DENSITY, THERMAL EXPANSION COEFFICIENT AND VISCOSITY OF SODIUM TETRABORATE (BORAX)-UO₂ AND OF SODIUM METABORATE-UO₂ SOLUTIONS AT HIGH TEMPERATURES

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Received 22 March 1983; accepted 19 May 1983

Measurements have been performed of the density, of the volumetric thermal expansion coefficient and of the viscosity of liquid sodium tetraborate (borax) and of sodium metaborate both pure and with two different amounts of UO₂ dissolved in each. The viscosity measurements have been performed for the solution of sodium tetraborate with UO₂ and CeO₂, and with CeO₂ only as well. These data are required for the design of core-catchers based on sodium borates. The density measurements have been performed with the buoyancy method in the temperature range from 825°C to 1300°C, the viscosity measurements in the temperature range 700–1250°C with a modified Haake viscosity balance. The balance was previously calibrated at ambient temperature with a standard calibration liquid and at high temperatures with data for pure borax available from the literature.

1. Introduction

Sodium borates appear to be quite suitable as sacrificial materials for a core-catcher of a nuclear reactor. Sodium tetraborate (borax) (Na₂O · 2B₂O₃ = Na₂B₄O₇) and sodium metaborate (Na₂O · B₂O₃ = 2NaBO₂) dissolve UO₂, PuO₂ and the fission product oxides, provided they remain in contact for a sufficiently long time at sufficiently high temperatures. The resulting power density in the solution due to the decay heat of the fission products is greatly reduced and the melt can be contained.

The so-called melting point of borax (740°C) and of sodium metaborate (966°C) are relatively low in comparison to those of other oxides foreseen as sacrificial materials (MgO, UO₂). This has the advantage of keeping the temperature level of the melt (sacrificial material plus molten-down fuel) at lower values, thus avoiding unwanted chemical reactions and excessive sodium

 Delegated from Euratom to the Karlsruhe Nuclear Research Center, Institute for Neutron Physics and Reactor Engineering. vapor pressure. The core-catcher concept and design, and the development work have been reported elsewhere [1-3]. Since in the literature no data were available on the relevant physical properties of liquid mixtures of borax and of sodium metaborate with UO₂, it was decided to measure them in our Institute. The knowledge of these physical properties is important for the calculations of the temperatures and heat flux distributions in the catcher after a melt-down accident.

In the present paper we report on our measurements of the density, of the volumetric thermal expansion coefficient and of the dynamic viscosity at high temperatures of liquid sodium tetraborate and of sodium metaborate both pure and with two different amounts of UO2 dissolved in each. First results of density measurements have been already published [4]. However some of those measurements have been repeated because during the first series of experiments the temperature measurements were not accurate enough. Furthermore viscosity measurements have been performed for the solution of sodium tetraborate with UO2 and CeO2 and with CeO2 only as well. CeO2 has been used because it is very similar chemically to PuO₂. Table 1 shows data on the eight different types of molten salts which have been tested during the present experiment.

Tabl	e l
Test	samples

Test series number	Type of sodium borate	Quantity of sodium borate	Quantity of dissolved (g)		Weight percent- age of dissolved		M_{Pts} (g)	<i>M</i> _{Ptw} (g)	М _{Р11} (g)
		(g)	UO ₂	CeO ₂	UO ₂	CeO ₂			
1		150	-	_	0%	_		1.991	42.526
2	borax	149.84	12.77	_	7.9%	-	_		42.542
3		151.06	28.39	_	15.8%	-	42.472	2.561	42.647
4		150	_	_	0%	-			42.542
5	sodium	148.569	15.323	_	9.3%				42.542
6	metaborate	147.538	30.601		17.2%	_			42.647
7	borax	76.05	~-	8.45	_	10%	_		_
8	borax	78.165	14.52	2.045	15.3%	2,16%	-	-	

2. Measurements of density and of volumetric thermal expansion coefficient

2.1. Experimental apparatus and data evaluation

The measurements performed during the present experiment were essentially measurements of the density, and thus of its inverse the specific volume, of a liquid at various temperatures. The variation of the specific volume with temperature is of course the volumetric expansion coefficient. Various systems have been suggested to perform these measurements [5], however the requirement to operate at relatively high temperatures (850-1300°c) has dictated the choice of the measure-

