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ANP CHEMISTRY SECTION PROGRESS REPORT

Phase Equilibria

The System RbF-UF₄

The system RbF-UF₄ has been investigated recently because of its importance in the quaternary fuel system NaF-RbF-ZrF₄-UF₄. The earlier work of C. J. Barton et al.,¹ is largely confirmed by thermal analysis of slowly-cooled melts. In addition to the compounds originally reported, there is an incongruent compound 7RbF·6UF₄.

Quenching experiments on the system are now underway, but the work is not yet far enough advanced to present a phase diagram of the system. Petrographic examinations of a quench series containing 33.3 mole % UF₄ indicated that a rapid inversion of 2RbF·UF₄ occurs at some temperature below 572°C. The congruent melting point of RbF·UF₄ (727°C) was confirmed by petrographic examination of a gradient quench series of a 50 mole % UF₄ composition. The composition of an incongruent compound previously thought to be RbF·4UF₄ has now been established as RbF·3UF₄ by the fact that single phase material was found in an equilibrated quench series of a composition containing 75 mole % UF₄. This phase is not stable above 714°C. In the region between 50 and 100 mole % UF₄ two phases in addition to RbF·3UF₄ have been observed in both slowly-cooled and quenched samples. One contains more than 80 mole % UF₄ while the composition of the other phase lies between 50 and 66.7 mole % UF₄.

The System NaF-RbF-ZrF₄-UF₄

Two areas of the quaternary system NaF-RbF-ZrF₄-UF₄ offer mixtures which may be suitable fuels for circulating fused salt reactors.

Solvent compositions near 10 NaF-52 RbF-38 ZrF₄ (mole %) provide a 4 mole % UF₄ fuel with liquidus temperatures of approximately 500°C, and a 7 mole % UF₄ fuel with liquidus temperatures of approximately 545°C. Mixtures in this solvent area display RbF·UF₄ or RbF·3UF₄ as the primary phase. Serious segregation of the uranium phase has not been evident in the experimental work. These mixtures are not optimal choices as a fuel because (1) the uranium phase contains a high concentration of UF₄, and (2) there

1. C. J. Barton et al., ANP Quar. Prog. Rep. June 10, 1951, ANP-65, p. 134.

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is a large difference between the temperature at which the uranium phase and the primary phase of the solvent separates.

Solvent compositions near 35 NaF-25 RbF-40 ZrF₄ (mole %) provide a 4 mole % UF₄ fuel with liquidus temperatures of approximately 480°C and a 7 mole % UF₄ fuel with liquidus temperatures of approximately 510°C. At both UF₄ levels the uranium is contained entirely in the solid solution 7NaF·6Zr(U)F₄.

The System NaF-RbF-BeF₂

Further study of the 1:1:1 (molar ratio) composition in the NaF-RbF-BeF₂ system that was reported to contain two phases¹ showed that one of the phases has the probable formula Na₂RbBeF₅ while the other principle phase is RbBeF₃. A small amount of Rb₂BeF₄ was also noted in the X-ray diffraction pattern. Study of other mixtures on the NaF-RbBeF₃ join indicates that NaF is probably the primary phase from 50 to 100% NaF and that the Na₂RbBeF₅ compound probably melts incongruently. The thermal analysis data show that NaF lowers the incongruent melting point of RbBeF₃ and a shift in the X-ray lines of this compound was also noted. Quenching studies with several compositions on this join will be needed to give a clearer picture of the phase relationships in this region. Melting points in this system are much higher in general than in the NaF-LiF-BeF₂ system but mixtures high in NaF and BeF₂ content give very poorly marked thermal effects on cooling curves in both systems so that comparisons on the basis of thermal analysis data are not significant. Quenching studies are planned or in progress in both systems in order to obtain reliable liquidus temperatures in parts of the systems that are not amenable to thermal analysis.

The System NaF-LiF-BeF₂

Additional quenching studies indicate that the ternary compound 5NaF·LiF·3BeF₂ reported earlier¹ decomposes to give LiF, Na₂LiBe₂F₇ and Na₂BeF₄ at 320°C, below the solidus temperature for this composition. Since only one composition in this system has been found that melts below 320°C, the ternary eutectic near the composition NaF-LiF-BeF₂ (52-6-42 mole %) melting at about 318°C, it appears unlikely that the above-mentioned compound will have a primary phase field of appreciable size in the system. There are now believed to be three compatibility triangles in the half of

1. (ANP) Chemistry Section Progress Report for February and March, 1956, CF 56-4-161.

