Table VI. Values of Coefficients for Empirical Polynomial[a] for Densities of the Molten $K_2S_2O_7$-$KHSO_4$-V_2O_5 System at Compositions around $X_{KHSO_4} = 0.6$ and $0.0000 \leqslant X_{V_2O_5} \leqslant 0.0450$

A_0	2.049 12
A_1	0.781 56
A_2	−6.700 15
B_0	$-0.114\,68 \times 10^{-2}$
B_1	$0.322\,00 \times 10^{-2}$
B_2	$-1.210\,90 \times 10^{-2}$
SE, g/cm³	0.0003

[a] $\rho = A_0 + A_1X_2 + A_2X_2^2 + (B_0 + B_1X_2 + B_2X_2^2)X_1(t - 400)$; $X_1 = X_{KHSO_4}$, $X_2 = X_{V_2O_5}$.

$A_2X_2^2 + (B_0 + B_1X_2 + B_2X_2^2)X_1(t - 400)$. The values for the employed parameters are given in Table VI. The nonlinearity of the system suggests that depending on concentration different complexes of vanadium(V) are formed. In contrast to the $K_2S_2O_7$–$KHSO_4$ system, the $K_2S_2O_7$–$KHSO_4$–V_2O_5 system is not ideal.

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Received for review January 11, 1980. Accepted September 2, 1980. We acknowledge financial support from Statens teknisk-videnskabelige Forskningsråd.

Vapor–Liquid Equilibria of the NaCl–H_2O System in the Temperature Range 300–440 °C

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A complete apparatus is described for the phase study of saltwater systems at high pressures and temperatures. An original technique is presented for the direct sampling of solutions with high salt concentration. These have been applied to the liquid–vapor equilibrium determination of the system NaCl–H_2O in the range 300–440 °C at concentrations up to quasisaturation. The data obtained confirm previously published work and solve the uncertainty in several sources of data. Diagrams and correlations are presented for eight isotherms.

Introduction

Since the late thirties, a large amount of work has been published on saltwater systems at high temperatures and pressures. The interest of these studies is scattered in such different fields as fundamental geology, geothermal power plant technology, high-pressure steam power plants, hydrothermal synthesis, fundamental thermodynamics of solutions, and seawater desalination processes.

High-temperature distillation has been mentioned by several authors (*1–3*) as a possible alternative to water (especially seawater) desalination, and some work has been published in the field (*4–6*). However, some thermodynamic parameters, such as phase equilibria in the high-concentration range and the latent heat of vaporization under these conditions, have not been determined up to now and are the focus of our present investigations (*7*). We have limited our interest to the system NaCl–H_2O, which is the best-known binary system at high temperature, the knowledge of which is fundamental for extrapolation to seawater.

Previous Work

The system NaCl–H_2O has been extensively studied up to 300 °C, allowing publication of preliminary steam tables for NaCl solutions by Haas (*8*, *9*).

At higher temperatures, under the conditions of coexisting liquid and vapor phases, the system has been investigated by Keevil (*10*), who studied the parameters of the saturated solution in the range 180–650 °C. Oelander et al. (*2*) determined the liquid–vapor equilibria between 350 and 475 °C, at liquid-phase concentration up to 25%, work which has been extended up to 700 °C by Sourirajan et al. (*1*). Using radioisotopes, Khaibullin et al. (*11*, *12*) measured the same equilibria in the range 100–440 °C. Other results have been presented by Urosova (*13*), Copeland et al. (*14*), and Styrikovich et al. (*15*).

However, there are large divergences among the three major contributions (*1*, *2*, *12*) as pointed out in the Gmelins Handbuch (*16*). Moreover a lack of data at concentrations higher than 25%, which is attributed to the difficult handling of solutions under these conditions, encouraged us to reascertain the vapor–liquid equilibria and extend the measurements up to the saturation point.

Apparatus

Our measurements are based on the sampling method described in the literature (*1*, *2*). This technique has important advantages over other indirect methods as long as the ratio of the weight of the sample to the total weight of the phase remains low. For this purpose we have designed a complete high-pressure, high-temperature apparatus which is schematically represented in Figure 1.