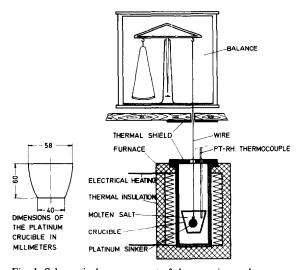


Fig. 1. Schematical arrangement of the experimental apparatus for the density measurements.

ment system: the so-called buoyancy system. This method allows a continuous measurement of the density and the determination of the temperature is precise and simple. The buoyancy method has often been used to measure the density of molten salts [6].

Fig. 1 shows schematically the experimental apparatus. One pan of a precision balance has been replaced by a sinker of known weight attached to the bar of the balance by means of a long wire. In our experiment we used a platinum sinker about 25 mm long and weighting 42.472 g, attached to a 440 mm long wire also of platinum. During the experiments the sinker and a small portion of the wire are immersed in the molten salt, whose density has to be measured. The molten salt is contained in a platinum crucible, the form and dimensions of which are given in fig. 1. The volumetric capacity of the crucible is about 115 cm³. During the tests the volume occupied by the molten salt and by the sinker was about 80 cm³. The crucible was contained in an electrically heated furnace. By varying the heating power, it was possible to obtain various temperature levels in the furnace. The temperature was measured by a platinum/platinum-rhodium (18%) thermocouple immersed in the molten salt. The measurements were performed at temperature intervals of 25°C. After each power variation to obtain another temperature level, the measurement was performed when new stationary temperature conditions in the furnace were achieved, as indicated by the thermocouple reading.

From Archimedes' principle the density of the liquid contained in the crucible is given by:

$$\rho = \frac{M}{M_{\rm Pt}/\rho_{\rm Pt}(T)} = \rho_{\rm Pt}(T) \frac{M_{\rm Pt} - (M_{\rm Pt} - M)}{M_{\rm Pl}}, \quad (1)$$

where:

M = mass of the liquid displaced by the sinker,

 M_{Pt} = total mass of the platinum sinker (M_{Pts}) and platinum wire (M_{Ptw}) ,

 M_{PtI} = mass of the sinker and platinum wire immersed in the liquid,

 $\rho_{Pt}(T)$ = density of platinum as a function of temperature,

 $M_{\rm Pl} - M$ = balance reading at equilibrium.

Experiments were performed with six different types of molten salts (s. table 1). For the first tests with pure borax a thinner wire was used. $M_{\rm Pt}$ and $M_{\rm Pt1}$ are found by weighing sinker and wire before the measurements and recording the small portion of the wire which is immersed during each experiment (see table 1). $M_{\rm Pt}-M$ is given by the balance reading at equilibrium. The density of platinum as a function of temperature was obtained from ref. 7. Eq. (1) therefore allows the determination of the density of the liquid salt.

After the experiments it was observed that the solidified melt of borax containing 15.8% UO_2 presented two well separated regions: one yellow and one green. It was therefore feared that a separation in two regions with different UO_2 concentrations had taken place. Similar, if less pronounced separation of regions were observed with the metaborate $-UO_2$ samples. The samples were therefore subjected to a X-ray fluorescence analysis with

an EXAM-MAX system for the determination of the uranium distribution in the samples. It was found that the uranium is uniformly distributed within the accuracy of the instrument (10%). The different colour and the phase separation is likely to be due to the presence in the $\rm UO_2$ of two slightly different oxidation degrees. It is indeed known that a borax bead containing uranium oxide is orange-yellow in the oxidation flame and green in a reduction flame [8].

2.2. Results

2.2.1. Density

Tables 2 and 3 give the experimental results for the six series of experiments: one with pure borax, one with pure metaborate and the others with two different amounts of dissolved UO_2 in each. The density of the molten salt ρ is calculated with eq. (1) from the balance reading $M_{\rm Pt}-M$, the other parameters being obtained for each series from table 1. Generally the measurements were repeated once, the reproducibility was very good. Only in one case were there differences of up to 1.5% (table 3).