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the system comprised by the compounds LiF-NaF-NaBeF_3 . They are:

Binary and ternary eutectic temperatures and compositions have been determined with fairly good accuracy so that phase boundaries and drainage paths can be drawn for this part of the system. Mixtures covering a rather large area of this part of the system having BeF_2 concentrations of 35 mole % BeF_2 or more and LiF concentrations of about 5 to 25 mole % LiF melt below 350°C . Available viscosity data for ternary mixtures in this region indicate that the kinematic viscosity of mixtures containing less than 38 mole % BeF_2 is not excessively high.

The System $\text{UF}_4\text{-UO}_2$

Preliminary data for mixtures in this system were reported recently.¹ In recent experiments, heating and cooling curves with mixtures containing from 2 to 10 weight % of UO_2 have been obtained in sealed nickel capsules to minimize changes in oxide content of the mixtures while cooling curves were being run. The previously mentioned undercooling difficulty continued to make both liquidus and solidus determinations from cooling curves unreliable. However, heating curves with mixtures containing 4, 6, 8 and 10 weight % UO_2 , as well as a few cooling curves, showed evidence of a eutectic melting at approximately 910°C . Extrapolation of the available liquidus temperature data involves a considerable amount of uncertainty but it appears that the eutectic composition probably contains 9 to 11 weight % UO_2 .

Alkali Fluoride- CeF_3 Systems

Thermal analysis data on the system LiF-CeF_3 show that there is a eutectic composition melting at $755^\circ\text{C} \pm 5^\circ$ containing about 19 mole % CeF_3 . Extrapolation of thermal analysis data on three mixtures in the NaF-CeF_3 systems indicates that the eutectic in this system melting at $730^\circ\text{C} \pm 5^\circ$ contains approximately 28 mole % CeF_3 . Study of the RbF-CeF_3 system continued but the available data do not give a clear picture of phase relationships in this system.

1. ~~ANP Chemistry Section Progress Report for February and March 1956, CF 56-4-161.~~

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~~SECRET~~The System MgF_2 - CaF_2

In the course of an investigation of the system NaF - MgF_2 - CaF_2 ¹ it was noted that MgF_2 - CaF_2 mixtures showed a higher eutectic temperature than that shown in the literature.² Therefore, cooling curves were obtained for several mixtures in the vicinity of the eutectic. The data obtained show that the eutectic contains approximately 43.5 mole % CaF_2 and melts at $985^\circ\text{C} \pm 5^\circ$ as compared to the literature value of 940°C .

A Method to Aid Eutectic Composition Determination

A technique for aiding in the determination of eutectic compositions has shown some promise. A capsule with a side arm to allow evacuation and containing a 60-mesh silver screen at right angles to the capsule wall is heated with pre-melted pellets of the presumed eutectic mixture on top of the screen. A thermocouple located on the wall of the capsule section containing the pellets and one located on the bottom of the capsule, which is out of the heating zone, give the pellet temperature and indicate when melting has commenced, respectively. When melting is noted the heat source is removed and the tube cooled by an air blast.

Analysis of the filtrate and residue give an indication of the eutectic composition although it is recognized that the filtrate may mechanically entrain some of the still solid phases of the residue and thus not be the true eutectic composition.

A mixture believed to be at or near a eutectic and having the composition NaF - KF - ZrF_4 (24-33-43 mole %) was heated to 420°C (slowly) before dripping was noted by a sudden rise in temperature of the bottom thermocouple. The results are shown in Table 1.

Table 1Results of Heating to 420°C NaF - KF - ZrF_4 (24-33-43 mole %)

	<u>NaF</u>	<u>KF</u>	<u>ZrF₄</u>
Residue	29.8	29.0	41.2
Filtrate	23.3	34.0	42.7

1. ~~ANP Quar. Prog. Rep. for Period Ending March 10, 1956, ORNL-2061.~~
2. E. Beck, Metallurgie 5, 504 (1908).

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A new mixture containing 23-34-43 mole % NaF-KF-ZrF₄ was prepared and heated in the apparatus to 420°C, at which temperature the entire mixture melted through the screen leaving no residue. From this it would seem that the composition of the first drops correspond closely to the eutectic composition.

Chemical Equilibria

Reaction of UF₄ with Cr⁰ in LiF-ZrF₄

Further studies have been made on the reaction of UF₄ with Cr⁰ at 600 and 800°C using LiF-ZrF₄ (52-48 mole %) as the reaction medium. In these latest studies iodide chromium of high purity (10 ppm or less oxygen) obtained from Battelle Memorial Institute was used in place of the electrolytic chromium that has been employed in all previous studies. The iodide chromium was not hydrogen fired so that a comparison of the blanks obtained with this pure chromium to those found for the electrolytic chromium, hydrogen fired under the standard conditions, should demonstrate the effectiveness of the hydrogen treatment. Data obtained for the iodide chromium using 12.0 wt. % UF₄ (3.9 mole %) are given in Table 2.

