}\text{Cm}(^{26}\text{Mg},5n)$	6 pb	$3\ \text{d}^{-1}$	18
112	$^{283}112$	5.1 min	$^{238}\text{U}(^{48}\text{Ca},3n)$	3 pb	$1.5\ \text{d}^{-1}$	19

*Assuming typical values of $800\ \mu\text{g}/\text{cm}^2$ target thickness and $3 \times 10^{12}\ \text{s}^{-1}$ beam particles. Partly adapted from Reference 10.

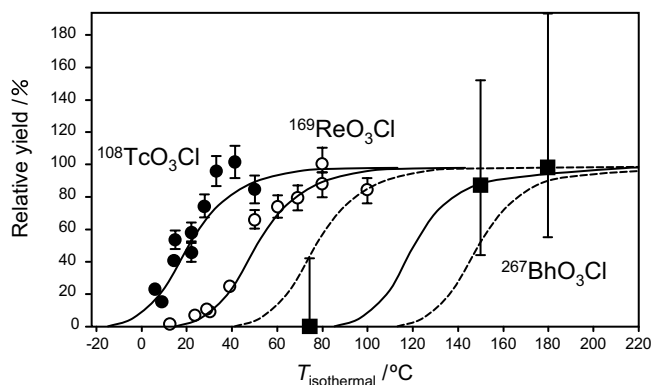


Figure 5. Isothermal gas chromatographic behavior of the oxychloride compounds of Bh, Tc, and Re. The solid lines indicate the results of simulations with the microscopic model with the adsorption enthalpies given in the text. The dashed lines represent the calculated relative yield concerning the 68% confidence interval of the standard adsorption enthalpy of BhO_3Cl from -66 to -88 kJ/mol. Adapted from Reference 11.

^{267}Bh were detected at an isothermal temperature of 180°C , while at 150°C , 2 decay chains were observed and no decay chains were registered at 75°C . A total of 6 atoms were detected during the experiment.¹¹

Figure 5 shows the isothermal gas chromatographic behavior of Bh oxychloride, presumably BhO_3Cl , together with that of the group 7 metal oxychlorides in quartz columns. The data for Tc were obtained using a ^{252}Cf spontaneous fission source in a separate experiment.²¹ The relative yields of BhO_3Cl after separation with the OLGAs device are plotted as a function of isothermal temperatures and are compared with those of TcO_3Cl and ReO_3Cl . The relative yield of BhO_3Cl observed at 180°C was normalized to 100%. Although no $^{267}\text{BhO}_3\text{Cl}$ was observed at 75°C , $^{169}\text{ReO}_3\text{Cl}$ still passed the isothermal part of the column with about 80% of relative yield. From the gas adsorption chromatographic behavior of those molecules, the following enthalpies of adsorption on the column surface were deduced: $-\Delta H_a^0$ (TcO_3Cl) = 51 ± 3 kJ/mol, $-\Delta H_a^0$ (ReO_3Cl) = 61 ± 3 kJ/mol, and $-\Delta H_a^0$ (BhO_3Cl) = 75^{+6}_{-5} kJ/mol (68% confidence interval).¹¹ The result clearly indicates that the sequence in volatility is $\text{TcO}_3\text{Cl} > \text{ReO}_3\text{Cl} > \text{BhO}_3\text{Cl}$. It is interesting to note that this sequence is consistent with predictions from relativistic density-functional calculations for group 7 oxychlorides²² and also from classical extrapolations of periodic trends.¹¹ This means that the volatile BhO_3Cl behaves like a typical member of group 7 of the periodic table.¹¹

4. Aqueous Chemistry of Element 104, Rutherfordium (Rf)

Rutherfordium-261 ($T_{1/2} = 78$ s) was produced at the JAERI tandem accelerator. The mixed target of ^{248}Cm ($610 \mu\text{g}/\text{cm}^2$ in thickness) and Gd (39.3%-enriched ^{152}Gd of $36 \mu\text{g}/\text{cm}^2$ in thickness) prepared by electrodeposition onto $2.4\text{-mg}/\text{cm}^2$ beryllium foil was used for simultaneous production of ^{261}Rf and 3.24-min ^{169}Hf via the $^{248}\text{Cm}(^{18}\text{O},5n)$ and $\text{Gd}(^{18}\text{O},xn)$ reactions, respectively. Hafnium-169 was served as a monitor of the on-line chemistry experiment. The reaction products recoiling out of the target were transported through a Teflon-capillary to the chemistry laboratory by the He/KCl gas-jet system as mentioned in Section 2. To perform rapid and cyclic anion-exchange experiments with Rf, we employed an apparatus AIDA (Automated Ion-exchange separation apparatus coupled with the Detection system for Alpha-spectroscopy)^{12,23} that consisted of a modified ARCA (Automated Rapid Chemistry Apparatus),²⁴ which was a miniaturized computer controlled liquid chromatography system, and an automated on-line α -particle detection system. AIDA enables us to perform cyclic discontinuous column chromatographic separation of short-lived nuclides in