Figs. 2 and 3 show the average values of the two measurements in the plot of density ρ versus temperature T, for borax and sodium metaborate respectively. The present measurements for pure borax lie in between the data of Volarovich [9] and the only point of Pascal

Table 2 Density of borax and of borax-UO₂ solutions [$\rho(g/cm^3)$ or $\rho(10^3 \text{ kg/m}^3)$]

T	Borax Borax with 7.9% UO ₂				Borax with 15.8% UO ₂			
(°C)		lst measur.	2nd measur.	Average	lst measur.	2nd measur.	Average	
850	2.086	2.205	2.219	2.212				
875	2.066	2.192	2.210	2.201				
900	2.056	2.179	2.199	2.189				
925	2.042	2.163	2.188	2.176				
950	2.028	2.152	2.176	2.164				
975	2.017	2.140	2.163	2.151	2.357	_	2.357	
1000	2.006	2.131	2.149	2.140	2.354	_	2.354	
1025	1.989	2.119	2.135	2.127	2.350	_	2.350	
1050	1.980	2.104	2.124	2.114	2.347	2.345	2.346	
1075	1.963	2.093	2.106	2.099	2.343	2.340	2.342	
1100	1.951	2.083	2.092	2.088	2.340	2.338	2.339	
1125	1.941	2.072	2.080	2.076	2.335	2.334	2.335	
1150	1.926	2.061	2.071	2.066	2.332	2.332	2.332	
1175	1.911	2.048	2.053	2.051	2.327	2.327	2.327	
1200	1.896	2.036	2.043	2.040	2.323	2.323	2.323	
1225	1.886	2.028	2.033	2.030	2.320	2.320	2.320	
1250	1.866				2.318	2.316	2.317	
1275	1.849							

Table 3	
Density of sodium metaborate and of sodium metaborate-UO ₂ solutions [ρ (g/cm ³) or	$\rho (10^3 \text{ kg/m}^3)$

T	Sodium m	Sodium metaborate			Sodium metaborate with 9.3% UO ₂			Sodium metaborate with 17.2% UO ₂		
(°C)	lst measur.	2nd measur.	Average	lst measur.	2nd measur.	Average	lst measur.	2nd measur.	Average	
925				1.998	1.969	1.983	2.231	en.	2.231	
950				1.986	1.958	1.972	2.223	_	2.223	
975	1.947	-	1.947	1.967	1.948	1.958	2.216	_	2.216	
1000	1.934	_	1.934	1.953	1.939	1.946	2.207	_	2.207	
1025	1.920	1.911	1.915	1.940	1.921	1.931	2.199	2.207	2.203	
1050	1.910	1.901	1.905	1.929	1.911	1.920	2.191	2.196	2.194	
1075	1.898	1.888	1.893	1.918	1.899	1.909	2.182	2.185	2.183	
1100	1.886	1.876	1.881	1.905	1.890	1.898	2.173	2.176	2.174	
1125	1.865	1.859	1.863	1.891	1.880	1.886	2.164	2.166	2.165	
1150	1.854	1.846	1.850	1.876	1.872	1.874	2.156	2.158	2.157	
1175	1.840	1.831	1.835	1.860	1.864	1.862	2.148	2.149	2.149	
1200	1.823	1.814	1.818							
1225	1.803	1.797	1.800							
1250	1.787	1.777	1.782							
1275	1.767	1.759	1.763							
1300	1.741	_	1.741							

[10], the maximum discrepancy between the data of Volarovich and the present data being of 1.4%. The data-point of Pascal is 1.7% higher than the present prediction (see fig. 2). In the literature data for pure metaborate are also available [11]. These data are about 1.3% lower than the values of the present measurement

(see fig. 3). As one would expect the dissolved UO_2 produces an increase of the density in both cases (borax and metaborate), the increase being greater with an increasing amount of dissolved UO_2 . Only in the case of metaborate with 9.3% UO_2 dissolved is the increase of density quite small (see fig. 3).