A 1-L 316 SS autoclave with standard ¹/₄-in. o.d. tubing and high-pressures valves has been used throughout this investigation. The autoclave is fitted out with two heating devices: an internal coaxial insulated heater CH1 driven by a high-precision PID regulator and an external heater CH2 made of two half-cylindrical aluminum blocks equipped with coaxial heating elements, piloted by a PI regulator. The conjugated use of two

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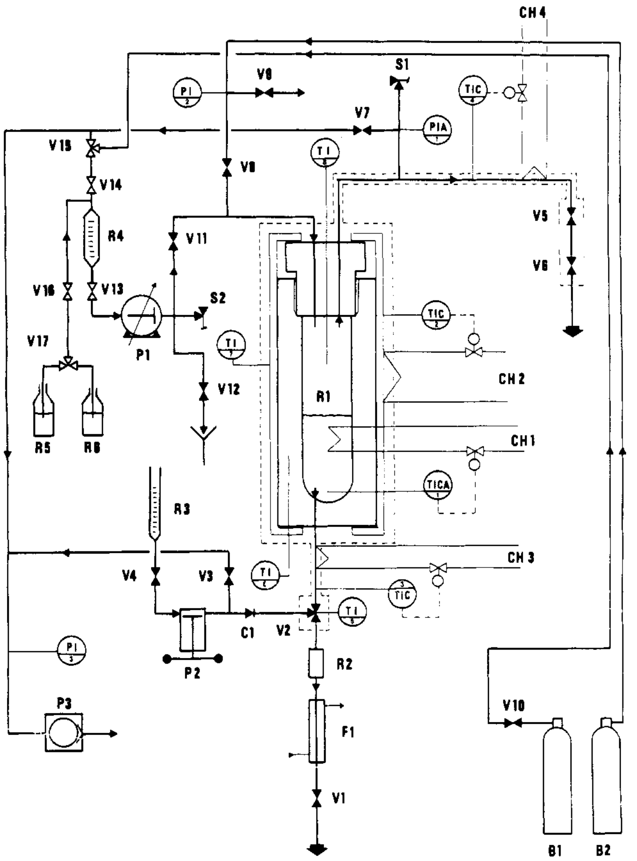

Figure 1. Experimental apparatus for equilibria measurements at high pressures and temperatures.

regulating loops (TIC 2 and TIC 1) allows a quick and very effective stabilization of temperature (ripple < 0.1 °C).

The feed line includes a tank of pure bidistilled water and one of standard salt solution (R5 and R6), the quantities of which are measured in volumeter R4 before being pumped into the autoclave R1 through the high-pressure piston pump P1.

The vapor sampling line, which was made as short as allowed by design, has its temperature independently regulated by heater CH_4, so that the vapor samples can be withdrawn without temperature drop along the line.

The sampling of solutions with a NaCl concentration higher than 25% usually leads to partial crystallization of salt as the temperature drops and to plugging of the line (*1*). To solve this problem, we have designed a special high-pressure dilution unit comprising the volumeter R3, the precision screw pump P2, the double valve V2, the dilution volume R2, the heat exchanger F1, and the valve V1. Moreover the tubing between the autoclave and the valve V2 has its temperature regulated by heater CH3.

All temperatures are measured by coaxial insulated Inconel sheathed thermocouples (type chromel-alumel) and recorded on a 12-channel Philips recorder. The thermocouples were calibrated before the experiments. The temperature of the solution is simultaneously measured on a precision digital voltmeter with a 1-μV resolution. The pressure is determined by a digital pressure gauge whose precision is 0.1% full scale ($\pm$0.7 kg/cm^2).

