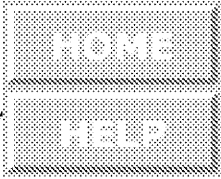


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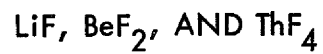
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SOLUBILITY OF CERIUM TRIFLUORIDE IN MOLTEN MIXTURES OF
LiF, BeF₂, AND ThF₄

Judy A. Fredricksen*, L. O. Gilpatrick,
C. J. Barton

JANUARY 1969

* Summer ORAU Participant from St. Cloud State College,
St. Cloud, Minnesota

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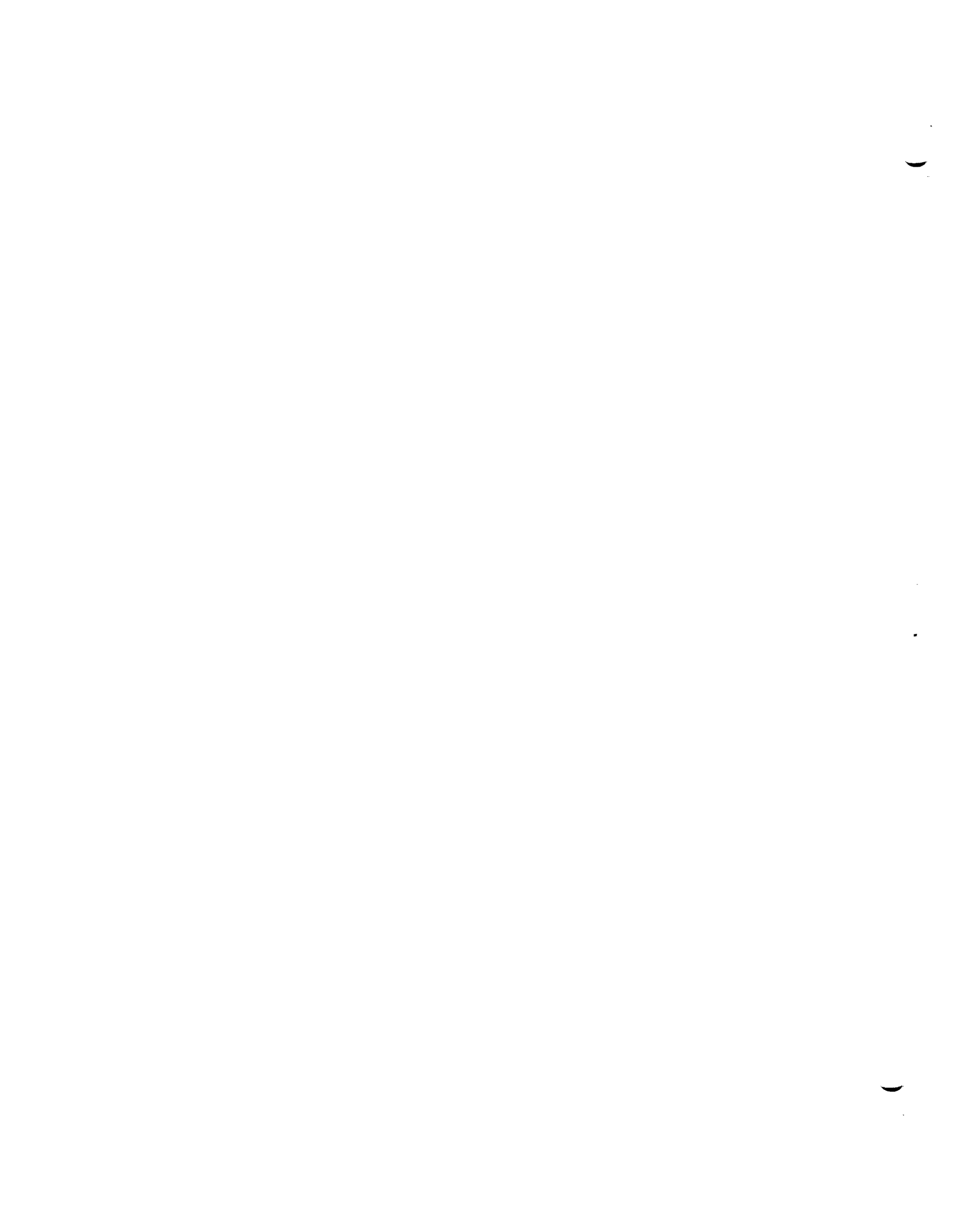
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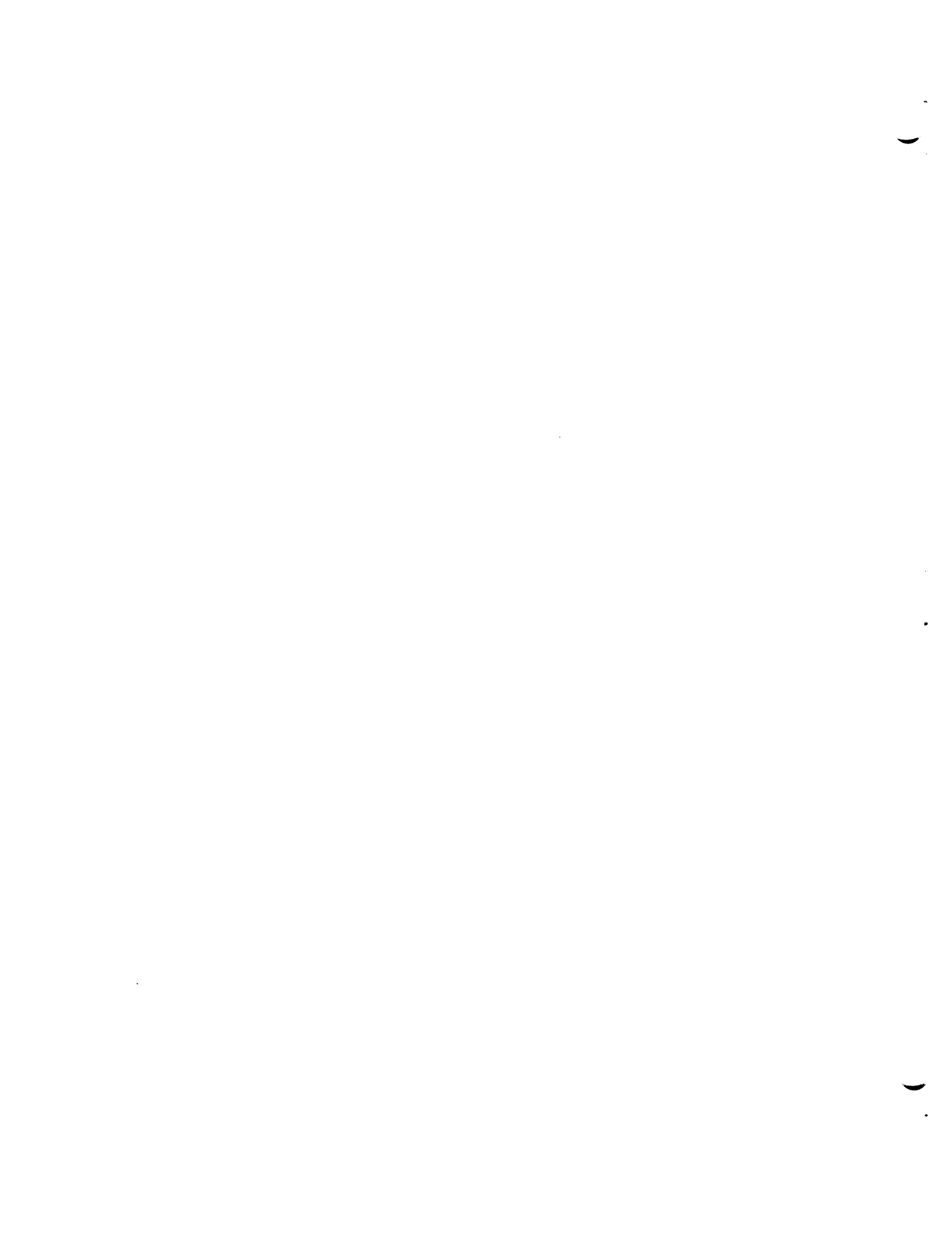
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Judy A. Fredricksen,* L. O. Gilpatrick,
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ABSTRACT