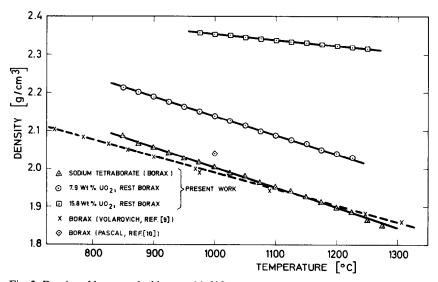


Fig. 2. Density of borax and of borax with UO₂ versus temperature.

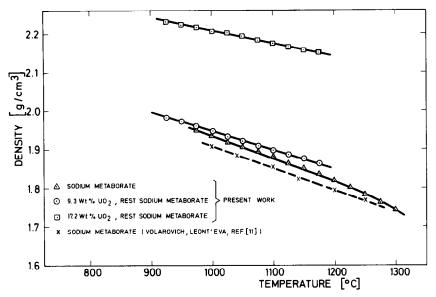


Fig. 3. Density of sodium metaborate and of sodium metaborate with UO2 versus temperature.

2.2.2. Volumetric thermal expansion coefficient

The specific volume of the molten salt

$$v = 1/\rho, \tag{2}$$

and the coefficient β of volumetric thermal expansion are defined by eq. (3):

$$v = v_0 (1 + \beta T), \tag{3}$$

where T is the temperature in ${}^{\circ}C$ and v_0 the specific volume of the molten salt, if it were still liquid, at $0{}^{\circ}C$.

From figs. 2 and 3 it can be seen that up to 1200° C the variation of ρ with temperature is approximately linear. The same can be said of v, and thus the value of β is a constant up to 1200° C for each molten salt investigated. Table 4 and fig. 4 show these constant values of β , valid up to $T = 1200^{\circ}$ C, obtained with eq. (3). For pure borax one has $\beta = 3.83 \times 10^{-4}$ K⁻¹. This

Table 4 Thermal expansion coefficient β up to 1200°C

Test series number	Type of salt tested (K ⁻¹)	$\beta \times 10^4$
1	pure borax	3.83
2	borax with 7.9% UO ₂	3.08
3	borax with 15.8% UO ₂	0.66
4	pure sodium metaborate	4.40
5	sodium metaborate with 9.3% UO ₂	3.41
6	sodium metaborate with 17.2% UO ₂	1.83

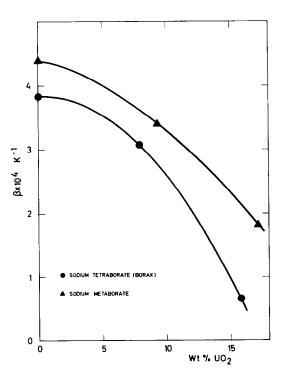


Fig. 4. Coefficient of volumetric thermal expansion as a function of UO₂ weight percentage.

value can be compared with the value obtained by Volarovich by direct measurement of the volume change with temperature: in the temperature range from 859°C to 1305°C β is equal to 2.6×10^{-4} K⁻¹ [9] as quoted in ref. 11. The definition of β used in this relatively old paper is however different from ours. Volarovich uses the definition of β :

$$\beta_{\rm v} = \frac{v(T = 1305^{\circ}\text{C}) - v(T = 859^{\circ}\text{C})}{v(T = 859^{\circ}\text{C}) \times (1305 - 859)}$$
$$= 2.6 \times 10^{-4} \,\text{K}^{-1}. \tag{4}$$

A value of $\beta_{\rm v}=2.6\times 10^{-4}~{\rm K}^{-1}$ corresponds to a value of $\beta=3.35\times 10^{-4}~{\rm K}^{-1}$ with our definition. Thus the difference between our value of β and that of Volarovich is only 12.5%. As far as pure sodium metaborate is concerned the Russian data obtained with a direct measurement of the volume increase with temperature [11] result in a coefficient of thermal expansion β which is with our definition equal to $4.73\times 10^{-4}~{\rm K}^{-1}$ (with the Russian definition $\beta_{\rm v}=3.2\times 10^{-4}~{\rm K}^{-1}$) against our value of $4.40\times 10^{-4}~{\rm K}^{-1}$ (see table 8), the difference being 7.5%.