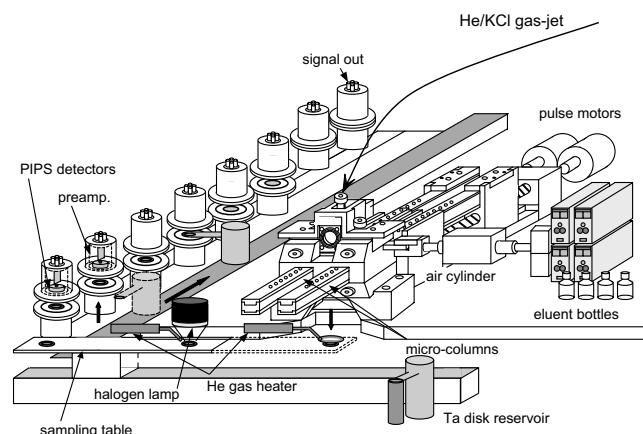


Figure 6. Schematic drawing of AIDA (Automated Ion-exchange separation apparatus coupled with the Detection system for Alpha spectroscopy). Adapted from Reference 23.

aqueous solutions and automated detection of α -particles within a typical cycle of 1–2 min. A schematic drawing of AIDA is given in Figure 6. There are two column magazines, each containing 20 micro columns. On-line anion-exchange experiments on Rf together with the group 4 elements Zr and Hf in acidic solutions, HCl, HNO_3 , and HF, were conducted with AIDA. The adsorption behavior of Rf as a function of the acid concentration was systematically studied.^{12,13} Quite recently, an elution curve of Rf has been obtained for the first time in anion-exchange chromatography on an atom-at-a-time basis.²⁵

In the following, the experimental procedures and the result on the anion-exchange behavior of Rf in HCl solution are summarized. The reaction products continuously transported through the He/KCl gas-jet system deposited on a collection site of AIDA. After collection for 125 s, the site was mechanically moved on the top of one of the micro columns, where the products were dissolved with $170 \mu\text{L}$ of hot ($\approx 80^\circ\text{C}$) 11.5 M HCl and then fed onto the chromatographic column (length 7mm, inner diameter 1.6 mm) filled with an anion-exchange resin MCI GEL CA08Y (particle size of about $20 \mu\text{m}$) at a flow rate of 1.0 mL/min. The products were eluted with $290 \mu\text{L}$ of 4.0–9.5 M HCl at a flow rate of 1.1 mL/min. The effluent was collected on a Ta disk as fraction 1 and then evaporated to dryness using hot He gas and a halogen heat lamp for α -particle measurement. The remaining products in the column were eluted with $250 \mu\text{L}$ of 4.0 M HCl at a flow rate of 1.1 mL/min. The effluent was collected on another Ta disk and evaporated to dryness as fraction 2. The pair of disks was automatically transported to the α -spectroscopy station equipped with PIPS detectors. After the α -particle measurement, γ -radiation of ^{169}Hf was monitored with Ge detectors to determine the elution behavior of Hf and its chemical yield. Each separation was accomplished within 20 s and the α -particle measurement was initiated within 80 s after the collection of products at the AIDA collection site. The chemical yield of Hf, including collection and dissolution of the aerosols at the AIDA collection site, was approximately 60%.¹²

From 1893 cycles of the anion-exchange experiments, a total of 186 α events from ^{261}Rf and its daughter ^{257}No were registered, including 35 α - α correlation events.¹² Figure 7 shows the adsorption behavior of Rf, Zr, and Hf as a function of HCl concentration. We measured the adsorption probability of Zr and Hf that were produced in the reactions $\text{Ge}(^{18}\text{O},xn)$ and $\text{Gd}(^{18}\text{O},xn)$, respectively, under strictly identical conditions as those with Rf and Hf.¹⁵ The ordinate shows the adsorption probabilities of these elements, $100A_2/(A_1+A_2)$, where A_1 and A_2 are the eluted radioactivities observed in fractions 1 and 2, respectively. The adsorption behavior of Rf is quite similar to that of group 4 elements Zr and Hf. The adsorption of these

