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Thermal Dissociation of Molten Complex Compounds Me_3XF_7 (Me = Li, Na, K; X = Ti, Zr, Hf)

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The degree of the thermal dissociation on melting of complex compounds Me_3XF_7 , where Me = Li, Na, K and X = Ti, Zr, Hf, has been calculated on the basis of the known phase diagrams of the systems $\text{MeF}-\text{Me}_3\text{XF}_7$. It was found that the thermal stability of the investigated complex compounds increases with the increasing size of the central transition metal as well as with decreasing polarizability of the present alkali metal cation. The substitution of the fluorine atom in the coordination sphere of the transition metal by chlorine one leads to the decrease of the thermal stability of the compound.

Besides, the enthalpies of fusion of the investigated complex compounds were calculated on the basis of the cryoscopic measurements in independent systems.

In the binary systems of alkali metal fluorides and other salts of alkali metals, such as sulfates, chromates, molybdates, tungstates, transition metal fluorides, etc., complex compounds like Na_3FSO_4 , K_3FMoO_4 , K_3TiF_7 , K_3ZrF_7 , etc. are formed. These compounds exhibit a lowered symmetry of the coordination sphere of the central atom of the complex anion. Owing to this fact and obviously a relatively high energetic state such compounds undergo often at melting a more or

less extended thermal dissociation, in some cases they melt even incongruently. Evidences of such behaviour may be found e.g. in [1, 2].

Into this group belong also Me_3XF_7 compounds, where Me represents an alkali metal and X represents titanium, zirconium, and hafnium. The thermal stability of the complex XF_7^{3-} anion depends on the chemical nature of the central transition metal as well as of the present alkali metal. Besides F^- , in some cases also the ion Cl^-

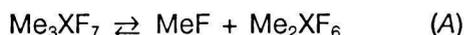
may be located as a ligand in the coordination sphere of the transition metal, forming an $\text{XF}_6\text{Cl}^{3-}$ complex anion.

On the basis of the experimentally determined phase diagrams and the density of the melts in the $\text{KF}-\text{K}_2\text{TiF}_6$ and $\text{KCl}-\text{K}_2\text{TiF}_6$ systems the degree of thermal dissociation of the anions TiF_7^{3-} and $\text{TiF}_6\text{Cl}^{3-}$ has been calculated in [3]. It was found that the degree of the thermal dissociation of K_3TiF_7 attains at the melting temperature the value $\alpha_0 = 0.64$, while the dissociation degree of $\text{K}_3\text{TiF}_6\text{Cl}$ at the melting temperature is $\alpha_0 = 0.78$. Approximately the same values were obtained also from the density measurements. From the temperature dependence of the dissociation degree obtained on the basis of the density measurements the values of the dissociation enthalpy $\Delta H_{\text{dis}}(\text{K}_3\text{TiF}_7) = 52.4 \text{ kJ mol}^{-1}$ and $\Delta H_{\text{dis}}(\text{K}_3\text{TiF}_6\text{Cl}) = 46.6 \text{ kJ mol}^{-1}$ were calculated, which represents a substantial part of the enthalpy of fusion ($\Delta H_f(\text{K}_3\text{TiF}_7) = 56.0 \text{ kJ mol}^{-1}$ and $\Delta H_f(\text{K}_3\text{TiF}_6\text{Cl}) = 52.9 \text{ kJ mol}^{-1}$).

In the present work the thermal dissociation of molten Me_3XF_7 complex compounds (Me = Li, Na, K; X = Zr, Hf) has been calculated on the basis of published phase diagrams of the $\text{MeF}-\text{Me}_3\text{XF}_7$ systems and compared with the results of the thermal dissociation calculations of the fluorotitanate compounds given in [3]. Besides, the enthalpies of fusion of the investigated complex compounds were estimated on the basis of the cryoscopic measurements in independent systems.