The solubility of CeF₃ was determined at various temperatures in six mixtures of LiF, BeF₂, ThF₄ of the type that may be used to fuel a molten salt breeder reactor. Comparison of earlier data on the solubility of PuF₃ and CeF₃ in fluoride solvents makes it possible to predict that the solubility of PuF₃ in single-region fuel compositions at reactor operating temperatures will be more than adequate. The solubility data as a function of solvent composition were best correlated by a model that assumes BeF₂ to be complexed as the BeF₄²⁻ ion and ThF₄ as the ThF₅¹⁻ ion.

INTRODUCTION

Studies performed earlier at the Oak Ridge National Laboratory demonstrated the solubility of PuF₃ in certain molten fluoride solvents,¹ but no data are available on the solubility of PuF₃ in single-fluid reactor fuel containing a high concentration of ThF₄. Previous data indicated that the solubility would probably be adequate at proposed reactor

* Summer ORAU Participant from St. Cloud State College, St. Cloud, Minnesota.

operating temperatures. To verify this belief, the most obvious approach would be direct measurement of PuF_3 solubility in molten fluoride salts of interest, but we have chosen to study the solubility of another trivalent fluoride, CeF_3 , because of its similar behavior and simpler handling. There is evidence indicating that PuF_3 and CeF_3 are quite similar in their solubility behavior in fluoride melts.^{2,3} A thorough study of the effect on the solubility of CeF_3 of varying the concentrations of LiF , BeF_2 and ThF_4 in melts will indicate the probable solubility of PuF_3 in such melts. This investigation may be followed by limited determinations of PuF_3 solubility to confirm predictions based on the CeF_3 investigation. The chemical feasibility of fueling molten salt reactors with PuF_3 has been considered in another report.⁴

This report gives the results obtained to date in an investigation on the solubility of CeF_3 in mixed LiF , BeF_2 and ThF_4 molten fluoride solvents.

EQUIPMENT

The reaction vessel in which the solubility measurements were made was a welded cylindrical nickel container with an internal diameter of $1\frac{1}{2}$ in., a depth of $6\frac{1}{2}$ in., and a wall thickness of $1/8$ in. A seven-inch section of $\frac{1}{2}$ -in. I.D. nickel pipe was welded to the lid and was closed at the top with a stainless steel ball valve having an internal clearance of $\frac{1}{2}$ in. This cooling and loading stem was also equipped with a $1/4$ -in. gas discharge port welded near its upper end. An

additional opening in the lid accommodated a thermocouple well of 1/4-in. thin wall (10 mil) nickel tubing closed at the lower end, which extended to within 1/8 in. of the bottom. The remaining lid opening held a 1/4-in. nickel dip leg of heavy wall (35 mil) nickel tubing extending to within 1/4 in. of the vessel bottom to facilitate agitation and purification of the melt by admitting gases beneath the melt surface.

The vessel was mounted vertically in a 3-in., 1400-watt electric tube furnace whose temperature was regulated by means of a chromel-alumel thermocouple placed between the furnace wall and the vessel and a high-sensitivity bucking circuit controller.

Melt temperatures were determined by a second chromel-alumel thermocouple located in the thermocouple well surrounded by the melt. An ice bath, a standard cell, and a Leeds and Northrup type K-2 potentiometer were used to measure the E.M.F. in this measuring circuit. Temperatures were deduced from standard tables of temperature versus E.M.F.

Copper filter sticks were constructed from 3/8-in. diameter sintered disk fritts 1/8-in. thick with a nominal pore size of 0.0004 in. These were welded into sections of 3/8-in. O.D. tubing 1/2-in. long. At the opposite end, this tube was reduced in diameter and welded to a 20-in. length of 1/8-in. O.D. tubing. These tubes were slipped through a Teflon gland made to fit a 1/2-in. Swagelok tubing connector and compressed by a standard connector collar. This gland formed a vacuum tight

seal around the 1/8-in. filter tube but it allowed movement of the filter through the gland which was fitted to the top end of the ball valve. Sufficient space was provided between the closed ball of the valve and the gland to accommodate the filter unit for evacuation and flushing with helium prior to admitting it to the clean (oxide free) melt. A piece of soft rubber tubing which fitted over the free end of the filter stick could be connected to either a vacuum pump or a helium source. A manifold system consisting of valves, pressure gages, and flow meters controlled admission of helium, hydrogen, HF or application of vacuum to the apparatus.

Samples containing ^{144}Ce tracer were analyzed using a 256-channel or 400-channel analyzer at fixed geometry with a 3 x 3 in. NaI, thallium activated crystal. Sample preparation and weighing was done in a hood equipped with a dust containment glove box.

MATERIALS

Some of the compositions used in these studies were supplied by J. H. Shaffer and F. A. Doss of ORNL. Mixtures of LiF, BeF_2 , and ThF_4 (72-16-12 and 68-20-12 mole %) were used as received as was a mixture of LiF and ThF_4 (73-27 mole %). The (72.7-4.8-22.5) mixture was prepared by adding 53 g of (72-16-12) to 198 g of the 73-27 residue left in the vessel after previous solubility determinations. Likewise a mixture calculated to have the composition (72.3-11.0-16.7) was prepared by adding 195.5 g of (72-16-12) to 225.1 g of (73.1-4.8-22.4) remaining

at the end of a series of measurements. A mixture having the composition (67.8-25.2-7.0) was prepared by mixing 94.7 g of 66 LiF - 34 BeF₂ with 102.1 g of the 73-27 preparation.

Ten millicuries of ¹⁴⁴Ce in the form of an aqueous HCl solution was secured from the Isotopes Division at ORNL. This was mixed with a solution containing 308 g of CeCl₃ x H₂O.* The resulting solution was heated to 90°C and digested with 214 g of NH₄F·HF** dissolved in 1000 ml of H₂O, which yielded a homogeneous precipitate of CeF₃ containing the radioisotope. This precipitate was washed with distilled water four times and centrifuged before drying at 110°C for 24 hours.