3. Measurements of viscosity

3.1. Experimental apparatus and data evaluation

Fig. 5 shows schematically the experimental apparatus used for the viscosity measurements. The instrument used was a modified Haake viscosity balance [12]. A sphere is connected by means of a rigid rod to the pan of a precision balance and it is placed in a cylinder containing the liquid whose viscosity has to be measured. The measurement of the velocity of the upward movement of the sphere, caused by placing a known weight on the other pan of the balance, allows the determination of the viscosity of the liquid. Due to the high temperatures, the standard sphere and cylinder of the Haake viscosimeter have been replaced by a sphere and a cylinder of platinum-rhodium (20%). The cylinder is placed in an electrically heated furnace. The platinum-rhodium cylinder is 200 mm long, has an outer diameter of 20 mm and an inner diameter of 15.94 mm. The first series of measurements were performed with a sphere of 15.81 mm diameter (sphere No. 1). This sphere and the cylinder were calibrated at room temperature (20°C) with the calibration liquid E200 [12], which

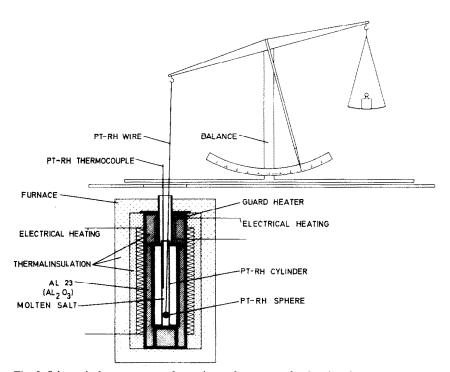


Fig. 5. Schematical arrangement of experimental apparatus for the viscosity measurements.

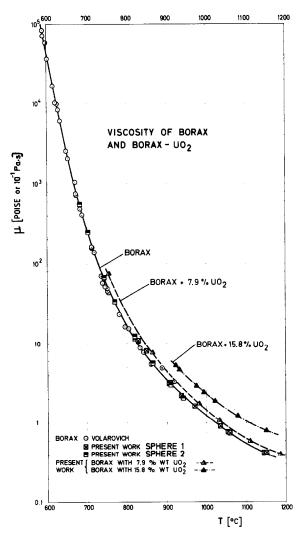


Fig. 6. Viscosity of borax and of borax with UO_2 versus temperature.

has a viscosity of 0.14 Pa s * at 20°C. The same apparatus was calibrated at high temperatures using sodium tetraborate. A considerable amount of literature is available on the viscosity of sodium tetraborate [13–18], however in the range of temperature of interest to us (> 740°C) only the data of the original measurements performed by Volarovich in 1930 are available [19]. These measurements were performed with a rotating cylinder viscosimeter, previously calibrated with a solution of sugar in glycerine of known viscosity. The

data of Volarovich on Na₂B₄O₇ are in the range of temperatures 615-920°C and are shown in fig. 6.

The equation used in the present experiment to evaluate the viscosity is [12]

$$\mu = FGt, \tag{5}$$

where μ is the dynamic viscosity of the melt in Pa s (kg/m s), G is the weight on the pan of the balance (kg), t is the time required by the pointer to cover a pre-set range on the balance dial (s) (see fig. 5). F is the calibration constant (m⁻¹ s⁻²). The pre-set range on the balance dial was chosen so that $F = 4.00 \times 10^{-2}$ m⁻¹ s⁻². This value of F was obtained with the calibration with E200 at room temperature and with the sphere of 15.81 mm diameter. Using the same calibration constant we obtained values for the viscosity of borax in the temperature range 828-1151°C, which agree well with the Volarovich data [19].

For the measurements of the higher values of viscosity a sphere with a smaller outer diameter is more appropriate, otherwise the values of G and/or t become too big. A second platinum-rhodium (20%) sphere was chosen of 15.00 mm outer diameter. The calibration constant for this sphere was obtained by performing measurements with pure borax at various temperatures and by comparison with the borax data of Volarovich for the lower temperatures and with our own borax data with sphere 1 for the higher temperatures. The resulting constant is $F = 2.90 \text{ m}^{-1} \text{ s}^{-2}$ for the same range on the balance dial.

Using these calibration constants we could then perform the measurements for the same borax-UO₂ and sodium metaborate-UO₂ solutions for which we performed the density measurements, and for a borax-CeO₂ solution.