Experimental Method

Before the introduction of solution, the air is removed by vacuum pump P3, and the calibration and the zero point of the pressure gauge PIA 1 are adjusted. Then, known amounts of standard salt solution and water (the respective quantities are calculated according to the density data of Khaibullin (*12*) to have ~5% salt solution at first equilibrium) are pumped into the autoclave, and 3 h is allowed to reach equilibrium. However, we have observed that internal heating provides an excellent convective agitation so that equilibrium is actually quickly established. Eventually, after recording of pressure and temperature, the first sample of the liquid phase is withdrawn according to the following procedure. The circuit from valve V4 to valve V1, completely filled with water, is put under moderate pressure by manual pump P2, the valves V2 and V1 are slowly opened, and a sample of solution goes from the autoclave to the dilution volume R2. The expelled water from valve V1 is collected in a tared flask. Then V2 is closed (V2 is a double valve, and the valve from C1 to R2 is usually opened). Afterward, pure water is pushed by pump P2 to dilute the sample and to rinse out the circuit between V2 and V1. At the same time we collect the sample plus the dilution water, the quantity of which we measure in volumeter R3. (The washing quantity is 3 × 10 mL. The exact amount of water is known simultaneously from the pump scale and from volumeter R3 after filling up the line). By difference one calculates the weight of the sample, and an analysis gives its salt concentration. This technique enables the sampling of quasi-saturated solutions without plugging of the line by salt crystals. This was not possible with the techniques used heretofore. Control experiments at room temperature with solutions of known concentrations showed that the above procedure enables the concentration to be measured with a ±1% precision.

The sampling of the vapor phase is performed immediately after as described below. The output tubing from valve V6 is plunged into a flask containing a weighed amount of bidistilled water. Then the valve is carefully opened, and bubbles of steam immediately condense in the water. The weight of the sample is known exactly by difference. The temperature and the pressure are recorded immediately afterward. The pressure drop caused by the above operations in all cases was less than 1 kg/cm^2, and the temperature drop less than 0.5 °C. The samples range from 2 to 5 g for the liquid phase and from 1 to 2 g for the vapor phase. Between each removal, 20 min was allowed for restoring equilibrium.

After a series of samplings at a given pressure, the concentration of the solution is increased by injection of more concentrated solution and/or removal of steam to maintain roughly constant masses of both phases, so the measurements are performed along isotherms of increasing concentration in salt (and correspondingly decreasing pressure). The analysis of liquid-phase samples is performed by potentiometric titration by $AgNO_3$. For the vapor phase we used a specific electrode for Na^+ ions which is suitable for the concentration range down to 10^{-4}% (precision: ±2%).

Results and Discussion

At 300, 320, 340, 360, 380, 400, 420, and 440 °C, we have determined the salt concentration of the liquid phase as a function of saturation pressure. The results are presented in Table I. For each temperature, the highest concentration measured corresponds to the last series of samples removed without trouble (plugging of the line or unhomogeneous sample with erratic salt content). After the last sampling, we always checked that the saturation point was not reached by removing vapor and observing a pressure drop.

At supercritical temperatures (with respect to pure water), where salt dissolution in steam becomes appreciable, we have measured the salt content of steam, and the data are presented in Table I. The pressure, the temperature dependance of K_D, and the distribution ratio of salt between phases are given in Figure 2.

All data presented are mean values of 2, sometimes 3, consistent samples, each of them having been twice analyzed. For each equilibrium the first sample of liquid and vapor has

Table I. Pressure-Temperature-Composition Data in the Vapor-Liquid Equilibrium Region in the NaCl-H_2O System