Commercial hydrogen was purified by passage through a Deoxo unit, a magnesium perchlorate drying tube, and a liquid N₂ trap. Anhydrous HF (99.9%), was used from the cylinder as received without purification. Commercial helium was purified by passage through an Ascarite trap, a magnesium perchlorate trap, and a charcoal trap at liquid N₂ temperature.

PROCEDURE

Helium leak testing was done at room temperature prior to loading the unit until a vacuum of at least 74 microns was secured. A weighed amount of fuel salt, usually about 250 grams, and more than the amount of CeF₃ expected to dissolve at the maximum temperature were added to the vessel through

* A. D. MacKay, Inc., C.P. grade.

** Baker and Adamson, technical grade.

the open ball valve by means of a long-necked funnel. The apparatus was then connected to the manifold system. A heated sodium fluoride trap was placed at the outlet to prevent HF from escaping into the hood. This was followed by a bubbler to indicate when gas was flowing through the system.

Purification was carried out at about 625°C by treating the melt with gaseous HF (20 ml/min), H₂ (100 ml/min), and helium (100 ml/min) for at least three hours. Hydrogen fluoride eliminated any products of hydrolysis resulting from adsorbed water on the surface of the fuel salt by converting them to fluorides. The hydrogen helped to minimize the corrosiveness of the HF by reducing any NiF₂ produced to Ni while the helium served as a carrier gas. Next, the melt was subjected to two hours of hydrogen (100 ml/min) -- helium (100 ml/min) treatment to complete the reduction of any NiF₂ formed.

Hydrogen and helium flow rates were measured by rotameters calibrated with a "Bubble-O-Meter." The hydrogen fluoride flow rate was measured by passing the gas mixture through a measured volume of 0.1 M KOH solution using phenolphthalein as an indicator and a stop watch to determine the time necessary for neutralization.

The mixture in the apparatus was allowed to equilibrate for one hour starting at the highest sampling temperature while agitation was maintained by a slow helium flow of 30 ml per minute. Each melt, with an excess of CeF₃, formed a

saturated solution at the selected temperature which was then sampled to determine the concentration of CeF_3 in the filtered melt. Sampling was performed by assembling the filter stick, after polishing with steel wool to remove the oxide coating, and the gland above the closed ball valve. This area was sealed by tightening the threaded collar around the Teflon gland, and a vacuum was applied followed by flushing with helium to remove air. This flush was repeated before the ball valve was opened and the filter was inserted to within 1/2-in. of the vessel bottom. A small flow of helium was maintained through the filter stick while it was being inserted and submerged. Five or ten minutes was allowed for the filter to reach the melt temperature before the helium flow was stopped and a vacuum was applied to the stem. The salt froze in the 1/8-in. diameter cold stem of the filter stick. Samples were withdrawn slowly to protect the Teflon gland from over heating. After closing the ball valve, the filter stick was removed by disassembling the gland and compression collar. It was then cut open and emptied in the glove box where the melt samples were ground and 50 mg \pm 3 mg was weighed from each filter for counting and one gram for wet chemical analysis.

The 50 mg portions of ground salt were weighed and placed in plastic vials 1-in. in diameter and 2-in. high. Six samples were drawn at 40°C intervals ending at about 20°C above the melting point of the salt compositions. Seven channels centering around the most energetic γ disintegration at

0.124 M.E.V. were integrated during the counting which was done mostly for one minute intervals.

Analyzing the samples radiochemically involved preparing standard samples made for each molten salt mixture consisting of 50 mg of solvent ($\pm 5\%$) plus varying amounts of accurately weighed CeF_3 tracer salt. A blank was also prepared which contained only the solvent which was used to determine the value to be subtracted from the total count to correct for the gamma activity of thorium daughter products. The net counting rate obtained for each standard was plotted on a linear scale against milligrams of labeled cerium fluoride. These calibration data made it possible to determine the number of milligrams of CeF_3 present in each sample from the count rate. From these values we calculated the mole percent of cerium fluoride present in each molten salt mixture at the various temperatures. No adjustment was made for radioactive decay since the half life of ^{144}Ce is 285 days and all counting for a given set of samples, including calibrations, was performed in sequence on a single day.

RESULTS AND DISCUSSION

The data obtained are plotted in Figs. 1-6, inclusive. Both radiochemical and wet chemical analysis are shown in these plots. In general, the agreement between the two methods of analysis is considered quite satisfactory. In some cases, comparison of the data required rechecking the wet chemical analyses and, in others, re-examination of the radiochemical