COMPUTATIONAL PROCEDURE

Let us consider an arbitrary mixture of $(1 - x)$ mol of the substance MeF and x mol of the substance Me_3XF_7 . In the case of the thermal dissociation of the substance Me_3XF_7 according to the scheme



with the degree of dissociation α , the resulting amounts of substance of components in the system can be expressed as

$$\begin{aligned} n(\text{MeF}) &= 1 - x(1 - \alpha) \text{ mol} \\ n(\text{Me}_2\text{XF}_6) &= \alpha x \text{ mol} \\ n(\text{Me}_3\text{XF}_7) &= x(1 - \alpha) \text{ mol} \end{aligned}$$

The total amount of substance of all components is $(1 + \alpha x)$ mol. Consequently, the equilibrium mole fractions of components are given by eqns

$$\begin{aligned} x(\text{MeF}) &= \frac{1 - x(1 - \alpha)}{1 + \alpha x}; & x(\text{Me}_2\text{XF}_6) &= \frac{\alpha x}{1 + \alpha x}; \\ x(\text{Me}_3\text{XF}_7) &= \frac{x(1 - \alpha)}{1 + \alpha x} \end{aligned} \quad (1)$$

Assuming the ideal behaviour of the solutions, the equilibrium constant of eqn (A) can be written as

$$K = \frac{\alpha_0^2}{1 - \alpha_0^2} = \frac{\alpha[1 - x(1 - \alpha)]}{(1 - \alpha)(1 + \alpha x)} \quad (2)$$

where α_0 is the dissociation degree of pure Me_3XF_7 . By means of eqn (2) it is possible to calculate the degree of thermal dissociation α of Me_3XF_7 for any arbitrary selected value of α_0 and, after inserting the calculated value α into eqns (1), to obtain the values of the equilibrium mole fractions of the individual components. In the first approximation it was assumed that in the given temperature range of the liquidus the equilibrium constant does not change with the temperature. Introducing the values of the equilibrium mole fractions of the individual components $x(i)$ into the simplified ($\Delta H_f(i) = \text{const.}$) and modified Le Chatelier—Shreder equation

$$T_{\text{pc}}(i) = \frac{\Delta H_f(i) T_f(i)}{\Delta H_f(i) - RT_f(i) \ln x(i)} \quad (3)$$

where $T_f(i)$ and $\Delta H_f(i)$ are the temperatures and enthalpies of fusion of the individual components, the values of the temperature of primary crystallization of components $T_{\text{pc}}(i)$ may be calculated, which made it possible to draw the hypothetical liquidus curves of the investigated systems corresponding to the appropriate value of the dissociation degree α_0 . The melting temperatures of the hypothetical undissociated compounds Me_3XF_7 for the given equilibrium composition were calculated from the experimentally determined melting temperatures of these compounds.

The criterion for the selection of the correct value of the dissociation degree α_0 is the best fit between the experimental and calculated liquidus temperatures according to the condition

$$\sum_{i=1}^n [T_{\text{pc}}(i, \text{exp.}) - T_{\text{pc}}(i, \text{calc.})]^2 = \min \quad (4)$$

In the calculation of the temperatures of primary crystallization of components, the data on the temperatures and enthalpies of fusion of the pure components are indispensable. The data for the alkali metal halides were taken from the JANAF

Table 1. Values of the Enthalpies of Fusion and Melting Temperatures of Components Employed in the Calculation

Component	$T_f(i)$	$\Delta H_f(i)$	Ref.
	K	kJ mol^{-1}	
LiF	1121	27.06	[4]
NaF	1269	33.36	[4]
KF	1131	27.20	[4]
KCl	1043	26.30	[4]
Li_3ZrF_7	935	30.00	This paper
Na_3ZrF_7	1123	50.50	This paper
K_3ZrF_7	1183	77.40	This paper
$\text{K}_3\text{ZrF}_6\text{Cl}$	1003	66.00	This paper
Na_3HfF_7	1133	54.00	This paper
K_3HfF_7	1196	95.70	This paper

Thermochemical Tables [4], however, the corresponding values for Me_3XF_7 have not been published thus far and had to be estimated. The procedure employed is presented in the Appendix. The corresponding values are summarized in Table 1.

