value from four measurements in Pittsburgh (slightly different concentrations) is $\Delta H_1 = -97.11$ (±0.13) kcal. per mole.

Auxiliary data that have been combined with the above values of ΔH_1 are as follows: H_2O(liq), $\Delta H_f^\circ = -68.32$ kcal. per mole (*5*). For the heat of solution of sulfamic acid, $\Delta H = 4.54$ kcal. per mole (*6*). For $NaHSO_4$(c), $\Delta H_f^\circ =$ -269.8 kcal. per mole. This value is derived from that given by Rossini *et al.* (*4*) following recent revision (*5*) of ΔH_f° ($H_2SO_4 \cdot 200\ H_2O$). Independent measurements in Manchester lead to $\Delta H = -4.0 \pm 0.3$ kcal. per mole for the heat of solution of $NaHSO_4$(c) in a solution of $NH_2SO_3H \cdot 20000\ H_2O$. The salt dissolved slowly and more precise measurements were not possible with the calorimeter available. For $NaNO_2$(c), the authors calculate $\Delta H_f^\circ = -85.7$ kcal. per mole from its heat of solution (*6*) and ΔH_f° values (*5*) for Na^+ (aq) and NO_2^- (aq).

The measured ΔH_1 values are combined with the above auxiliary data to obtain $\Delta H_f^\circ = -164.0_2$ (Manchester) and -163.8_5 (Pittsburgh) kcal. per mole for crystalline sulfamic acid. The average $\Delta H_f^\circ = -163.9$ kcal. per mole for NH_2SO_3H(c) is, the authors believe, the best value presently available.

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RECEIVED for review October 18, 1967. Accepted November 29, 1967. Work supported in part by the National Science Foundation.

Density, Surface Tension, and Viscosity of NaF–NaCl–NaI Eutectic

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Measurements of the density, surface tension, and viscosity of the 6.1 wt. % NaF–17.7 wt. % NaCl–76.2 wt. % NaI salt eutectic mixture, as a function of temperature, were undertaken in connection with the use of this salt as an electrolyte in engineering studies of lead-sodium and bismuth-sodium galvanic cells at Argonne National Laboratory. The salt mixtures used were prepared from reagent grade materials. No information on these physical properties for the NaF–NaCl–NaI eutectic mixture were noted in the literature.

THE eutectic mixture of NaF–NaCl–NaI was contained in a stainless steel crucible for making measurements of the density, surface tension, and viscosity as functions of temperature. The crucible was placed in a well of stainless steel pipe, which was attached to the bottom of a helium atmosphere glovebox; the well was heated by an electric resistance furnace. The temperature of the eutectic mixture, which melts at about 535° C., was measured by a Chromel-Alumel thermocouple.

The density measurements consisted of measuring the depth of a known weight of the salt in a crucible of calibrated cross sectional area and volume. The depth of the liquid was measured by means of a pointed stainless steel rod attached to a micrometer depth gage measuring apparatus. The rod was $\frac{1}{16}$ inch in diameter. To make a measurement, the rod was lowered to the bottom of the crucible; after the rod had reached temperature equilibrium, the micrometer was set to zero. The rod was then raised to just above the liquid; after reaching temperature equilibrium, it was lowered until the point just touched the surface of the liquid. Contact with the melt was determined by an electrical resistance meter (ohmmeter). The micrometer was read giving the liquid salt depth. Repeated measurements were made to check the readings.

Corrections were made for the thermal expansion of the crucible, which had been calibrated at room temperature, and for the thermal expansion of the measuring rod. At the end of a series of runs, the crucible and salt were weighed to check with the weight of the salt placed in the crucible initially.

The surface tension was determined by measuring the force required to detach a calibrated surface tension ring from the surface of the molten salt. The force was measured by a balance, from which the ring was suspended. The balance, which could be raised or lowered, was located in the glovebox above the well. The surface tension ring was 3.000-cm. O.D. by 2.901-cm. I.D. The use of such a ring is described in the literature (*2, 3*).