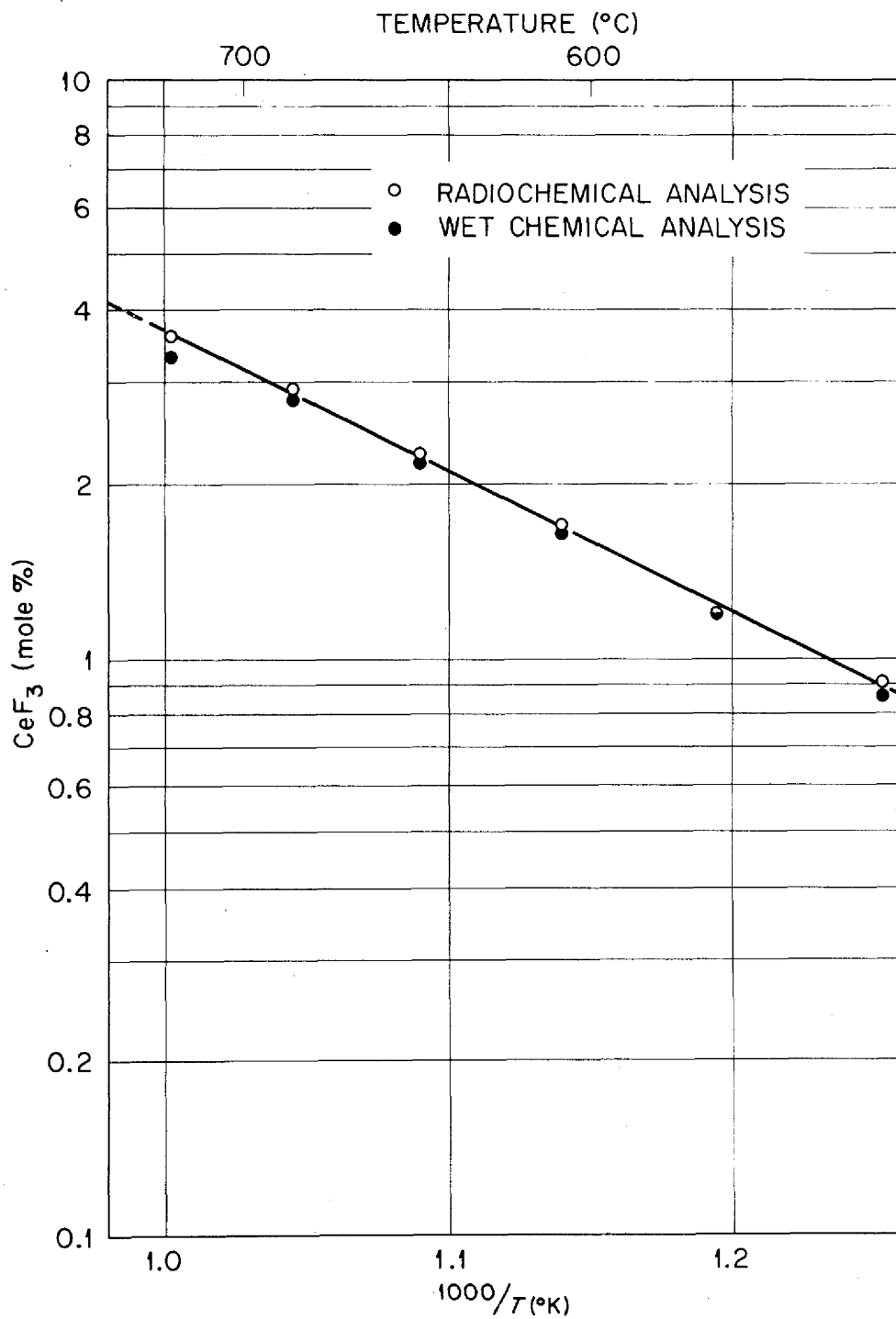


Fig. 1. Solubility of CeF₃ in LiF-BeF₂-ThF₄ (72-16-12 mole %).

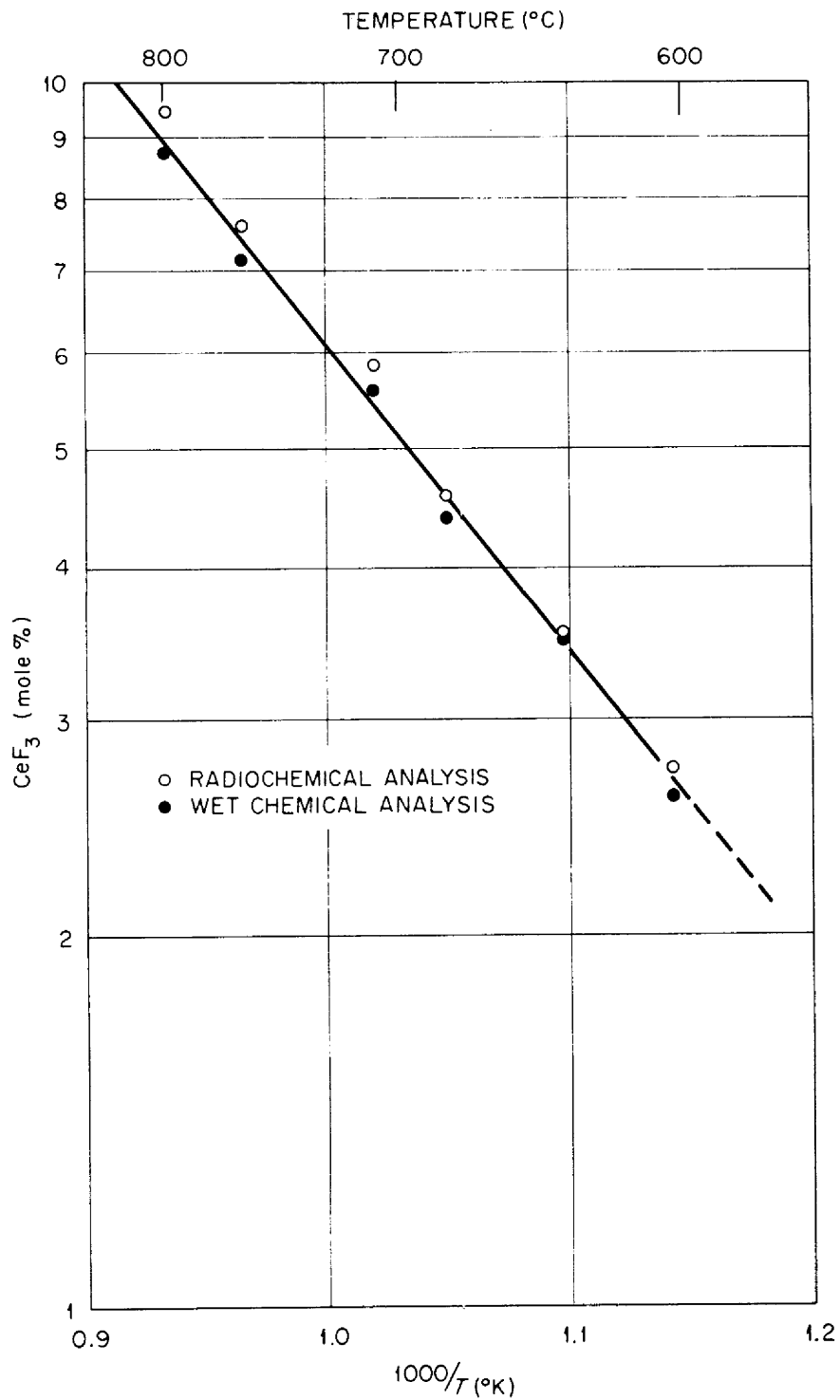


Fig. 2. Solubility of CeF₃ in LiF-ThF₄ (73-27 mole %).