Temperature [°C]	Pressure [kg/cm^2]	[NaCl]$_\ell$ [%]	[NaCl]$_v$ [%]	K_D
300	84.8	6.0	not measured	-
"	81.0	10.1	"	-
"	77.8	15.3	"	-
"	74.1	19.0	"	-
"	71.2	23.8	"	-
"	69.4	25.0	"	-
"	67.2	29.2	"	-
"	64.3	32.4	"	-
"	60.2	35.1	"	-
320	109.8	6.9	not measured	-
"	105.7	10.1	"	-
"	101.2	17.4	"	-
"	97.2	20.7	"	-
"	90.2	26.7	"	-
"	84.2	31.7	"	-
"	80.8	36.8	"	-
"	77.4	37.8	"	-
340	142.5	8.3	not measured	-
"	136.6	10.1	"	-
"	131.1	18.4	"	-
"	126.8	20.5	"	-
"	119.7	24.9	"	-
"	116.7	28.6	"	-
"	113.2	29.7	"	-
"	108.1	32.0	"	-
360	180.8	6.8	not measured	-
"	174.0	9.4	"	-
"	162.3	18.5	"	-
"	160.5	19.5	"	-
"	154.3	23.2	"	-
"	150.5	26.8	"	-
"	142.9	29.8	"	-
"	136.5	33.9	"	-
"	130.8	37.5	"	-
380	227.1	6.3	$7.0\ 10^{-2}$	$1.1\ 10^{-2}$
"	220.5	8.6	$3.4\ 10^{-2}$	$3.9\ 10^{-3}$
"	209.7	15.0	$2.8\ 10^{-2}$	$1.9\ 10^{-3}$
"	203.4	18.6	$1.7\ 10^{-2}$	$9.1\ 10^{-4}$
"	191.3	23.7	$1.3\ 10^{-2}$	$5.5\ 10^{-4}$
"	184.0	28.3	$9.1\ 10^{-3}$	$3.2\ 10^{-4}$
"	175.9	29.7	$8.5\ 10^{-3}$	$2.8\ 10^{-4}$
"	168.4	34.4	$7.2\ 10^{-3}$	$2.1\ 10^{-4}$
"	162.5	40.5	$4.1\ 10^{-3}$	$1.0\ 10^{-4}$
"	154.3	42.	-	-
400	283.7	7.3	$2.1\ 10^{-1}$	$2.8\ 10^{-2}$
"	274.4	9.8	$1.0\ 10^{-1}$	$1.0\ 10^{-2}$
"	248.3	21.1	$3.3\ 10^{-2}$	$1.5\ 10^{-3}$
"	238.6	25.1	$7.4\ 10^{-3}$	$2.9\ 10^{-4}$
"	231.6	27.2	-	-
"	225.5	30.2	$3.6\ 10^{-3}$	$1.2\ 10^{-4}$
"	214.7	32.1	$3.3\ 10^{-3}$	$1.0\ 10^{-4}$
"	209.0	39.1	$3.4\ 10^{-3}$	$8.7\ 10^{-5}$
"	194.5	42.6	$1.3\ 10^{-3}$	$3.0\ 10^{-5}$
420	329.8	10.7	$8.0\ 10^{-1}$	$7.4\ 10^{-2}$
"	320.6	15.1	$5.4\ 10^{-1}$	$3.5\ 10^{-2}$
"	309.8	18.5	$3.4\ 10^{-1}$	$1.8\ 10^{-2}$
"	303.5	20.5	$2.7\ 10^{-1}$	$1.3\ 10^{-2}$
"	292.0	25.6	$1.4\ 10^{-1}$	$5.4\ 10^{-3}$
"	288.9	25.8	$1.4\ 10^{-1}$	$5.4\ 10^{-3}$
"	279.3	28.7	$9.7\ 10^{-2}$	$3.4\ 10^{-3}$
"	265.8	32.5	$6.0\ 10^{-2}$	$1.8\ 10^{-3}$
"	253.5	35.3	$4.2\ 10^{-2}$	$1.2\ 10^{-3}$
"	239.9	38.5	$2.5\ 10^{-2}$	$6.5\ 10^{-4}$
"	231.1	41.6	$2.1\ 10^{-2}$	$5.0\ 10^{-4}$
"	221.3	45.2	$1.6\ 10^{-2}$	$3.5\ 10^{-4}$
440	395.9	10.1	3.4	$3.4\ 10^{-1}$
"	379.9	16.0	1.3	$8.1\ 10^{-2}$
"	366.2	19.5	$6.5\ 10^{-1}$	$3.3\ 10^{-2}$
"	346.3	26.5	$3.4\ 10^{-1}$	$1.3\ 10^{-2}$
"	342.3	29.4	$2.4\ 10^{-1}$	$8.2\ 10^{-3}$
"	324.0	32.3	$1.9\ 10^{-1}$	$5.9\ 10^{-3}$
"	303.7	41.3	$8.0\ 10^{-2}$	$1.9\ 10^{-3}$
"	284.9	45.5	$4.9\ 10^{-2}$	$1.1\ 10^{-3}$
"	266.5	48.8	-	-

always been disgarded as not truly representative of the equilibrium. One has to notice that the two or three successive samples correspond to slightly different equilibria since each removal is not compensated by a corresponding injection of solution. Actually the difference of pressure between the first and the last one was less than 0.5%. For every series of data, we calculated the coefficient of variation $CV = 100V^{1/2}/\bar{x}$, where V is the variance and $\bar{x}$ the mean value. In all cases $CV \leq 2.5\%$.

