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Cite as: Journal of Physical and Chemical Reference Data **9**, 791 (1980); <https://doi.org/10.1063/1.555634>  
Published Online: 15 October 2009

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# Molten Salts Data as Reference Standards for Density, Surface Tension Viscosity and Electrical Conductance: $\text{KNO}_3$ and $\text{NaCl}$

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Accuracy estimates for physical property measurements are usually based on somewhat subjective quality judgements, and the difficulties encountered in interpreting accuracy statements in the literature are frequently compounded through lack of details on the methods of measurements, chemical purity, and related experimental aspects. In the present communication we report the results of a Standards Program initiated in 1973 with participating laboratories in Czechoslovakia, German Democratic Republic-DDR, Japan, Norway, Poland, Rumania, and USA. Potassium nitrate (m.  $335^\circ\text{C}$ ) and sodium chloride (m.  $800^\circ\text{C}$ ) were selected as the two reference salts for the properties: density, surface tension, viscosity, and electrical conductance. The results of the measurements have been critically examined, and are reported herewith. It has been possible to resolve some of the difficulties encountered in accuracy estimates through this "round-robin" series of measurements, and to up-grade some of the data-sets to calibration-quality reference standards.

Key words: Calibration-quality standards; density; electrical conductance; molten salts; potassium nitrate; sodium chloride; surface tension; viscosity.

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## 1. Introduction

Accuracy estimates given in the scientific literature are based on somewhat subjective quality judgements, and the difficulties encountered are frequently compounded through the lack of details in methods of measurement, chemical purity, and related experimental aspects. The Molten Salts Standards Program, initiated in 1973 [1]<sup>1</sup>, was undertaken as an approach to resolving some of the difficulties being encountered with reference to accuracy estimates in the NSRDS molten salts data compilations for the properties of density, electrical conductance, viscosity, and surface tension.

<sup>1</sup> Figures in brackets indicate literature references.

For publications in this series see [2-10] and table 1. Participating in the measurements of the Standards Program were:

Czechoslovakia: Dr. Ing. K. Matiasovsky; CSC; Slovak Academy of Sciences, Institute of Inorganic Chemistry, Bratislava.

German Democratic Republic-DDR: Professor Dr. H. H. Emons; Technische Hochschule "Carl Schorlemmer," Leuna-Merseberg, and Bergakademie, Freiberg.

Japan: Professor Tatsuhiko Ejima; Department of Metallurgy, Tohoku University, Sendai.

Norway: Professor Harald A. Øye; Institute of Inorganic Chemistry, Technical University of Norway, Trondheim.

Poland: Professor L. Suski; Polish Academy of Sciences, Institute of Physical Chemistry, Krakow.

Rumania: Dr. Stefania Zuca; Central Institute of Chemistry, Center of Physical Chemistry, Ministry of Industrial Technology, Bucharest.

USA: Dr. D. A. Nissen; Materials Research Laboratories, Sandia Corporation, Albuquerque, New Mexico.

USA: Professor G. J. Janz; Molten Salts Data Center, Cogswell Laboratory, Rensselaer Polytechnic Institute, Troy, New York.

Technical monitors included: France: Professor M. Chemla; Laboratoire d'Electrochimie, Universite de Paris; Netherlands: Professor J. A. A. Ketelaar; Laboratory for Electrochemistry, University of Amsterdam; USA: Dr. Jerry Braunstein, Oak Ridge National Laboratory; Dr. L. H. Gevantman, Office of Standard Reference Data, National Bureau of Standards.

In this program samples of highest purity were distributed to various laboratories for physical properties measurements. Guidelines for sample pre-treatment were normalized. The "raw" data from the measurements of these "round-robin" reference samples, together with experimental details were submitted to the Molten Salts Data Center—RPI for critical analysis and value judgements. The results of the critical assessments thus accomplished, both from viewpoints of experimental limits of accuracy and "calibration-quality" data sets, are reported herewith.

## 2. Symbols, Units and Fundamental Constants

The symbols, units and fundamental constants are in tables 2 and 3, respectively. Unless otherwise noted, all temperatures are in kelvins.