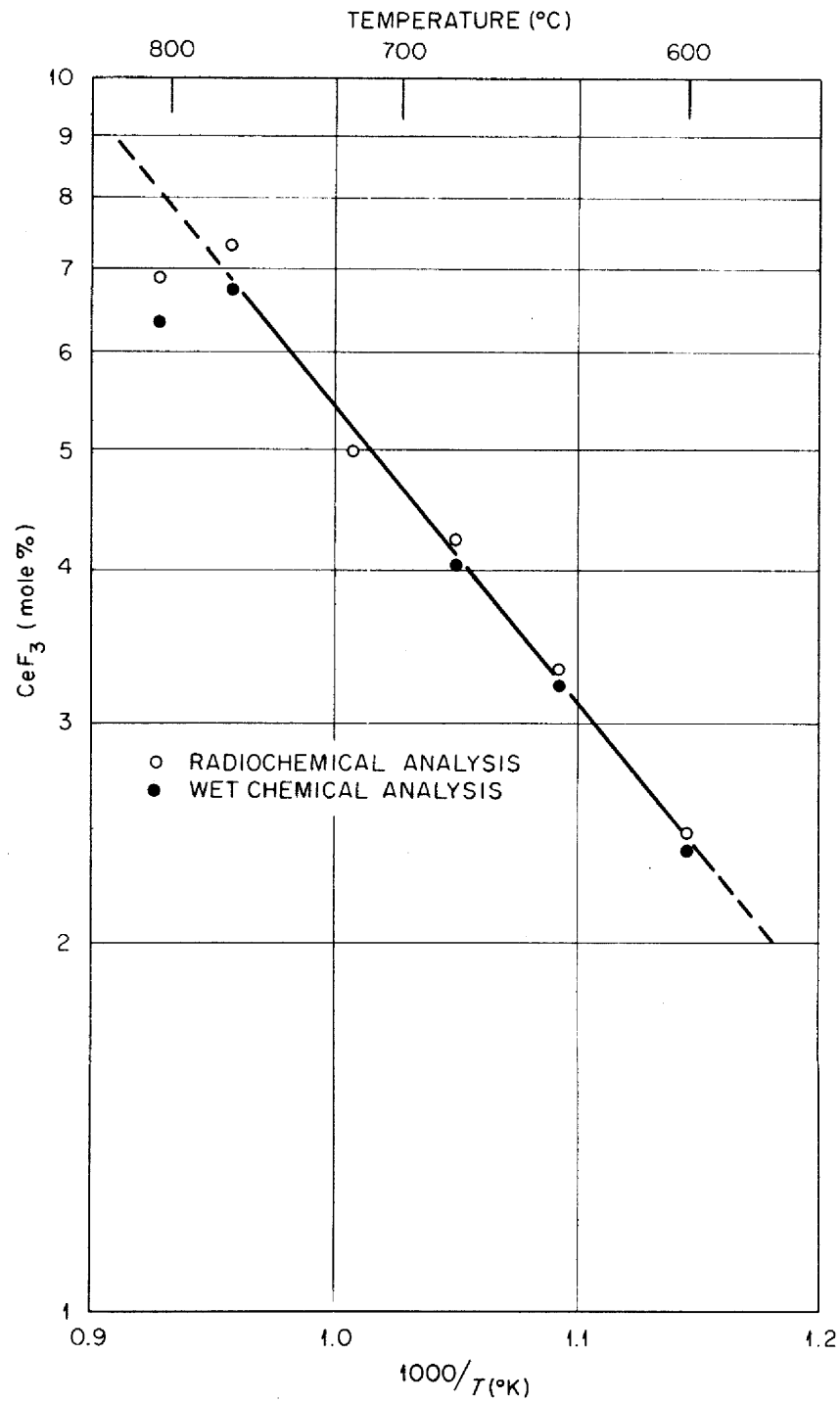


Fig. 3. Solubility of CeF₃ in LiF-BeF₂-ThF₄ (72.7-4.8-22.5 mole %).

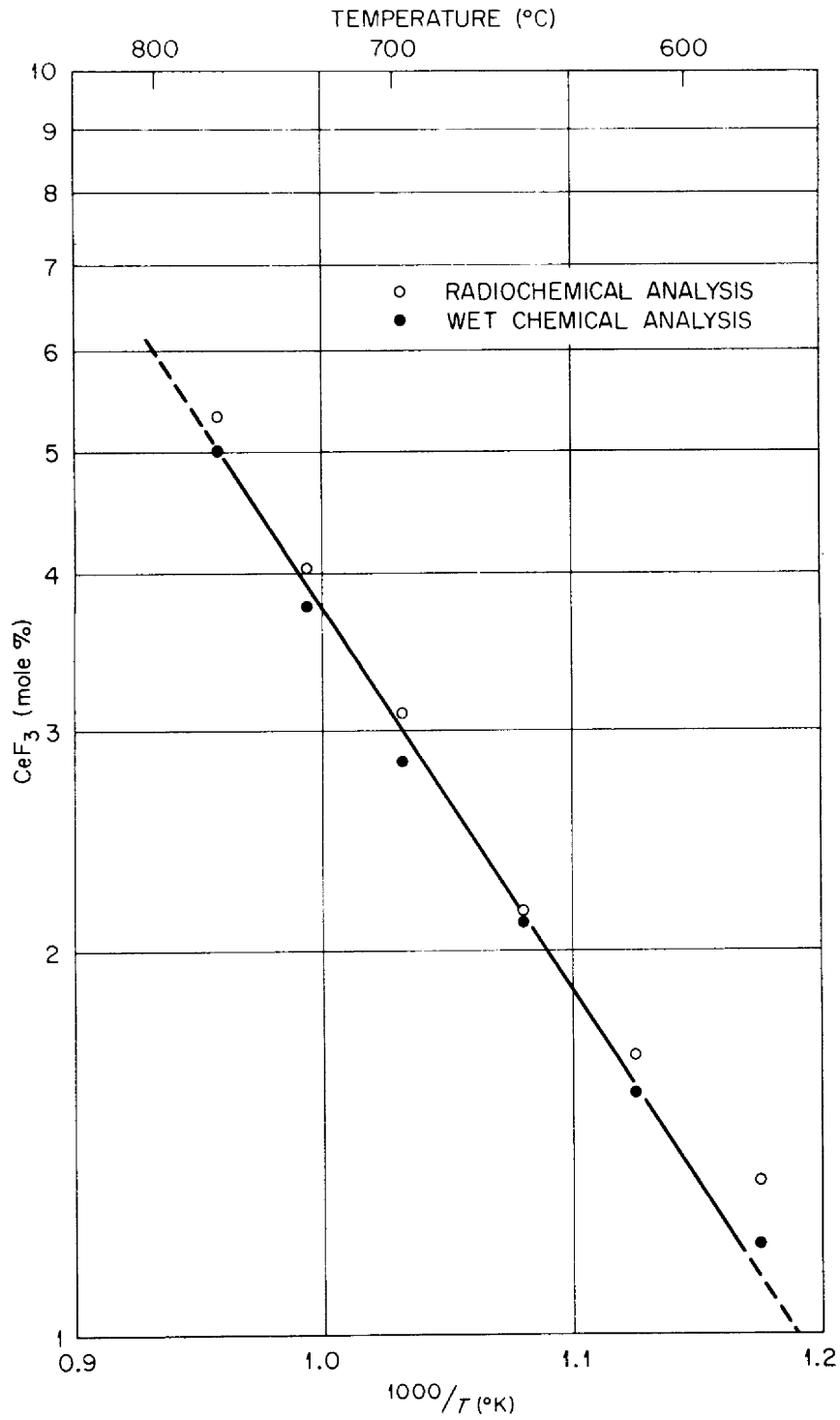


Fig. 4. Solubility of CeF₃ in LiF-BeF₂-ThF₄ (68-20-12 mole %).