The ring was calibrated by using it to measure the surface tensions of methanol, 2-propyl alcohol, benzene, chloroform, and water at room temperature and of sodium iodide at elevated temperature. Corrections were made for thermal

Table I. Density Measurements of 6.1 Wt. % NaF–17.7 Wt. % NaCl–76.2 Wt. % NaI Eutectic

Temperature, ° C.	Density, Grams/Cc.
541	2.56
550	2.52
551	2.57
552	2.57
554	2.57
566	2.57
575	2.50
580	2.53
599	2.51
600	2.55
601	2.49
625	2.48
631	2.47
651	2.47
674	2.45
700	2.41
751	2.38
800	2.33

Table II. Surface Tension Measurements of 6.1 Wt. % NaF–17.7 Wt. % NaCl–76.2 Wt. % NaI Eutectic

Temperature, ° C.	Surface Tension, Dynes/Cm.
550	102.3
578	101.5
600	100.5
602	97.4
651	98.2
681	94.8
693	95.5
741	91.0

Table III. Viscosity Measurements of 6.1 Wt. % NaF–17.7 Wt. % NaCl–76.2 Wt. % NaI Eutectic

Temperature, ° C.	Kinematic Viscosity, Centistokes	Viscosity, Centipoises
557	0.69	1.76
581	0.68	1.72
601	0.62	1.56
610	0.60	1.50
661	0.545	1.34
679	0.53	1.29
696	0.53	1.29
734	0.525	1.25
755	0.47	1.11
794	0.465	1.10

expansion. Bloom's (*1*) data for sodium iodide were used.

The viscosity was determined by measuring the time required for the liquid between two levels in a calibrated cup to flow through a vertical capillary (0.04458 cm. in diameter and 1.5748 cm. long) in the bottom of the cup. The two levels were determined by two pointed electrodes mounted in the cup. Movement of the liquid level surface past an electrode point was indicated by a sudden change in electrical resistance. To make a run, the cup was filled by dipping it into the crucible; it was then raised above the crucible, and the time interval during which the liquid level fell from the top to the bottom electrode points was measured.

The viscosity capillary cup was calibrated by mathematical computations based on the physical dimensions (*5*). These computations were checked at room temperature using water and mercury.

RESULTS

The density measurements are given in Table I. The following equation fits the data with a standard deviation of 0.020: Density, g./cc. = $3.057 - 0.0009t$ (for t from 540° to 800° C.).

The measured values of density were very nearly equal to the values of density computed using additive volumes of the pure components. As an example, at 800° C. the measured density is 2.33 grams per cc., whereas the value computed from the volumes of the pure components is 2.29 grams per cc.

The surface tension measurements are given in Table II. The following equation fits the data with a standard deviation of 1.25: Surface tension, dynes/cm. = $133.18 - 0.05578t$ (for t from 550° to 750° C.). Values of the surface tensions of the pure salt components are not available in the literature.

The viscosity measurements are given in Table III. The following equation fits the data with a standard deviation of 0.0624: Viscosity, centipoises = $3.2952 - 0.002854t$ (for t from 560° to 800° C.). The measured viscosities of the salt mixture do not vary widely from those of its components. For example, the viscosity at 800° C. is 1.0 cp., whereas, the viscosities of the sodium chloride and sodium iodide components are given (*4*) as 1.556 and 1.10 cp., respectively. The viscosity of sodium fluoride is not reported. The authors believe that the accuracy in the determinations are as follows: for density, ±0.9%; for surface tension, ±1.2%; and for viscosity, ±6%.

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RECEIVED for review October 24, 1967. Accepted January 23, 1968.

Thermal Study of the Glass-Type Transformation of 2-Methylthiophene

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The detailed trend of the heat capacity in the region of 2-methylthiophene transformation has been delineated from 110° to 195° K. by equilibrium adiabatic calorimetry. The temperature dependence is that of a glass-type transition with a continuous inflected curve without a local maximum.

SECOND-ORDER glass-type transitions, which involve no apparent isothermal adsorption of heat, have been reported for 2-methylthiophene (*7*) and 3-methylthiophene (*6*). Since the detailed trend of the heat capacity in the transition region was not delineated in these investigations, a calorimetric sample of 2-methylthiophene was borrowed for a careful investigation of the thermal properties in this region.

The 2-methylthiophene studied was a portion of the standard sample of sulfur compound API-USBM Serial No. 21. In view of the previous characterization of the sample (*7*), further fractional fusions were not studied; however, microchemical analysis indicated 72.9% by weight of carbon and 7.52% of hydrogen (theoretical: C = 73.2; H = 7.37). A sample massing 62.631 grams was distilled into calorimeter W-14, and measurements were made in the Mark III adi-