## 3. Treatment of Data

### 3.1. Precision

Estimates of precision were based on a least squares statistical analysis of the data with computer-assisted techniques. The standard error of estimate thus derived is given by the equation:

$$s = \left[ \frac{\sum (\gamma_e - \gamma_c)^2}{n - q} \right]^{1/2} \quad (3.1.1)$$

where  $\gamma_e$  = the experimental value at each temperature,  $\gamma_c$  = the value calculated from the least squares equation at the same temperature as  $\gamma_e$ ,  $n$  = the number of experimental data points, and  $q$  = the number of coefficients in the least squares equation (2 for linear, 3 for quadratic). The precision is taken as the value of the standard error expressed as a percent. As a general guide, about 60% of the results lie within the standard error, 95% within three times the value.

### 3.2. Accuracy

Accuracy estimates are normally based on assessments of experimental details including method of measurements, techniques, analytical characterization of chemicals, and

intercomparisons with results from the same and/or different laboratories. In the present project, the experimental uncertainties due to chemicals-quality were essentially minimized through the distribution of Standards Program Reference Compounds from RPI to the participating laboratories, with recommendations for sample pretreatments. Details are given in section 4. For evaluations of the experimental techniques see section 5.

### 3.3. Percent Departure

The percent departure may be used to compare the results of different investigations. The percent departure is defined as:

Percent departure =

$$\left[ \frac{\text{"reported value"} - \text{"comparison value"}}{\text{"comparison value"}} \right] \cdot 100 \quad (3.3.1)$$

The "reported values" refer to the numerical data of the various studies under consideration; the "comparison value" is from the data set that has been selected as the reference base for this part of the data analyses. The results give a sensitive insight on the accord (or lack of it) either through inspection of two points, such as maximum and minimum departures, or through a point by point inspection for the whole range (best illustrated graphically). Both are used in this work (see sec. 5,6).

## 4. Standards Program Compounds

### 4.1. Selection of KNO<sub>3</sub> and NaCl

Potassium nitrate, KNO<sub>3</sub> (m. 335 °C ± 2 °C) and sodium chloride, NaCl (m. 800 °C ± 2 °C) were selected to meet the need for two salts bracketting the temperature range 350°–1050 °C, i.e., for measurements at moderately high and high temperatures, respectively. These salts are readily dried since they are not strongly hydrated; this is an important consideration since the presence of water, even in trace amounts at high temperatures, is highly undesirable from the viewpoints of stability and "container-chemistry." A further consideration is availability; these salts are commercially available in highest purity and/or can be readily purified to standard reference—quality specifications.

### 4.2. Purity

The analytical purity specifications for Standards-Quality grades of KNO<sub>3</sub> and NaCl and those for these chemicals as Reagent-grade quality are in tables 4 and 5. Commercially these would, generally, correspond to the grades: "highest purity standards quality or ultra-high purity quality," and "analytical reagent chemicals quality," respectively. The samples distributed by the MSDC-RPI to the participating laboratories as the "round-robin" samples for physical property measurements were Standards-Quality.

### 4.3. Pretreatment Recommendations

Because of the quality of the Standards purity specifications, the only pretreatment recommended was to normalize the drying procedure, i.e., moisture removal. Moisture-content analyses were undertaken at RPI [11] with electrochemically assisted Karl-Fischer type analyses, viz,

Aquastat II moisture analyzer (Photovolt Corp.)<sup>2</sup> or modifications thereof—Model 303 moisture analyzer (DuPont). With care, the limits of these analytical techniques were about 10 ppm H<sub>2</sub>O. The results are summarized in table 6. The drying treatments, accordingly, as recommended for the Standards Program samples were:

TABLE 1. NSRDS Molten Salts Data Series

[For Single Salts and their Binary Mixtures relative to the Data for Electrical Conductance, Viscosity, Density and Surface Tension]		
Molten Salts	NSRDS Recommendations	Ref.
Vol. 1 (1968)	Single Salts ( $\kappa, \eta, \rho$ )	[2]
Vol. 2 (1969)	Single Salts ( $\gamma$ )	[3]
Vol. 3 (1972)	Binary Mixtures: Nitrates, Nitrites	[4]
Vol. 4, Pt. 1 (1974)	Binary Mixtures of Fluorides	[5]
Vol. 4, Pt. 2 (1976)	Binary Mixtures of Chlorides	[6]
Vol. 4, Pt. 3 (1977)	Binary Mixtures: Bromides, Iodides	[7]
Vol. 4, Pt. 4 (1979)	Binary Mixtures: Mixed Halides	[8]
Vol. 5, Pt. 1	Binary Mixtures: Mixed Anions other than Nitrates, Nitrites, Halides	[9]
Vol. 5, Pt. 2	Additional Systems	[9]