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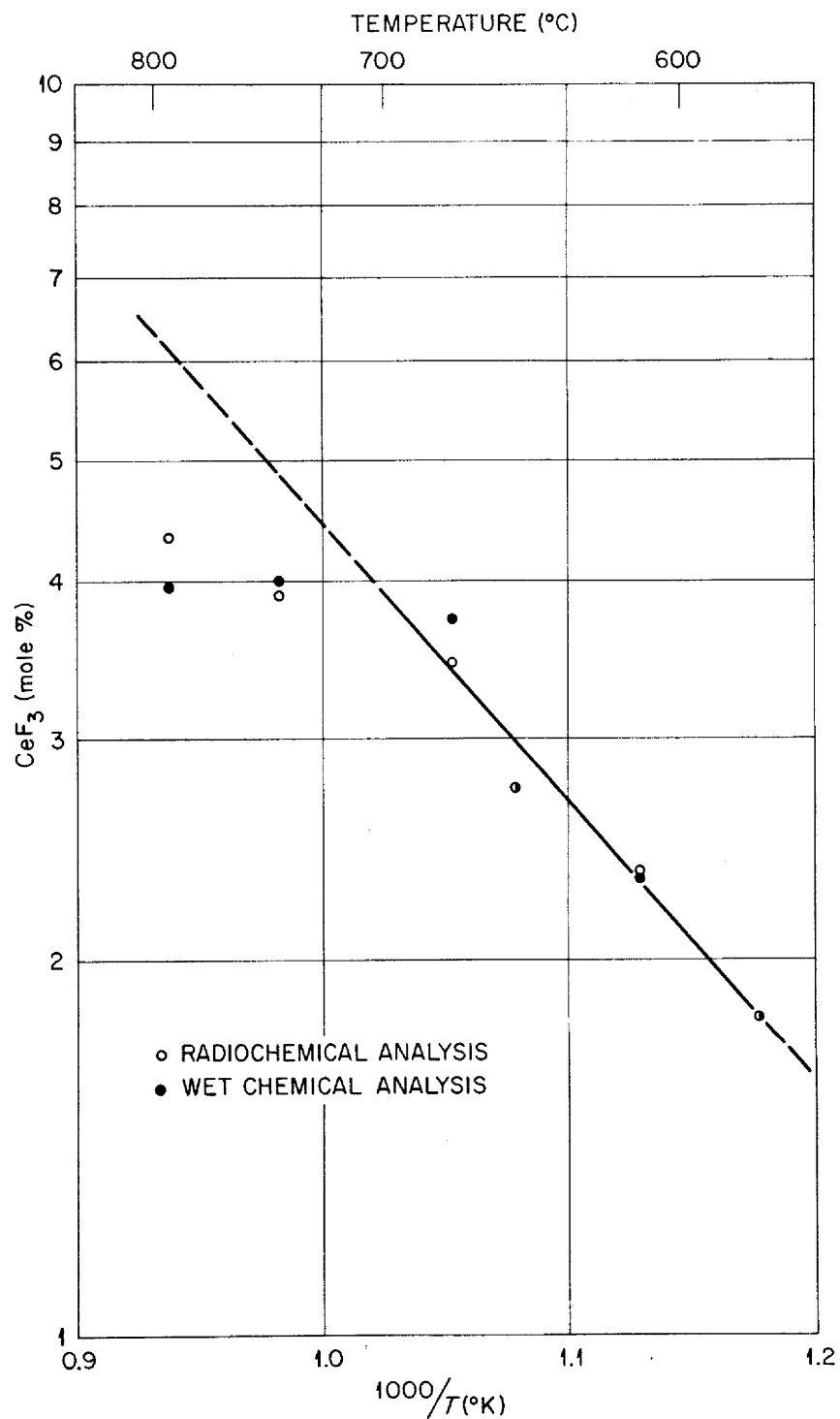


Fig. 5. Solubility of CeF₃ in LiF-BeF₂-ThF₄ (72.3-11.0-16.7 mole %).

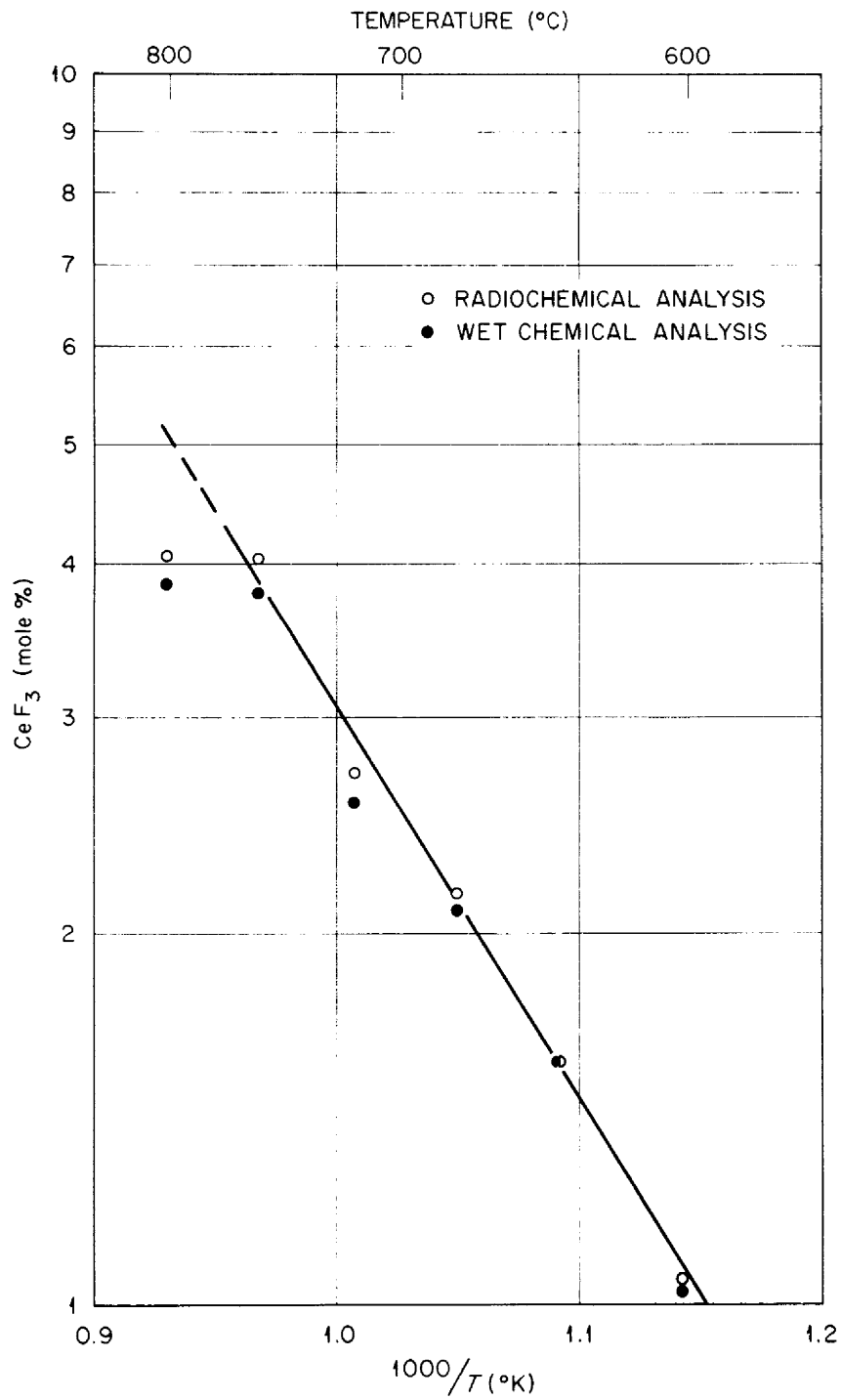


Fig. 6. Solubility of CeF₃ in LiF-BeF₂-ThF₄ (67.8-25.2-7.0 mole %).

calibration values. It is apparent in Figs. 3, 5, and 6 that insufficient CeF_3 was present in the system to saturate the melts at the highest temperatures. The data are summarized in Table 1 together with heats of solution calculated from the relation

$$\log \frac{S_2}{S_1} = \frac{\Delta H(T_2 - T_1)}{2.303 RT_1 T_2}$$

where S_2 is the solubility (in mole %) at the higher temperature and S_1 is the corresponding value at the lower temperature. The heats of solution are in approximately the same range as those reported¹ for solutions of PuF_3 in various fluoride solvents (12,000 to 16,800 cal per mole).