Finally we measured the viscosity of a solution of sodium tetraborate with 15.3 wt% UO₂ and 2.16 wt% CeO₂, the weight ratio between CeO₂ and UO₂ corresponding approximately to the weight ratio between UO2 and PuO2 in a large fast reactor. The measurements for this salt were performed with a platinum-rhodium (20%) cylinder of 16.04 mm inner diameter and with a sphere of 15.82 mm outer diameter (sphere No. 3). Also in this case the calibration constant was obtained by performing measurements with pure borax for various temperatures and comparing the new data with the previous borax measurements. The resulting calibration constant (for a slightly higher range on the dial as for the previous tests: 50 units instead of 47.77 units), $F = 4.72 \times 10^{-2} \text{ m}^{-1} \text{ s}^{-2}$, was then used to evaluate the measurements for the solution borax +15.3% UO₂ and 2.16% CeO₂.

^{* 1} Pa s = 1 kg/m s = 10 g/cm s = 10 poise.

During the calibration tests with pure borax we used a relatively large range of weights G (between 5 and 450 g) and of times t (between 14 and 200 s). This was done to extend as much as possible the range of our measurements to allow a better comparison of our data with those of Volarovich. For the measurements with the other salts we used narrower ranges for G and t (30-70 g and 25-60 s respectively) to allow a better reproducibility of the results. Each measurement was repeated 4 to 6 times, sometimes with different weights. The reproducibility of the measurements was always within $\pm 5\%$ and in most of the cases within $\pm 2\%$.

By means of a very thick extra thermal insulation (50 mm Kerlane plus 16 mm Al_2O_3 besides the normal furnace insulation) and the use of an independently controlled guard-heater to compensate for axial heat losses in the upper part of the furnace (see fig. 5) we obtained a relatively uniform and constant temperature in the region of the Pt-Rh cylinder. The uncertainty in the temperature (inclusive of measurement error and variation in time and space during the measurement) is estimated at $\pm 2^{\circ}C$.

3.2. Results

All the viscosity measurements are reported in tables 5 to 8. Fig. 6 and tables 5 and 6 show the results for borax and the borax-UO₂ solutions. The figure shows clearly that borax is a glass: it is not meaningful to speak of a melting point at 740°C, which sometimes is quoted in the literature, but the viscosity varies gradually down below this temperature. The adding of UO₂ produces an increase of the viscosity of borax. At 1000°C

Table 6 Viscosity of borax-UO₂ solutions

Borax + 7.9% UO ₂		Borax + 15.8% UO ₂			
T(°C)	μ (Pa s)	$\overline{T(^{\circ}C)}$	μ (Pa s)		
754	7.53	923	0.535		
864	0.774	931.5	0.478		
982	0.176	975	0.295		
1032	0.107	994	0.243		
1110	0.0593	1022	0.191		
1187	0.0401	1079	0.124		
		1151	0.0811		

the viscosity increases of 17% with 7.9% UO_2 and of 53% with 15.8% UO_2 . For temperatures below 920°C the measurements with the borax -15.8% UO_2 solution (not shown in fig. 6) indicated a phase separation in the cylinder.

The viscosity of sodium metaborate and its solutions with UO₂ are considerably smaller than that of borax: see table 7 and fig. 7. Also here the adding of UO₂ increases the viscosity: at 1000°C of 39% for 9.3% UO₂ and of 55% with 17.2% UO₂. Contrary to borax the sodium metaborate solutions show a well defined melting point: tentatives to measure the viscosity for temperatures only slightly below those indicated in table 8 indicated the formation of a solid phase.