Single Salt Updates: Some of the NSRDS recommendations advanced in 1968 and 1969 for single salt melts have been revised in the sequels. See the single-salts updates in Volumes 3-5, respectively, for information.

TABLE 2. Symbols and units

Symbol	Physical quantity	Units
A	Pre-exponential factor	as in text
C	Concentration	mol %
E	Energy of activation	cal mol <sup>-1</sup>
t	Temperature	°C,
T	Temperature	K
X	Mole fraction	dimensionless
$\gamma$	Surface tension	mN m <sup>-1</sup>
$\eta$	Viscosity	mN s m <sup>-2</sup>
$\kappa$	Electrical conductance	$\Omega^{-1}$ cm <sup>-1</sup>
$\rho$	Density	g cm <sup>-3</sup>

For conversion between SI and other units:

$$1 \text{ mN s m}^{-2} = 1 \text{ cp} = 1 \text{ mPa s}$$

$$1 \text{ mN m}^{-1} = 1 \text{ dyn cm}^{-1}$$

$$4.184 \text{ J mol}^{-1} = 1 \text{ cal mol}^{-1}$$

<sup>2</sup> Brand names are included for information purposes only and not as endorsements of these products.

$\text{KNO}_3$ : The samples should be dried under vacuo as the temperature is increased gradually to 120 °C and thus maintained for approx. 24 h.

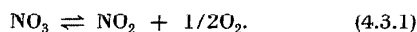
$\text{NaCl}$ : The samples should be dried in vacuo as the temperature is gradually increased to 400°–500 °C as upper limits; test of small aliquots for neutrality (pH) after drying is also recommended.

Limited cross-check measurements were undertaken with Reagent Grade Quality chemicals. Such measurements are of interest to sense out the effects of various additives as trace impurities relative to the thermo-physical properties under consideration in this study.

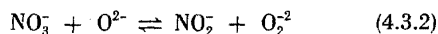
For  $\text{KNO}_3$ , with viscosity as "sensor," the results of measurements from three laboratories with the "single" capillary-type viscometers (designed for accurate molten salts measurements) are in table 7. For  $\text{NaCl}$ , and with electrical conductivity as "sensor," the results from Bucharest are illustrated as a percent departure analysis in figure 1. The departures, in each instance, fall well within the limits of experimental accuracy (viscosity,  $\pm 1.5\%$ ; conductance,  $\pm 0.5\%$ ).

Melting point measurements were undertaken at some of the participating laboratories. The observed melting points will vary with the measurements techniques, the most sensitive being the cryoscopic technique. The results are summarized in table 8. Supporting data, when available, on the measurement method are included. For  $\text{KNO}_3$  the variations are somewhat greater than expected.

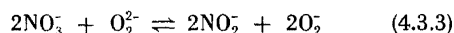
Decomposition temperatures for the  $\text{NO}_3^-$  are not well established. Onset of appreciable decomposition of molten  $\text{KNO}_3$  has been noted by Bartholomew [193] and Kerridge [194]. Brooker [195] has recently reinvestigated this problem with laser-Raman using the  $^{18}\text{O}^{16}\text{O}$  exchange as spectroscopic sensor. The results show that the  $\text{NO}_3^-$  is kinetically stable in molten  $\text{KNO}_3$  for long periods below the decomposition temperature; onset of decomposition at 400 °C is confirmed, and at 470 °C, this decomposition is appreciable. Direct thermal decomposition leads to nitrite and oxygen:



Kerridge [194] notes that although the forward reaction of (4.3.1) may be very fast, the reverse process appears to be very slow. This observation receives additional support from the study of Brooker [195]. Zambonin et al. [196,197] have suggested that glass in contact with molten  $\text{KNO}_3$  may be the source of oxide ions contributing to decomposition processes:



and



By the exchange rate criterion, it appears that the reverse reactions for the latter two processes must also be slow at least for situations where the oxide apparently comes from the silicate species.