The solubility data obtained in this investigation are very reassuring in respect to the potential use of PuF_3 as the fissionable species in single-region fuel compositions. The lowest solubility observed at 600°C was 1.05 mole %. A comparison of PuF_3 solubility data¹ with similar values reported² for CeF_3 in Fig. 7 indicates that the solubility of PuF_3 at 600° may be less than 1.0 mole % but will almost certainly exceed the few tenths mole % value required to fuel a single region breeder reactor.

Bredig has suggested⁵ that the "free fluoride" content of liquid mixtures of LiF , BeF_2 , and ThF_4 can be calculated from the following relation in terms of mole %:

$$\text{Free fluoride} = \text{LiF} - 2(\text{BeF}_2) - 3(\text{ThF}_4)$$

This relation is based on the assumption that LiF is complexed

Table 1. Solubility and Heat of Solution of CeF_3
in Mixtures of LiF , BeF_2 , and ThF_4

Salt Composition (mole %)			CeF ₃ Solubility (mole %)		Heat of Solution (cal per mole)
<u>LiF</u>	<u>BeF₂</u>	<u>ThF₄</u>	<u>600°C</u>	<u>800°C</u>	
72	16	12	1.6	5.5	11,500
73	0	27	2.6	9.0	11,560
72.7	4.8	22.5	2.4	7.9	11,100
68	20	12	1.35	6.0	13,890
72.3	11.0	16.7	2.1	6.3	10,230
67.8	25.2	7.0	1.05	5.0	14,530

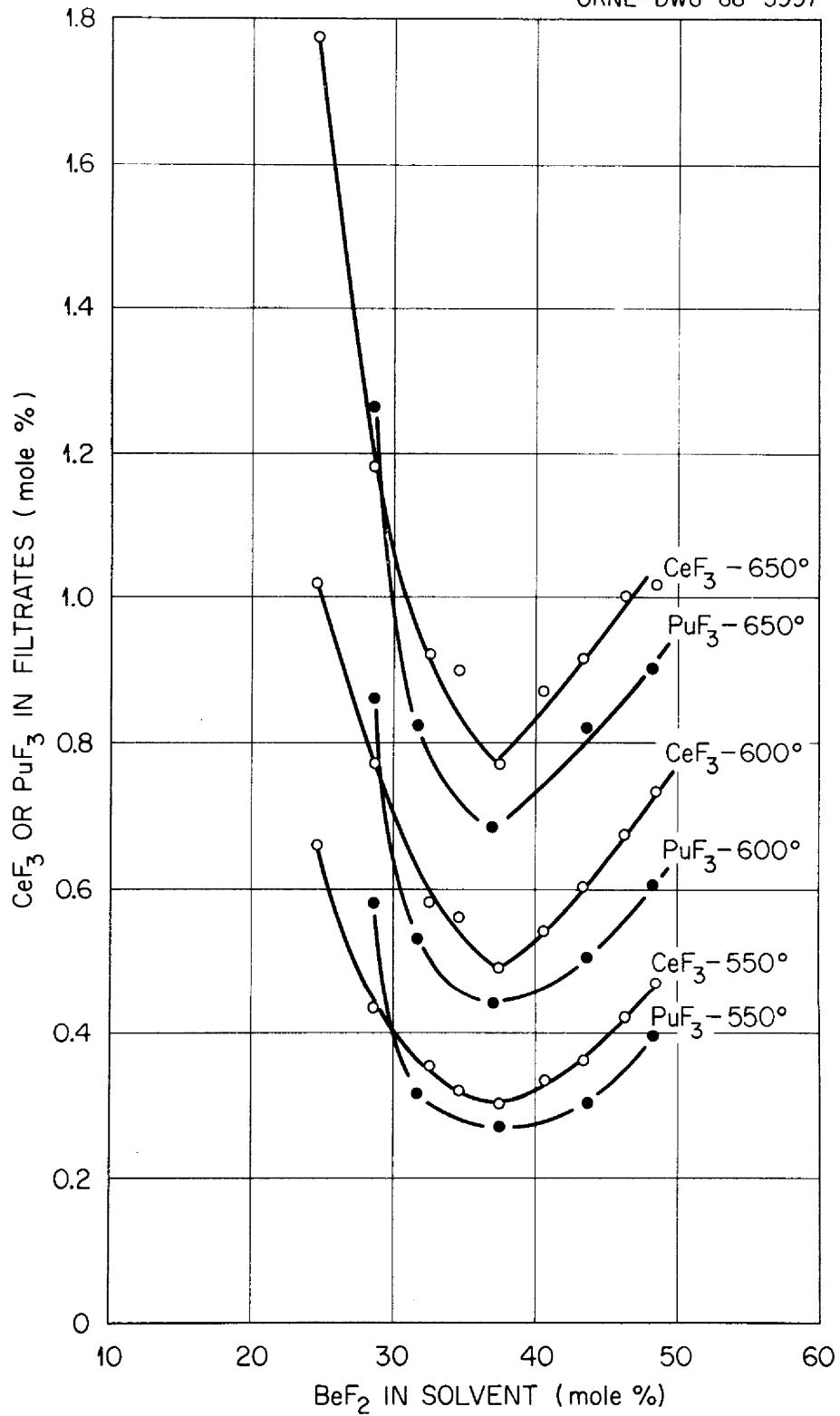


Fig. 7. Comparison of CeF₃ and PuF₃ Solubility in LiF-BeF₂ Solvents.

as Li_2BeF_4 and Li_3ThF_7 in the liquid state. It is interesting to test this concept with the CeF_3 solubility data reported here. The resulting graph (Fig. 8) shows a rather poor correlation based on this relationship. A somewhat better correlation, shown in Fig. 9, results from the assumption that BeF_2 is complexed in the liquid state as Li_2BeF_4 and ThF_4 as LiThF_5 . The latter assumption has a rather shaky basis since the published phase diagram⁶ for the system LiF-ThF_4 indicates that the 1:1 compound (as it is now known to be⁷) melts incongruently. The only defense for this assumption is that five of the six compositions tested to date show solubility data that correlate on this basis.