Moreover, we have compared our results to previously published data (*1*, *2*, *8*–*10*, *12*). At liquid-phase concentrations up to 25%, our results are in excellent agreement with those of Khaibullin (*12*) and Oelander (*2*) and differ from Sourirajan's (*1*) data, as shown in Figure 3. At 300 °C one can compare our results with compiled tables of Haas (*8*), and, here too, the agreement is good. At higher concentrations our measures converge satisfactorily on values at saturation calculated from quadratic interpolation of Keevil's data (*10*).

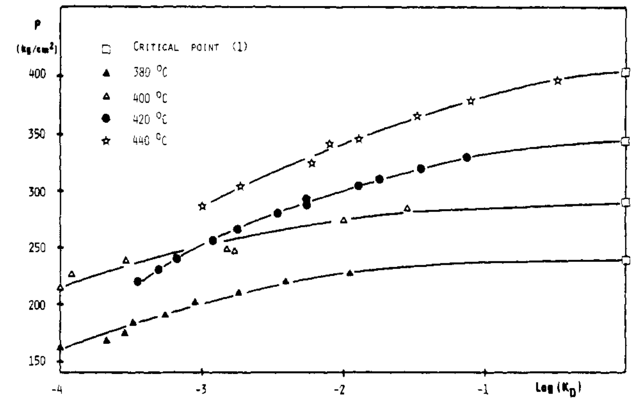

Figure 2. Coefficient of distribution of NaCl between vapor and liquid phases in function of the pressure and the temperature.

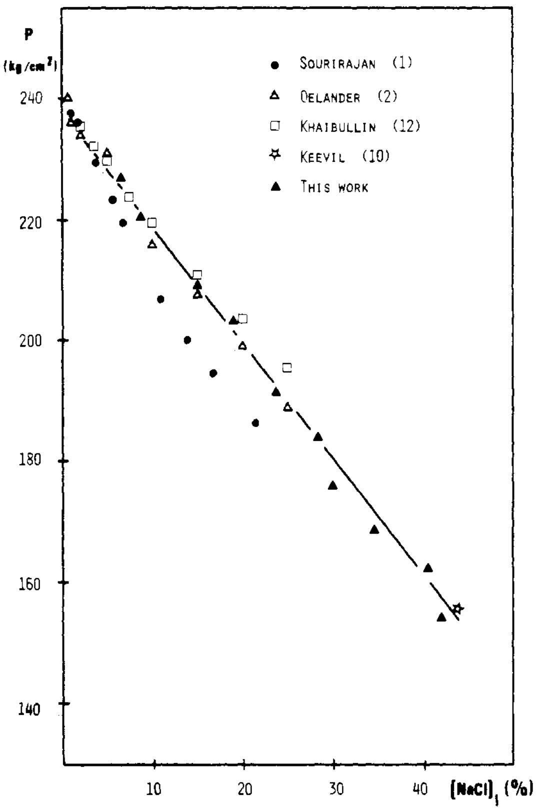

Figure 3. Comparison of experimental data of liquid-phase composition at 380 °C.

We have computed the function of the salt content of the liquid in relation to the saturation pressure for each isotherm by using Sourifajan's data for the critical line, Keevil's values for the saturation line, and Khaibullin's and our data for the intermediate liquid-phase composition. Three types of regression were considered: linear, quadratic, and exponential. The best correlations were always obtained with the quadratic regression $[NaCl]_l = A + BP + CP^2$ where P is the pressure and $[NaCl]_l$ the concentration of salt in the liquid phase in percent. The results are shown in Table II and Figure 4. On the other hand, we observed generally that the vapor-phase composition fits Khaibullin's data quite satisfactorily and differs systematically from Sourirajan's, as shown in Figure 5, at 380 °C.