The calculation was carried out on the basis of the published phase diagrams. The following systems were selected: The phase diagram of the system $\text{LiF}-\text{Li}_3\text{ZrF}_7$ measured by *Thoma et al.* [5] with the eutectic point at 871 K and 57 mole % Li_3ZrF_7 . The phase diagram of the system $\text{NaF}-\text{Na}_3\text{ZrF}_7$ published by *Barton et al.* [6]. The coordinates of the eutectic point are 1020 K and 50 mole % Na_3ZrF_7 . The phase diagram of the system $\text{NaF}-\text{Na}_3\text{HfF}_7$ measured by *Sheiko et al.* [7], the coordinates of the eutectic point being 53.1 mole % Na_3HfF_7 and 1035 K. The phase diagram of the system $\text{KF}-\text{K}_3\text{ZrF}_7$ studied by *Sheiko et al.* [8], the coordinates of the eutectic point being 1038 K and 24.1 mole % K_3ZrF_7 . The phase diagram of the system $\text{KF}-\text{K}_3\text{HfF}_7$ measured by *Sheiko et al.* [7]. The coordinates of the eutectic point are 1039 K and 21.2 mole % K_3HfF_7 . Besides, the degree of the thermal dissociation of a similar, congruently melting compound, $\text{K}_3\text{ZrF}_6\text{Cl}$, was studied. The phase diagram of the system $\text{KCl}-\text{K}_3\text{ZrF}_6\text{Cl}$ published by *Sheiko et al.* [8] with the eutectic point at 951 K and 30 mole % $\text{K}_3\text{ZrF}_6\text{Cl}$ was used for the calculation.

RESULTS AND DISCUSSION

Fig. 1 shows the phase diagram of the system $\text{LiF}-\text{Li}_3\text{ZrF}_7$ according to [5] and the liquidus curves calculated for the value of the equilibrium dissociation constant of the compound Li_3ZrF_7 ,

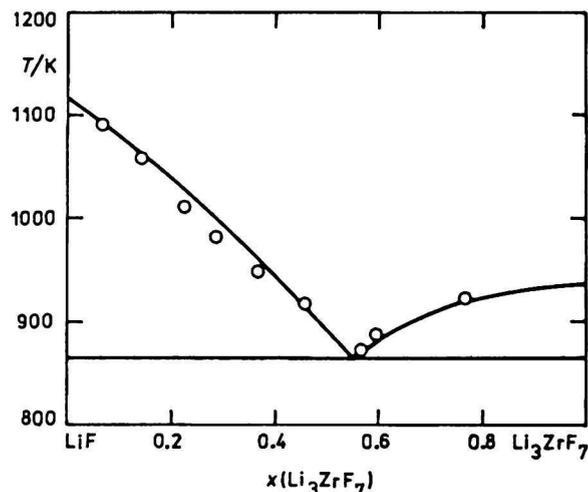


Fig. 1. Phase diagram of the system $\text{LiF}-\text{Li}_3\text{ZrF}_7$. \circ Experimental liquidus points according to [5]; — calculation.

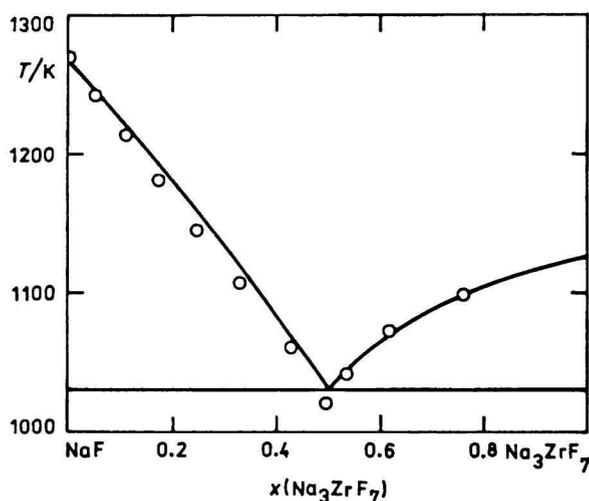


Fig. 2. Phase diagram of the system $\text{NaF}-\text{Na}_3\text{ZrF}_7$. \circ Experimental liquidus points according to [6]; — calculation.

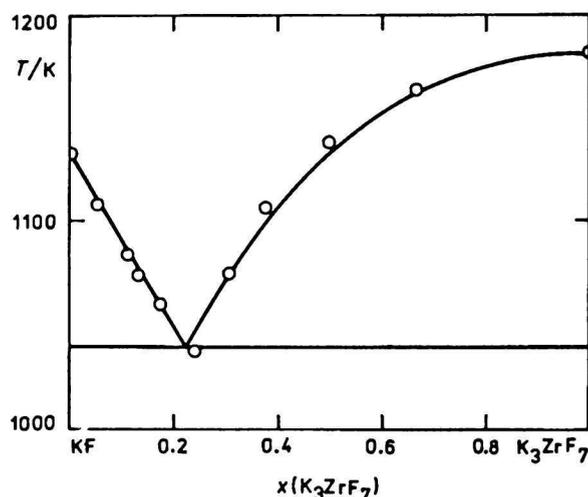


Fig. 3. Phase diagram of the system $\text{KF}-\text{K}_3\text{ZrF}_7$. \circ Experimental liquidus points according to [8]; — calculation.