From the preceding it is clear that some irreproducibility in physical properties data of molten  $\text{KNO}_3$  are to be expected if the measurements are extended above 400 °C and the measured properties are sensitive to nitrite as decomposition product. Due cognizance of this was taken in evaluating the results of the measurements that follow.

## 5. Experimental Accuracies

### 5.1. Density Techniques

For summaries of molten salts density techniques, see [4,5,6,9] and for percent applicability, see [4–10]. Density measurements were undertaken by Matiasovsky et al. [12] and Ejima et al. [13] with Standards-Quality  $\text{KNO}_3$ , using the Archimedean and maximum bubble pressure techniques, respectively. The latter has seen approx. 10% application for fused salt density determinations; the density and surface tension may be determined simultaneously for the same series of experiments, e.g., for density there is a linear relationship between the maximum rise of the manometer  $h$ , and the depth of the maximum bubble tip,  $z$  and it follows that:

$$h = (\rho_s/\rho_m)z + h_0 \quad (5.1.1)$$

where  $\rho_m$ ,  $\rho_s$  are the densities of the standard liquid in the manometer and molten  $\text{KNO}_3$ , and  $h_0$ , a constant. Thus, from the determination of the slope of the  $h$  vs  $z$  graph, the density of the salt is calculated, simply, from the expression:

$$\rho_s = k\rho_m \quad (5.1.2)$$

where  $k$  is the experimentally determined slope.

In figure 2 is shown a comparison of the results, using the NSRDS density recommendations [14] as the reference. The uncertainty limits of the Archimedean technique are  $\pm 0.2\%$ ; the uncertainty limits of the maximum bubble densities appear approximately  $\pm 0.5\%$ , i.e., the accuracy of this method is somewhat less.

The evaluation of the  $\text{NaCl}$  densities (Standards-Quality  $\text{NaCl}$ ) with the Archimedean technique [12] is illustrated in figure 3, as a percent departure relative to the NSRDS recommendations advanced in 1968 [14]. The limits of uncertainty assigned to the Archimedean technique in this higher temperature range are  $\pm 0.5\%$ , and within these limits the two data sets appear substantially in agreement.

### 5.2. Surface Tension Techniques

For summaries of molten salts surface tension techniques, see [3–6,9] and for percent applicability, see [4–10]. More than 80% of the measurements have been by the maximum bubble pressure technique; of the remaining methods (approx. 7), two variations of the detachment principle, the Wilhelmy slide plate, and the flat-pin have seen widest use. Measurements were undertaken in the molten salt Standards Program by Nissen [15], Ejima and Yamamura [16], and Suski and Tomczyk [17] with the maximum bubble pressure technique, and by Zuca and Terzi [18], with the flat-pin

TABLE 3. Fundamental constants<sup>a</sup>

Symbol	Name	Values <sup>b</sup>
N	Avogadro constant	$6.022045 (31) \times 10^{23} \text{ mol}^{-1}$
F	Faraday constant	$9.648456 (27) \times 10^4 \text{ C mol}^{-1}$
e	Electron charge	$1.6021892 (46) \times 10^{-19} \text{ C}$
R	Gas constant	$8.3143 \pm 0.0012 \text{ J K}^{-1} \text{ mol}^{-1}$ $1.98716 \pm 0.00029 \text{ cal K}^{-1} \text{ mol}^{-1}$

(a) Fundamental constants from: CODATA Bulletin No. 11 (Dec. 1973)

(b) In each case the digits in parentheses following a numerical value represent the standard deviation of that value in the decimal places indicated for its final digits

Table 4. Comparisons of purity specifications for Standards-Quality and Reagent Grade Quality  $\text{KNO}_3$