Glossary

$[NaCl]_l$	concentration of NaCl in the liquid phase (%)
$[NaCl]_v$	concentration of NaCl in the vapor phase (%)
P	pressure (kg/cm²)
T	temperature (°C)
K_D	distribution coefficient = $[NaCl]_v/[NaCl]_l$

Table II. Empirical Equations from Quadratic Regression Model[a] $y = A + Bx + Cx^2$

T, °C	A	B	C	N	r^2
300	66.803 412	0.005 757	−0.008 596	20	0.989
320	84.465 088	−0.321 681	−0.003 523	19	0.989
340	72.834 642	−0.070 610	−0.002 750	19	0.987
360	91.499 169	−0.291 293	−0.000 975	20	0.990
380	78.845 326	−0.087 008	−0.001 022	21	0.992
400	63.117 020	0.077 476	−0.000 993	18	0.990
420	46.570 273	0.183 662	−0.000 886	19	0.997
440	17.687 627	0.383 699	−0.001 025	14	0.991

[a] Nomenclature: y = % NaCl in liquid phase; x = pressure (kg/cm²); N = number of experimental points; $r^2 = [A\Sigma y + B\Sigma xy + C\Sigma x^2 y - (1/N)(\Sigma y)^2]/[\Sigma y^2 - (1/N)(\Sigma y)^2]$ correlation coefficient of the regression.

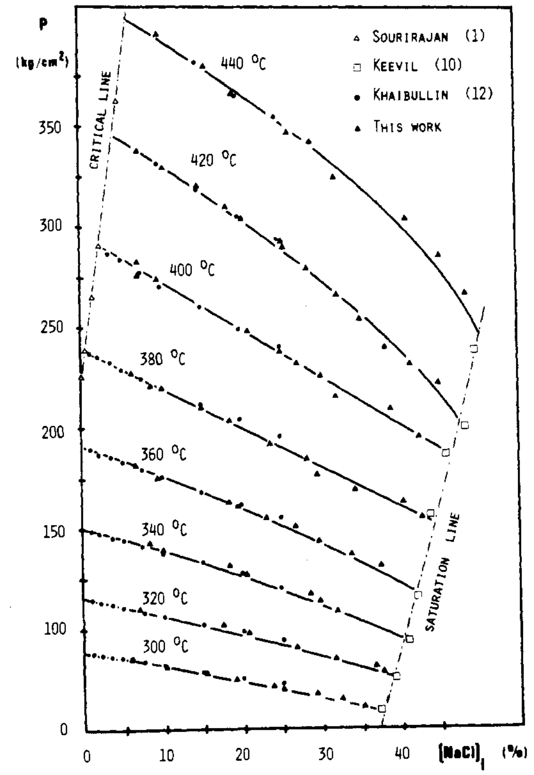

Figure 4. Liquid-phase composition in the range 300–440 °C based on regressions of consistent data.

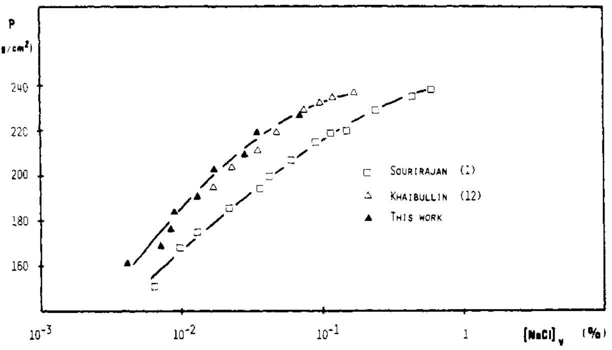

Figure 5. Comparison of experimental data of vapor-phase composition at 380 °C.

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Received for review February 5, 1980. Accepted August 29, 1980.

Density and Viscosity in the Binary System Octadecanoic Acid–Hexanedioic Acid

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The experimental values of density and viscosity and the calculated values of molar volume, volume coefficient of thermal expansion, and activation energy are given for the binary system octadecanoic acid–hexanedioic acid.