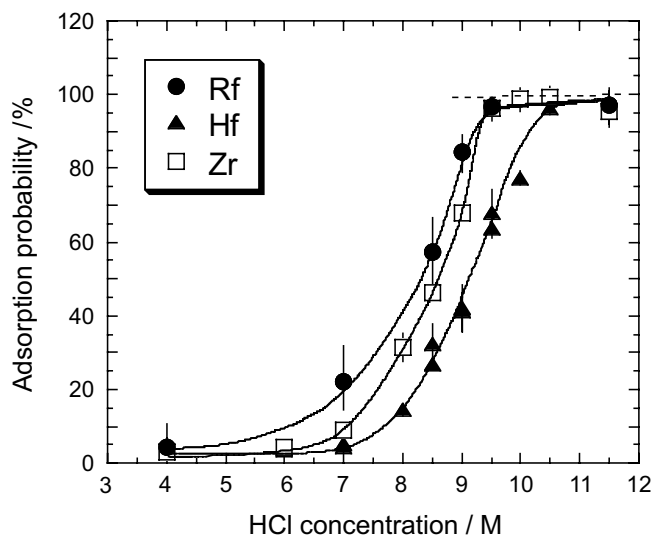


Figure 7. Adsorption probability of Rf, Zr, and Hf on the anion-exchange resin CA08Y as a function of HCl concentration. Adapted from Reference 12.

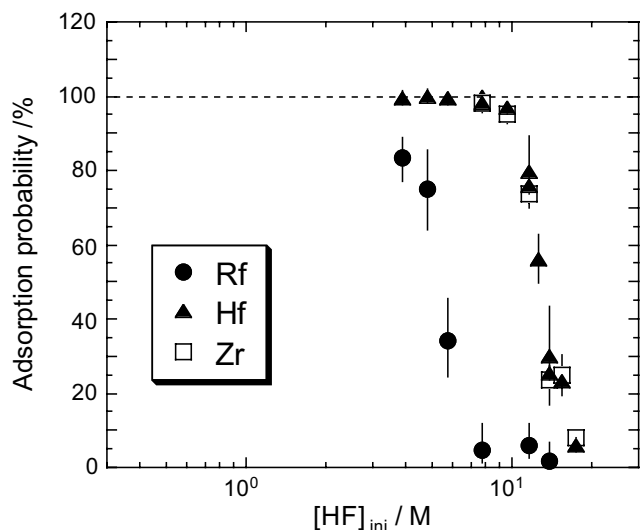


Figure 8. Adsorption behavior of Rf, Zr, and Hf on the anion-exchange resin CA08Y as a function of the initial concentration of HF, $[HF]_{ini}$. Adapted from Reference 13.

elements rapidly increases with increasing HCl concentration higher than 7 M: typical anion-exchange behavior of group 4 elements. This means that anionic chloride complexes of tetravalent Rf, Zr, and Hf are formed above 7 M concentration of HCl.¹²

Similar behavior of Rf was also probed with the anion-exchange experiment in 8 M HNO₃. From 217 experiments, a total of 20 α events from ²⁶¹Rf and ²⁵⁷No were observed, including 5 time-correlated α pairs. It was found that Rf was eluted from the column with 8 M HNO₃ as expected for a typical group 4 element.¹² The above results definitely confirm that Rf is an element of group 4.

Unexpected anionic-exchange behavior of Rf in HF solution was observed. The experimental procedures with AIDA are basically the same as those described above and the details are given in Reference 13. From 4226 cycles of the anion-exchange experiments, a total of 266 α events from ²⁶¹Rf and its daughter ²⁵⁷No were registered, including 25 time-correlated α pairs of ²⁶¹Rf and ²⁵⁷No. Figure 8 shows a variation of the adsorption probability of Rf, Zr, and Hf as a function of the initial concentration of HF, $[HF]_{ini}$.¹³ Adsorption behavior of Zr was obtained from the experiment using the Ge and Gd mixed target.¹³ The adsorption of Zr is fairly equal to that of Hf over a wide range of HF concentrations and steeply decreases with $[HF]_{ini}$, while

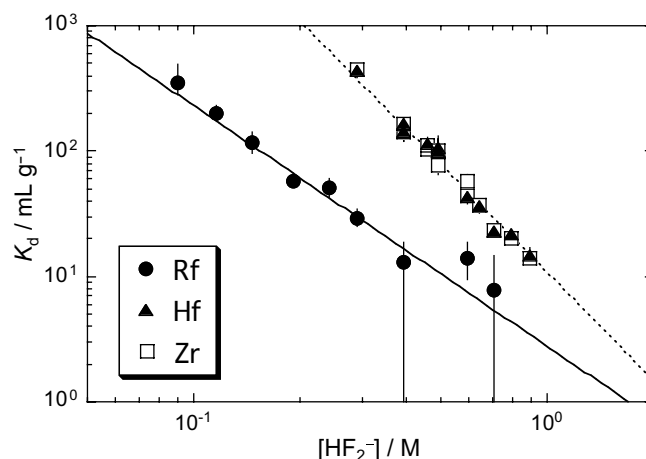
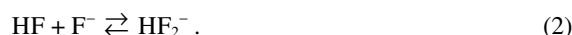
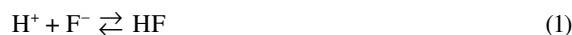