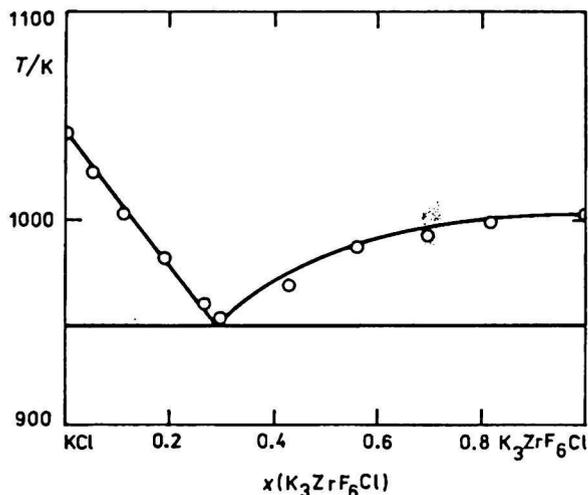


Fig. 4. Phase diagram of the system KCl— K_3ZrF_6Cl . ○ Experimental liquidus points according to [8]; — calculation.

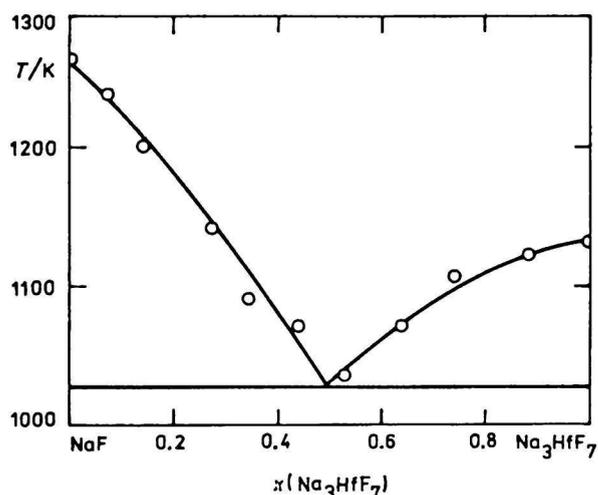


Fig. 5. Phase diagram of the system NaF— Na_3HfF_7 . ○ Experimental liquidus points according to [7]; — calculation.

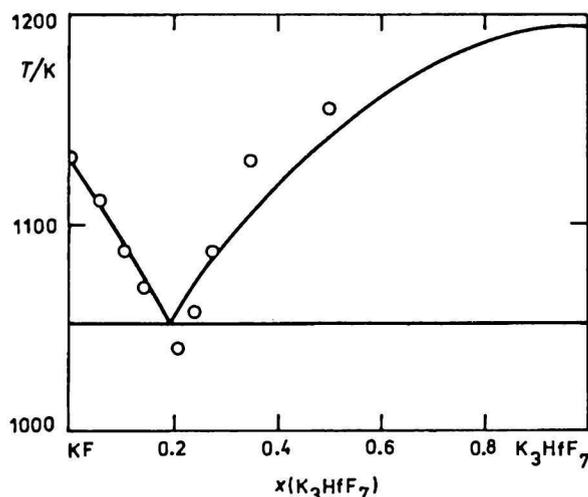


Fig. 6. Phase diagram of the system KF— K_3HfF_7 . ○ Experimental liquidus points according to [7]; — calculation.

$K_{dis}(Li_3ZrF_7) = 0.042$, the corresponding values of the dissociation degree and the hypothetical melting temperature of the undissociated compound Li_3ZrF_7 being $\alpha_0 = 0.2$ and $T_{f,nd}(Li_3ZrF_7) = 1045$ K. The standard deviation of the calculated values of the temperatures of primary crystallization from the experimentally determined values is 10.2 K, which is mainly due to the relatively low accuracy of the experimental data available.

Fig. 2 shows the phase diagram of the system NaF— Na_3ZrF_7 according to [6]. The best fit between the experimental and calculated liquidus curves has been achieved for the value of the equilibrium dissociation constant $K_{dis}(Na_3ZrF_7) = 0.015$ with the corresponding value of the dissociation degree $\alpha_0 = 0.12$ and the hypothetical melting temperature of undissociated additive compound $T_{f,nd}(Na_3ZrF_7) = 1175$ K. The standard deviation of the experimental and calculated values of the temperature of primary crystallization was determined to be 8.4 K.