Table 2

Data for the Reaction of UF₄ with Cr in Molten LiF-ZrF₄

(52-48 mole %) at 600 and 800°C

<u>Conditions of Equilibration</u>		<u>Present in Filtrate</u>		
<u>Temp.</u> <u>(°C)</u>	<u>Time</u> <u>(hrs)</u>	<u>Total</u> <u>U</u> <u>(wt.%)</u>	<u>Total</u> <u>Cr*</u> <u>(ppm)</u>	<u>Total</u> <u>Ni</u> <u>(ppm)</u>
600	5	9.5	2800	50
600	5	9.5	2790	50
800	5	9.2	3810	40
800	5	9.3	3770	60

* Blank of 190 ppm of Cr at 800°C.

A blank of 250 ppm was reported previously¹ for this system using hydrogen fired electrolytic chromium which is in fair agreement with the 190 ppm reported in Table 2 for the unfired iodide

1. ANP Chemistry Section Progress Report for February and March 1956, CF 56-4-161.

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chromium. It is evident from these values that the hydrogen firing procedure effectively reduces the oxide present in the electrolytic chromium (blank of 900 ppm without firing). A large portion of the 190 ppm of chromium reported in Table 2 must arise from oxidizing materials (H_2O and HF) present in the $LiF-ZrF_4$ mixture. The equilibrium concentrations of chromium arising from the reduction of UF_4 as given in Table 2 are in very good agreement with those previously¹ reported for the runs using electrolytic chromium. The averages of six runs for the latter are 2930 and 3890 ppm at 600 and 800°C, respectively.

Reaction of UF_4 with Fe^0 in $KF-ZrF_4$

The reaction of UF_4 with Fe^0 has been studied at 600 and 800°C using $KF-ZrF_4$ (52-48 mole %) as reaction medium. Data for this system using 10.6 wt. % UF_4 (4.0 mole %) are given in Table 3.

Table 3

Data for the Reaction of UF_4 with Fe^0 in Molten $KF-ZrF_4$

(52-48 mole %) at 600 and 800°C

Conditions of Equilibration		Present in Filtrate		
Temp. (°C)	Time (hrs)	Total U (wt. %)	Total Fe* (ppm)	Total Ni (ppm)
600	3	7.2	520	<1
600	3	8.0	630	10
600	5	7.7	490	5
600	5	7.9	510	10
800	3	8.2	330	65
800	3	8.1	280	30
800	5	8.1	310	70
800	5	8.1	330	35

* Blank of 40 ppm of Fe at 800°C

The iron values listed in Table 3 are in fair agreement with those reported¹ for the $LiF-ZrF_4$ case and also are similar to virtually all the other solvents studied. It will be noted that the present values, in agreement with those obtained for most of the other solvents, show a higher equilibrium concentration of iron at 600 than at 800°C.

1. ANP Chemistry Section Progress Report for February and March, 1956, CF 56-4-161.

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Reaction of UF_4 with Cr^0 in KF-ZrF_4

The reaction of UF_4 with Cr^0 has been studied at 600 and 800°C using KF-ZrF_4 (52-48 mole %) as the reaction medium. The results obtained for this system using 10.6 wt. % UF_4 (4.0 mole %) are given in Table 4.

Table 4

Data for the Reaction of UF_4 with Cr^0 in Molten KF-ZrF_4
(52-48 mole %) at 600 and 800°C

<u>Conditions of Equilibration</u>		<u>Present in Filtrate</u>		
<u>Temp.</u> (°C)	<u>Time</u> (hrs.)	<u>Total U</u> (wt. %)	<u>Total Cr*</u> (ppm)	<u>Total Ni</u> (ppm)
600	3	8.0	1030	35
600	3	9.4	1060	55
600	5	9.7	1180	55
600	5	8.5	1060	235
800	3	8.2	1250	35
800	3	8.4	1190	25
800	5	8.1	1150	295
800	5	8.1	1150	285
800	16	8.2	1060	30
800	16	8.2	1135	5

* Blank of 290 ppm of Cr at 800°C.

The equilibrium chromium concentration reported in Table 4 are considerably smaller and less temperature dependent than those found for the $\text{UF}_4\text{-Cr}^0$ system using either NaF-ZrF_4 (53-47 mole %) or LiF-ZrF_4 (52-48 mole %) as reaction medium. Equilibrium constants calculated from mole fractions assuming the chromium to be divalent are 2.4×10^{-5} and 3.2×10^{-5} at 600 and 800°C, respectively in the KF-ZrF_4 mixture.