The results of the measurements for the borax-10% CeO₂ solution are shown in table 8. For temperatures below 1159°C a phase separation in the cylinder was revealed by the measurements. Table 8 and fig. 8 show the measured viscosities for the solution borax + 15.3%

Table 5 Viscosity of pure sodium tetraborate (borax)

Sphere No. 1 $F = 4.00 \times 10^{-2} \text{ m}^{-1} \text{ s}^{-2}$		Sphere No. 2 $F = 2.90 \text{ m}^{-1} \text{ s}^{-2}$		Sphere No. 3 $F = 4.72 \times 10^{-2} \text{ m}^{-1} \text{ s}^{-2}$	
T(°C)	8gm (Pa s)	T(°C)	μ (Pa s)	T(°C)	μ (Pa s)
828	1.09	682	55.3	897	0.361
906	0.314	703	24.6	980	0.145
937.5	0.220	743	6.67	982	0.138
1054	0.0762	769	3.32	1084.5	0.0664
1151	0.0426	769.5	3.35	1086	0.0632
849	0.821	819.5	1.14	1087	0.0595
908	0.338	820	1.23	1164	0.0391
971	0.164	865	0.574		
1035	0.0905	910	0.300		
1059	0.0744	941	0.206		
1145	0.0427				

able 7
iscosity of sodium metaborate and of sodium metaborate-UO ₂ solutions

Pure sodium metaborate		Sodium metaborate + 9.3%UO ₂		Sodium metal + 17.2%UO ₂	borate
$T(^{\circ}C)$	μ (Pa s)	T(°C)	μ (Pa s)	T(°C)	μ (Pa s)
967	0.0230	963	0.0348	991	0.0364
997	0.0211	1010	0.0267	1005	0.0314
998	0.0209	1048	0.0213	1028	0.0274
1035	0.0177	1104	0.0166	1075	0.0221
1037	0.0174	1152	0.0119	1106	0.0190
1072	0.0141	1195	0.0112	1149	0.0155
1120	0.0120	1238	0.00911	1216	0.0115
1148	0.00975				
1222.5	0.00727				

 $\rm UO_2 + 2.16\%~CeO_2$. Comparison of fig. 8 with fig. 6 shows that for this solution the viscosity is higher than for the solution with 7.9% $\rm UO_2$ but lower than for the solution with 15.8% $\rm UO_2$. The viscosity for the mixture borax +15.3% $\rm UO_2 + 2.16\%~CeO_2$ is about the same as for the mixture borax with 10% $\rm CeO_2$.

Acknowledgements

The authors gratefully acknowledge the help of K. Schorb who carried out the density measurements and of Mrs I. Schub, who performed the X-ray fluorescence analysis.

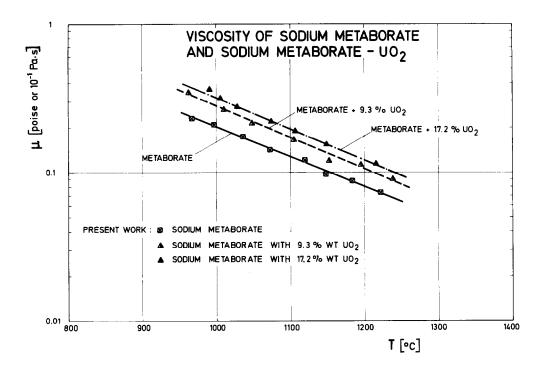


Fig. 7. Viscosity of sodium metaborate and of sodium metaborate with UO2 versus temperature.

Table 8	
Viscosity of sodium tetraborate (borax) with CeO2, U	JO_2

Borax + 10% CeO ₂		Borax + 15.3% UO ₂ + 2.16% CeO ₂					
		1st measurement		2nd measuren	nent		
T(°C)	μ (Pa s)	T(°C)	μ (Pa s)	T(°C)	μ (Pa s)		
1203	0.0480	1197	0.0480	1060	0.107		
1206	0.0438	1205	0.0458	1113	0.0798		
1213	0.0457	1180	0.0541	1141	0.0676		
1225	0.0437	1156	0.0639	1159	0.0597		
1186	0.0533	1125	0.0720	1202	0.0440		
1159	0.0616	1015	0.142	1252	0.0357		
		1029	0.127				
		1083	0.0860				
		1098	0.0781				

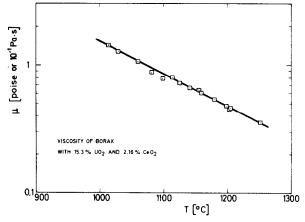


Fig. 8. Viscosity of borax with 15.3% UO_2 and 2.16% CeO_2 versus temperature.

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