Introduction

As a part of broader research program on physicochemical properties of pure and mixed organic melts (*4–6, 8*), the results of measurements of density and viscosity on the binary mixture octadecanoic (stearic) acid–hexanedioic (adipic) acid, in a temperature range above the melting curve (*1*), are presented herein.

Experimental Section

Density. The density measurements were performed by means of the Archimedean method described in the literature (*7*). A Mettler H 20 T analytical balance mounted above the thermoregulated furnace was used. The level of the furnace was changed by means of a laboratory elevator. From the pan of the balance a gold chain, joined to a steel double cone bob by means of a steel wire (0.05-mm diameter; 1-cm length), allowed the measurements to be performed. The melt was placed in a Pyrex container positioned in the central zone of the furnace, and the furnace was raised until the bob and half of the steel wire were submerged. This condition was observed by means of a set of mirrors. The temperature of the furnace was controlled by means of a chromel–alumel thermocouple connected to a Leeds and Northrup CAT system, and the temperature of the melt was read with a second thermocouple immersed in the fused substance. The thermocouple was checked against a Pt-resistance thermometer certified by NBS and connected to a Leeds and Northrup K-3 potentiometer. The system was calibrated by using H_2O, KCNS, $LiNO_3$, and $NaNO_3$ (*7*) for which the densities are known. The following equation was obtained for the volume of the bob up to 350 °C: $V(\text{cm}^3) = 1.172_0 + 8.71 \times 10^{-5}t$. In Table I the process of calibration in molten $NaNO_3$ is reported. In run I the level of the furnace is settled in order to submerge the bob and half of the steel wire. In run II the balance is locked and then released after some minutes. In run III the level of the furnace is changed and then brought again to the same level as in run I. A precision on the density of $\pm 2 \times 10^{-4}$ g/cm^3 is obtained. The molar volume of lauric acid (*2*) measured with this system agrees with that measured by a conventional pycnometric method (*3*): 234.5 and 234.0 cm^3/mol, respectively, at 345.1 K.

Table I. Measures of Calibration of the Bob in $NaNO_3$

run	t, °C	$W_{\text{air}}^{\text{bob}} - W_{\text{melt}}^{\text{bob}}$, g	V_{bob}, cm^3
I	329.8	2.266_0	1.199_7
II	329.9	2.266_0	1.199_7
III	329.9	2.265_2	1.199_4

Table II. Density as Function of the Temperature at Different Compositions[a]

T, K	$10^{-3}d$, kg/m^3	T, K	$10^{-3}d$, kg/m^3
	$x_1 = 0.0$		
351.8	0.8404	393.9	0.8102
352.7	0.8397	406.1	0.8011
368.7	0.8286	433.2	0.7826
380.9	0.8202	461.1	0.7609
383.4	0.8184		
	$x_1 = 0.2024$		
424.0	0.8145	453.4	0.7930
431.7	0.8083	476.0	0.7754
442.5	0.8007	477.2	0.7744
	$x_1 = 0.4002$		
435.1	0.8321	460.2	0.8135
437.9	0.8308	481.2	0.7960
448.4	0.8229	483.1	0.7945
	$x_1 = 0.6606$		
431.7	0.8888	456.7	0.8692
436.7	0.8866	465.7	0.8263
448.1	0.8751		
	$x_1 = 0.8064$		
434.1	0.9684	464.9	0.9447
448.7	0.9602	473.8	0.9298
458.4	0.9472	473.3	0.9294
	$x_1 = 1.0$		
434.5	1.0916	461.1	1.0700
436.8	1.0893	476.2	1.0582
459.8	1.0710		

[a] x_1 = molar fraction of the adipic acid in the mixture.

Viscosity. The viscosity was measured by means of a commercial VEB MLW, DDR Freital, rotating viscometer. The substance was placed in the annular space between two coaxial cylinders. The stationary outer cylinder of radius R was the sample container and was surrounded by a circulating thermostat liquid. The internal cylinder of radius r and length l rotated at a selected constant angular speed ω (24 different