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~~SECRET~~Compatibility of High Temperature MaterialsEffect of Atmosphere on Anodic Dissolution of Nickel in Molten NaOH

An attempt has been made to study the efficiency of anodic dissolution of Ni in NaOH under various atmospheres and various conditions of current density and temperature.

The apparatus consisted of a crucible of Morganite (recrystallized alumina) containing molten sodium hydroxide into which a nickel anode and cathode as well as a nickel thermo well were inserted. This electrolytic cell with its furnace was placed in a large chamber which could be evacuated or filled with the desired atmosphere. A lead storage battery served as the power supply; the circuit also contained a volt meter, ammeter and ampere hour meter as well as rheostats to control the current density applied.

When the assembled electrolytic cell was maintained at temperature without applied voltage, attack on the electrodes was negligible. Passage of current resulted in erosion of the anode with subsequent deposition of crystalline nickel in the cathode. Weight gain of the cathode did not correspond precisely to weight loss of the anode since the crystalline deposit is not perfectly adherent; in addition some sodium aluminate formed by reaction of NaOH with the Al_2O_3 apparently contaminates the cathode deposit. Loss in weight of the anode was accordingly used to evaluate the efficiency of the cell.

The data shown in Table 5 below was obtained at current density of 2 amps/cm². At this current density the cell efficiency is very poor under all conditions but the dissolution efficiency is decreased slightly by substituting H₂ for He as static cover gas and is reduced considerably by bubbling H₂ through the molten electrolyte.

Table 5

Efficiency of Anodic Solution of Ni⁰ in NaOH

Temp. (°C)	Cell Efficiency (%)		
	Under He	Under Static H ₂	While H ₂ Bubbled through NaOH
350	0.01	0.006	--
600	0.65	0.42	--
800	2.77	2.12	0.66

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~~SECRET~~Penetration of Molten Fluorides into Graphite

Some recent proposals on circulating fuel reactors contain arrangements in which the molten salt would be in contact with graphite. As no systematic work on penetration of graphite by molten fluorides has been done some effort has been expended on such determinations.

Preliminary results from soaking APC graphite cubes (1/4" x 1/4" x 1/2") in NaF-ZrF₄ (53-47 mole %) indicate complete penetration after one hour at 600°C. When the same type cubes were soaked for 10 hours at 600°C in NaF-ZrF₄-UF₄ (53.5-40.0-6.5 mole %) no penetration was detectable.

Physical Properties of Molten MaterialsThe "Snow" Problem as a Function of Composition in FuelsContaining ZrF₄

An important family of solvents for UF₄, giving rise to some low melting solutions of interest as reactor fuels, is comprised of ZrF₄ with the alkali fluorides. In such mixtures ZrF₄ is virtually the only volatile constituent and for practical purposes the partial pressure of ZrF₄ is equal to the total vapor pressure of the solution. The vapor condenses as a sublimate or "snow" of solid ZrF₄ causing considerable inconvenience in off-gas lines. The amount of "snow" produced by a particular fuel is directly proportional to the vapor pressure. The most relief from the snow problem that could be expected in the most favorable case would be about one tenth the amount of snow experienced with the current fuel (NaF-ZrF₄-UF₄, 50-46-4 mole %).

Suitably low melting points are found only in the vicinity of 50 mole % ZrF₄. For this reason, Figure 1 shows the decrease in vapor pressure between 60% and 40% ZrF₄. The data in Figure 1 which result from recent measurements on a complete series of liquid KF-ZrF₄ and RbF-ZrF₄ compositions giving pressures ranging from 10 mm to 250 mm Hg at temperatures below 1000°C show that the vapor pressure depends not only on the concentration of ZrF₄, but also on which alkali fluoride is present. The pressure range was determined by the limitations of the method,¹ which was the same

1. The method, due to W. H. Rodebush and A. L. Dixon, Phys. Rev., 26, 851 (1925), depends on using the vapor as a valve for inert gas; the inert gas escapes through the valve until its pressure equals that of the vapor.

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as used earlier to study the NaF-ZrF_4 ¹ and the LiF-ZrF_4 ² systems.

Vapor pressures in the NaF-ZrF_4 system have also been studied by a transpiration method;³ the disagreement between the two methods could not be explained by known experimental errors. The amount of UF_4 required in fuel mixtures does not alter the relative pressures developed with the various alkali fluorides and causes only a small decrease in the absolute pressure.