The phase diagram of the system KF— K_3ZrF_7 according to [8] is shown in Fig. 3. The calculated liquidus curves were obtained using the value of the dissociation constant $K_{dis}(K_3ZrF_7) = 0.067$. The corresponding value of the dissociation degree is $\alpha_0 = 0.25$ and of the temperature of fusion of undissociated K_3ZrF_7 $T_{f,nd}(K_3ZrF_7) = 1265$ K. The standard deviation of the calculated and experimental liquidus temperatures is 4.5 K, which refers to a very good fit.

The phase diagram of the system KCl— K_3ZrF_6Cl according to [8] is shown in Fig. 4. The best fit of the liquidus curves was obtained by means of the following values: $K_{dis}(K_3ZrF_6Cl) = 0.563$, $\alpha_0 = 0.68$, $T_{f,nd}(K_3ZrF_6Cl) = 1269$ K with the standard deviation of the experimental and calculated temperatures of primary crystallization of 3.7 K. Again a very good agreement was achieved.

Fig. 5 shows the phase diagram of the system NaF— Na_3HfF_7 according to [7] and the liquidus curves calculated for the value of the dissociation constant $K_{dis}(Na_3HfF_7) = 0.005$. The corresponding dissociation degree is $\alpha_0 = 0.07$ and the temperature of fusion of undissociated compound $T_{f,nd}(Na_3HfF_7) = 1161$ K. The standard deviation of the experimental and calculated temperatures of primary crystallization is 8.9 K, which is obviously due to the relatively high dispersion of the experimental data.

The phase diagram of the last investigated system KF— K_3HfF_7 according to [7] is shown in Fig. 6 and compared with the calculated liquidus curves using the value of the dissociation constant $K_{dis}(K_3HfF_7) = 0.030$. The corresponding dissociation degree is $\alpha_0 = 0.17$ and the hypothetical melting temperature of undissociated K_3HfF_7 is

$T_{f,nd}(K_3HfF_7) = 1240$ K. The value of the standard deviation of the experimental and calculated points, 11.8 K, is caused by evidently uncommon shape of the K_3HfF_7 liquidus curve, namely its sheer course in the region of high concentration of KF.

In general a good agreement of the experimental and calculated liquidus curves in all systems studied was obtained (Table 2). In some cases a lowered accuracy of the experimental data decreases the information testimony of the calculation, however, in spite of it some general conclusions may be drawn.

First of all, the correctness of the thermodynamic approach employed was confirmed. It does not introduce any sophisticated procedures for expressing the activity of components. The only simplification used is the assumption of the ideal mixing of components. The fulfilment of the limiting laws for $x(i) \rightarrow 1$ also demonstrates the plausibility of the calculated equilibrium composition as well as the thermodynamic consistency of the experimental phase diagrams. The introduction of the hypothetical melting temperature of undissociated additive compound does not contradict the thermodynamic principles.

From the calculated values of the dissociation degrees it follows that the thermal stability of the additive compounds depends on the chemical nature of both the central transition metal of the complex anion and the present alkali metal cation. From the comparison of the dissociation degrees of compounds with the same alkali metal cation, e.g. potassium, it is evident that the thermal stability increases with the increasing size of the transition metal, obviously due to the steric and consequently the energetic relations. The thermal stability of the complex anion is influenced also by the size of the present ligands. The substitution of the fluorine atom in the coordination sphere of the transition metal by the chlorine one leads to the decrease of the thermal stability of the compound.

The influence of the present alkali metal cation on the thermal stability of the Me_3XF_7 compounds follows the general trend of the alkali metal compounds, concerning e.g. their melting temperatures and other properties. With the exception of the lithium compound the thermal stability decreases with the increasing electronegativity of the alkali metal obviously due to the lowering of the electrostatic forces between the complex anion and the alkali metal cation.