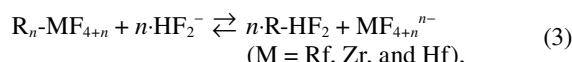
Figure 9. Distribution coefficients (K_d) for Rf, Zr, and Hf on the anion-exchange resin CA08Y as a function of the concentration of HF_2^- . Adapted from References 13 and 23.

that of Rf decreases at much lower $[HF]_{ini}$. The lower adsorption of Rf indicates that the fluoride complexation of Rf is weaker than those of Zr and Hf.

The dissociation of HF is expressed as,²⁶



In terms of the dissociation constants $K_1 = 935 \text{ M}^{-1}$ for Equation (1) and $K_2 = 3.12 \text{ M}^{-1}$ for Equation (2), above 1 M $[HF]_{ini}$, the concentration of anionic HF_2^- is more than one order of magnitude higher than that of free F^- . The K_d values for Rf, Zr, and Hf are plotted in Figure 9 as a function of the concentration of HF_2^- , $[HF_2^-]$. The K_d values for Rf were deduced from the adsorption probability in Figure 8 by assuming that the reaction rates in the fluoride complexation and the anion-exchange process of Rf are as fast as those of the homologues Zr and Hf as described in Reference 13. The K_d values for Zr and Hf were from the on-line column chromatographic experiments with AIDA. A decrease of K_d values for these elements with $[HF_2^-]$ is explained as the displacement of the metal complex from the binding sites of the resin with the counter ion HF_2^- as,



where R denotes the resin. As shown in Figure 9, K_d values of these elements decrease linearly with increasing $[HF_2^-]$ in the log K_d vs. log $[HF_2^-]$ plot. It should be noted that the slopes for Zr and Hf are clearly -3 (dashed line), while that for Rf is -2 (solid line). This means that Rf is likely to be present as a hexafluoro complex, $[RfF_6]^{2-}$ similar to the well known $[ZrF_6]^{2-}$ and $[HfF_6]^{2-}$ at lower $[HF]_{ini}$, while Zr and Hf are likely to be present in the form of heptafluoro complexes, $[ZrF_7]^{3-}$ and $[HfF_7]^{3-}$ as suggested in References 27 and 28. Activity coefficients of the chemical species involved are not available for higher concentration of HF, and the definite identification of the anionic fluoride complexes is still an open question.

Relativistic density-functional calculations of the electronic structures of hexafluoro complexes ($[MF_6]^{2-}$, M=Rf, Zr and Hf) have been performed to evaluate the stability of the complexes. The results of calculations that the sequence in the overlap population between the valence d orbitals of M^{4+} and the valence orbitals of F^- is $Zr \approx Hf > Rf$ suggest that the Rf complex is less stable than those of Zr and Hf in the $[MF_6]^{2-}$ structure.²⁹ Although the calculations qualitatively agree with the experimental results, further theoretical approaches are

needed to quantitatively understand the present interesting results.

5. Summary

Chemical studies of Bh and Rf on an atom-at-a-time basis gave the following outcomes. Bohrium behaves like a typical member of group 7 of the periodic table and forms a volatile oxychloride, presumably BhO_3Cl , which is less volatile than those of the lighter homologues Tc and Re. The adsorption behavior of Rf in anion-exchange chromatography is quite similar to that of Zr and Hf in HNO_3 and HCl solutions, clearly confirming that Rf is an element of group 4. On the other hand, a large difference was observed in the adsorption behavior of Rf on the anion-exchange resin in HF solution, i.e., the fluoride complexation of Rf is much weaker than those of the homologues.

From artistic chemical studies of the transactinide elements performed so far including the results described here, one can place the elements 104 through 108 in their respective group of the periodic table, i.e., Rf, Db, Sg, Bh, and Hs in group 4, 5, 6, 7, and 8, respectively.^{2, 7, 30} However, as observed in the fluoride complexation of Rf, unexpected chemical behavior partly caused by relativistic effects would appear in chemical properties of the transactinide elements. To enhance the understanding on chemical properties of the transactinide elements, more systematic experiments as well as quantitative theoretical calculations are required.

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