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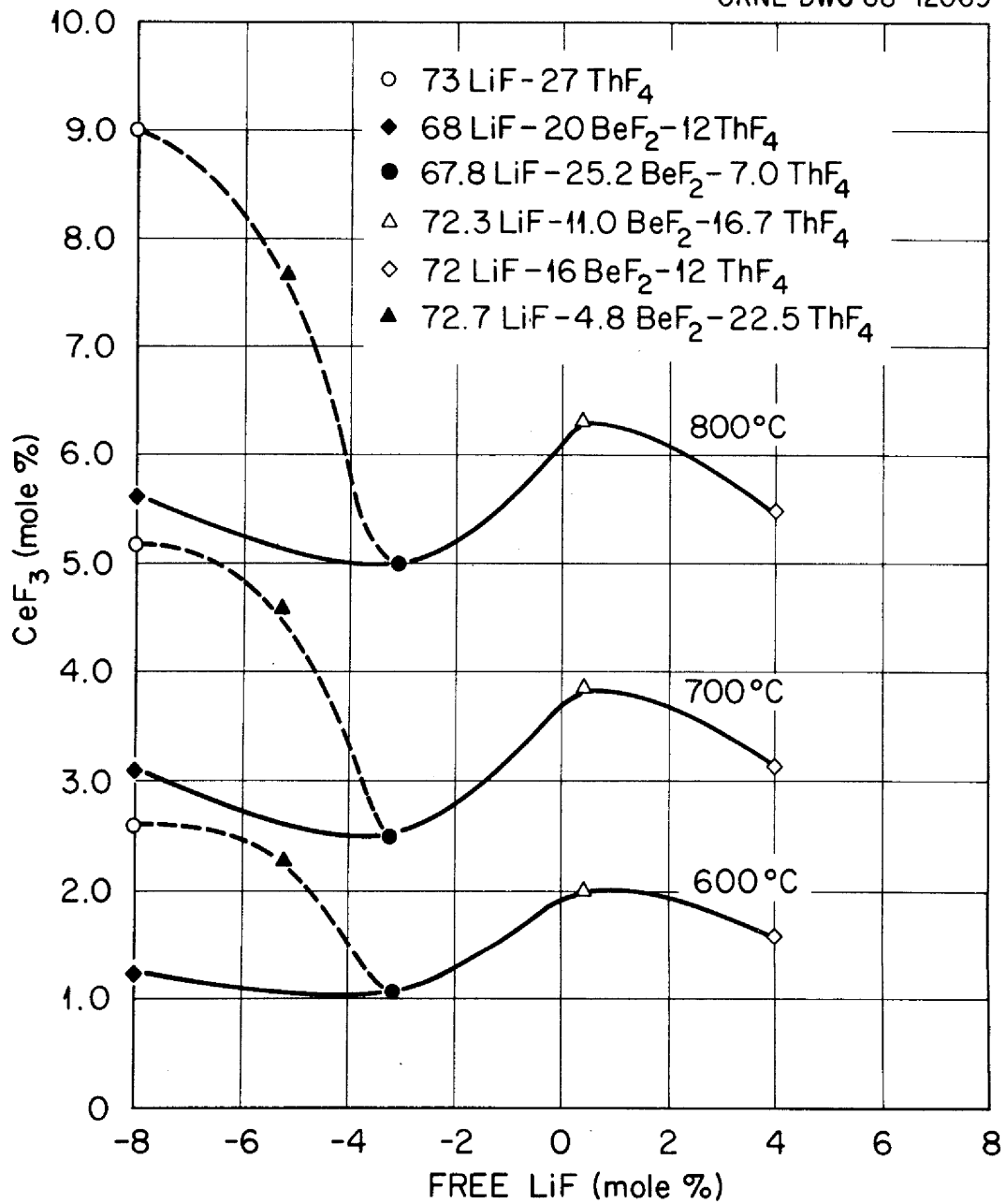


Fig. 8. Solubility of CeF_3 As A Function of Free LiF Assuming Li_2BeF_4 and Li_3ThF_7 In Liquid.

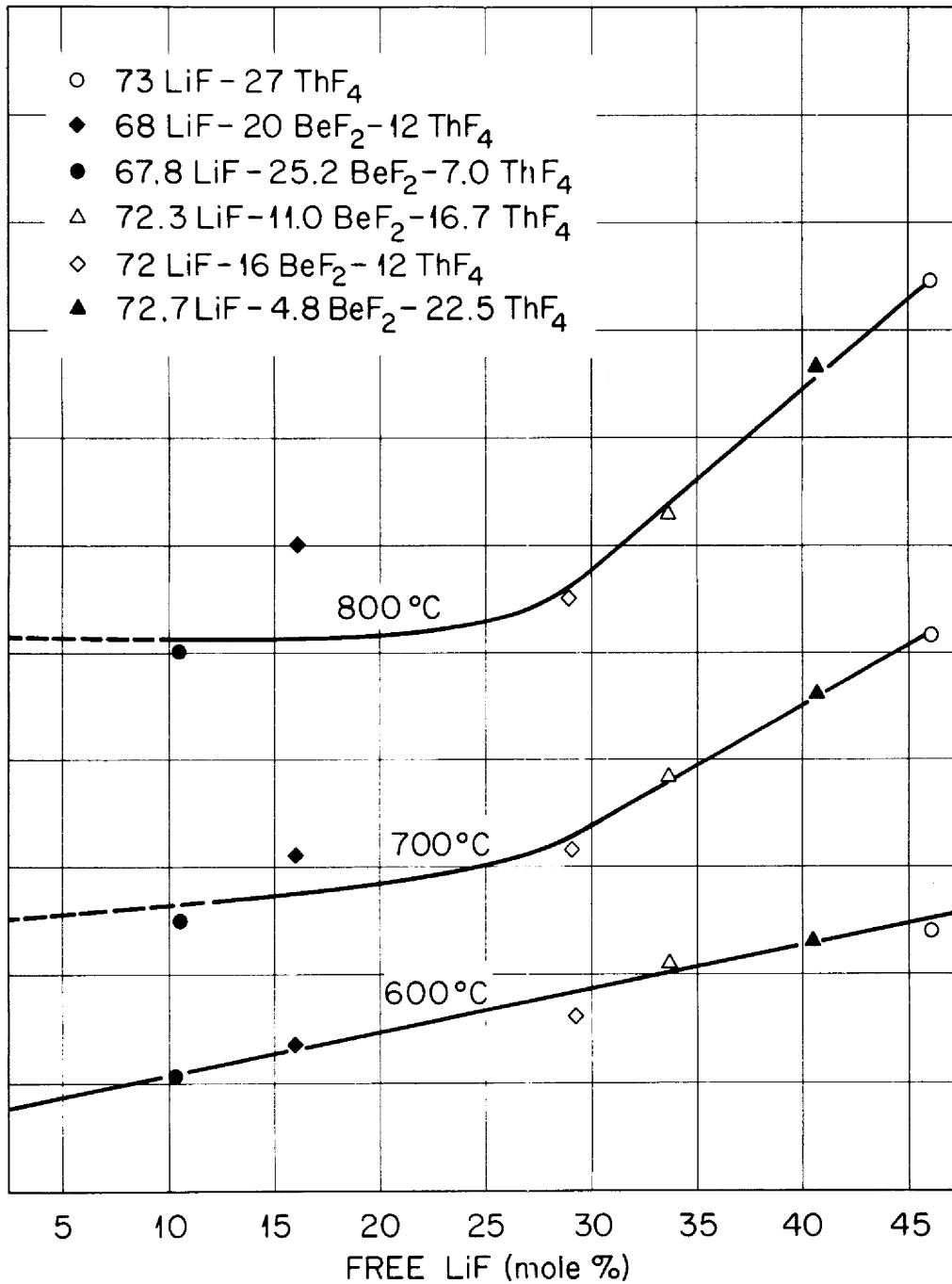


Fig. 9. Solubility of CeF₃ As A Function of Free LiF Assuming Li₂BeF₄ and LiThF₅ in Liquid.

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