The thermal stability of the investigated compounds is closely related to the symmetry of the coordination sphere of the central atom of the complex anion. From the proposed crystal struc-

Table 2. Calculated Values of the Equilibrium Constants, the Dissociation Degrees of Investigated Compounds, the Standard Deviations of the Experimental Liquidus Temperatures from the Calculated Ones, and the Hypothetical Temperatures of Fusion of the Undissociated Compounds

Compound	K	α_0	s/K	$T_{f,nd}/K$
$K_3TiF_7^a$	0.694	0.64	8.0	1382
$K_3TiF_6Cl^a$	1.554	0.78	6.5	1454
Li_3ZrF_7	0.042	0.20	10.2	1045
Na_3ZrF_7	0.015	0.12	8.4	1175
K_3ZrF_7	0.067	0.25	4.5	1265
K_3ZrF_6Cl	0.563	0.68	3.7	1269
Na_3HfF_7	0.005	0.07	8.9	1161
K_3HfF_7	0.030	0.17	11.8	1240

a) According to Ref. [3].

ture of the Me_3XF_7 compounds [9–11] it follows that the central X atom is coordinated by eight fluorine atoms forming a flat tetragonal prism. Such configuration is in accordance with the relatively high thermal stability of the heptafluorozirconates and heptafluorohafnates. Unfortunately, the crystal structure of the low-stable potassium heptafluorotitanate and hexafluorochloro derivatives is not known. At least the latter ones have probably the lowered symmetry.

APPENDIX

Estimation of the Enthalpy of Fusion of Me_3XF_7 Compounds

No data on the enthalpy of fusion of the investigated Me_3XF_7 compounds were found in the literature. Therefore the enthalpies of fusion were estimated on the basis of an independent phase diagram. For this purpose simple eutectic systems without solid solutions were chosen in which the second component introduces only one foreign particle into Me_3XF_7 . In most cases chlorides of the same alkali metal cation were chosen.

For the systems of this type the limiting law holds

$$\lim_{x \rightarrow 1} \frac{dT}{dx} = \frac{RT_f^2(i)}{\Delta H_f(i)} \quad (5)$$

where x is the mole fraction of Me_3XF_7 , $\Delta H_f(i)$ and $T_f(i)$ are the enthalpy and the temperature of fusion of Me_3XF_7 . The dependence of the temperature of primary crystallization of Me_3XF_7 on composition, $T_{pc} = f(x)$, was calculated from the course of the liquidus curve, expressed in the form of a polynomial of the second degree. Derivating the

Table 3. Coefficients of the Equations $\{T_{pc}(Me_3XF_7)\} = a + bx + cx^2$ and the Standard Deviations of the Temperatures of Primary Crystallization from the Calculated Ones. Temperature is given in K

System	a	b	c	s/K
Na ₃ ZrF ₇ —NaCl	609.1	794.0	- 296.0	3.65
Na ₃ HfF ₇ —NaCl	513.9	1040.8	- 422.6	1.47
K ₃ ZrF ₇ —KCl	865.1	502.1	- 174.4	1.78
K ₃ HfF ₇ —NaF	989.2	287.3	- 81.5	1.18

polynomial with respect to x and introducing it into eqn (5) the value of the enthalpy of fusion of Me₃XF₇ may be calculated.

For the calculation of the enthalpy of fusion of Me₃XF₇ the following phase diagrams were chosen: Na₃ZrF₇—NaCl measured by *Sheiko et al.* [12], Na₃HfF₇—NaCl measured by *Maltsev et al.* [13], K₃ZrF₇—KCl measured by *Sheiko et al.* [12], and K₃HfF₇—NaF measured by *Maltsev et al.* [13]. In the last system NaF introduces into K₃HfF₇ only one foreign particle, Na⁺, since fluoride anions are already present in molten K₃HfF₇ due to its thermal dissociation. The calculated coefficients of the second-degree polynomial for eqn $T_{pc} = f(x)$ are given in Table 3.

For the calculation of the enthalpy of fusion of Li₃ZrF₇ and K₃ZrF₆Cl no suitable phase diagrams were found in the literature. Therefore the enthalpies of fusion of these compounds were estimated on the basis of a thermodynamic analogy. For Li₃ZrF₇ it was assumed that the difference in the entropy of fusion between Li₃ZrF₇ and Na₃ZrF₇ is approximately the same as between Na₃ZrF₇ and K₃ZrF₇. For K₃ZrF₆Cl it was assumed that the entropy of fusion may be calculated by

exchanging the entropy of fusion of KF by the KCl one

$$\Delta S_f(K_3ZrF_6Cl) = \Delta S_f(K_3ZrF_7) - \Delta S_f(KF) + \Delta S_f(KCl) \quad (6)$$

The estimated values of the enthalpies of fusion of the investigated compounds are summarized in Table 1.

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