A small cation such as Li^+ has a relatively large $\frac{e}{r}$ ratio (where e is the charge and r the radius of the ion) and accordingly has a strong electrostatic attraction for fluoride ions. This reduces the activity of fluoride ions, and causes a relatively large ZrF_4 pressure. With larger ions, such as RbF , the activity of fluoride ions is increased and the ZrF_4 is more tightly bound in complexes such as ZrF_5^- ions, with a consequent decrease in vapor pressure.

The curves in Figure 1 were drawn from a large body of data covering an extensive range of compositions, pressures and temperatures. Tables 6 and 7 summarize the data applicable to KF-ZrF_4 and RbF-ZrF_4 respectively. The main theoretical interest in the vapor pressures derives from the fact that they provide a direct measure of the activity of ZrF_4 . From ZrF_4 activities, the relative alkali fluoride activities can be computed; these have a controlling influence on the complexing which governs the extent of the corrosion reactions. In general, less corrosion is expected from the mixtures having lower vapor pressures because UF_4 is more tightly complexed in such solution.

A peculiarity of the heats of vaporization shown under the column headed ΔH_v in Tables 6 and 7 is a linear decrease with composition (decreasing mole % ZrF_4) to a minimum at 50 mole % followed by a linear increase with composition as the mole % ZrF_4 decreases still further. The increase below 50 mole % ZrF_4 is doubtlessly due to increased complexing as the concentration of fluoride ions increases. The increase above 50 mole % ZrF_4 was not expected. The most plausible explanation would be that the region above 50 mole % consists of a solution of ZrF_4 in NaZrF_5 and that ZrF_4 exhibits positive deviation from the Raoult's law line between pure ZrF_4 and NaZrF_5 . This explanation is not satisfactory because there is no good evidence of the postulated positive deviation. Moreover, the close adherence to linearity and the sharp break at the minimum appear very unnatural.

1. R. E. Moore and C. J. Barton, ANP Quar. Prog. Rep. Dec. 10, 1953, ORNL-1649, p. 99; June 10, 1954, ORNL-1729, p. 101; Sept. 10, 1954, ORNL-1771, p. 129.
2. R. E. Moore and C. J. Barton, ANP Quar. Prog. Rep. June 10, 1955, ORNL-1896, p. 81.
3. K. A. Sense, et al., BMI-1064, January 9, 1956.

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Table 6

Summary of Vapor Pressures for the System KF-ZrF₄

$$\log_{10} P_{(\text{mm})} = A - \frac{B}{T^{\circ}\text{K}}$$

Composition (mole %)		A	B	ΔH_v ZrF ₄ kcal	Temp. Range of Measurement (°C)
ZrF ₄	KF				
65.9	34.1	9.754	8,813	40.4	757-905
59.6	40.4	9.142	8,321	38.1	751-889
55.0	45.0	8.403	7,778	35.6	789-901
49.9	50.1	7.722	7,363	33.7	821-936
47.3	52.4	7.679	7,467	34.2	875-932
45.3	54.7	7.649	7,721	35.3	959-1004
40.4	59.6	7.612	8,465	38.8	987-1105
34.9	65.1	7.387	9,020	41.3	1132-1290

Table 7

Summary of Vapor Pressures for the System RbF-ZrF₄

ZrF ₄	RbF	A	B	ΔH_v ZrF ₄ kcal	Temp. Range of Measurement (°C)
ZrF ₄	RbF				
81.6	18.4	10.778	9,506	43.5	802-853
74.6	25.4	10.359	9,189	42.1	721-812
70.0	30.0	9.808	8,790	40.3	758-818
65.1	34.9	9.143	8,358	38.3	742-866
60.0	40.0	8.789	8,078	37.0	773-863
54.8	45.2	8.307	7,902	36.2	789-934
50.4	49.6	7.555	7,310	33.5	849-977
46.2	54.8	7.512	7,794	35.7	902-1052
40.0	60.0	7.290	8,146	37.3	983-1180
35.0	65.0	7.296	8,790	40.3	1128-1273

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EFFECT OF ALKALI CATION ON VAPOR PRESSURE
OF BINARY FUEL SOLVENTS CONTAINING ZrF_4

(MF- ZrF_4 Mixtures at 800°C)

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Production of Purified Fluoride Mixtures

During April the 250 lb. fluoride production facility processed 10 batches totaling about 2500 pounds of purified fluoride compositions using the new copper-lined stainless steel equipment. The research-scale facilities processed 11 batches of varying compositions for use in small-scale corrosion testing and physical property studies.

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