$\text{KNO}_3$ (m. $335^\circ\text{C} \pm 2^\circ\text{C}$ )
A: Standards Quality $\text{KNO}_3$
(a) purity assay: 99.999%
(b) pH (25°C, 5% sol'n): 5.6
(c) impurities (tolerance range, ppm):
nitrite, <1; Fe, 1-5; Ca, 1-5; Mg, 1-5;
Al, 0.5-2.0; Cu, 0.5-2.0; Na, 0.5-2.1
B: Reagent Grade Quality $\text{KNO}_3$
(a) purity assay: not reported
(b) pH range (25°C, 5% sol'n): 4.5-8.5
(c) impurities (tolerance limits, ppm):
nitrite, $\leq 10$ ; Fe, $\leq 3$ ; Ca, Mg, and $\text{R}_2\text{O}_3$ ppt,
$\leq 100$ ; Na, $\leq 50$ ; heavy metals (as Pb), $\leq 5$ ;
sulfate, $\leq 30$ ; phosphate, $\leq 5$ ; $\text{IO}_3^-$ , $\leq 5$ ; Cl, $\leq 20$ ;
insoluble matter, $\leq 50$

Table 5. Comparisons of purity specifications for Standards-Quality and Reagent Grade Quality NaCl

NaCl (m. $800^\circ\text{C} \pm 2^\circ\text{C}$ )
A: Standard Quality NaCl:
(a) purity assay: 99.999%
(b) pH range (25°C, 5% sol'n): $\sim 7$
(c) impurities (tolerance limits, ppm): As, <0.04; Br,
<2.3; I, <0.1; N, <1; phosphate, <0.4; sulfate, <1;
Al, 0.08; Ba, <5; Bi, <0.01; Cd, 4; Ca <0.5; Cr,
<0.002; Co, <0.01; Cu, <0.01; Fe, 0.02; Pb, 0.02;
Li, <0.5; Mg, 1; Mn, 5; Hg, <0.001; Mo, <0.01;
Ni, <0.02; K, 4; Ag, 0.001; Sr, 5; Sn, <0.01; Ti,
0.01; V, <0.002; Zn, <0.2; Zr, <0.02; water insoluble
matter, <3

Table 5. Comparisons of purity specifications for Standards-Quality and Reagent Grade Quality NaCl—Continued

NaCl (m. 800°C $\pm$ 2°C)
<b>B: Reagent Grade Quality NaCl:</b>
(a) purity assay: 99.6%
(b) pH range (25°C, 5% solution): $\sim$ 7
(c) impurities (tolerance limits, ppm): Br, <50; I, <20; N, <10; chlorate and nitrate, <30; phosphate, $\sim$ 5; sulfate, $\sim$ 10; Ba, $\sim$ 10, [Ca, Mg, and $\text{Re}_2\text{O}_3$ ppt], <30; Fe, $\sim$ 2; heavy metals (as Pb), $\sim$ 2; K, $\sim$ 20; water insoluble matter, $\sim$ 30

detachment technique. The results, as percent departure relative to the 1969 NSRDS recommended data base, are illustrated in figures 4 and 5, respectively.

The results of Nissen and Ejima confirm that the maximum bubble pressure technique is capable of surface tension measurements to an accuracy of better than  $\pm 1\%$ ; details of the experimental assemblies have been described elsewhere, [19,20] and it is sufficient to note that careful attention must be paid to the capillary tip for precise and accurate results, and that the measurements should be under conditions justifying the use of the Schrodinger equation for surface tension [21,22]. In the work of Suski and Tomczyk, the Cuny-Wolf variant [23] of this technique was used for molten salts as described elsewhere [24]; in this approach two capillaries with differing radii and differing depths of immersion are used simultaneously, and one thereby bypasses the need to determine the tip immersion depth. The results fall well within the accuracy limits, and the applicability of the Cuny-Wolf variant of the maximum bubble pressure technique thus receives support. The accuracy limits of the flat-pin detachment measurements are estimated to be  $\pm 1.5\%$ . With precisely determined pin dimensions (e.g., pin radius, 0.05225 cm), it has been shown by Zuca and Terzi that the surface tensions calculated either by the detachment equation of Janz and Lorenz [25] or Lillebuen [26] are basically in agreement. The values from the Lillebuen equation are approx. 0.5% lower, and not 10% as reported elsewhere [27]. It is clear that the pin radius must be very accurately determined for fullest utilization of the Lillebuen equation.

Two laboratories extended measurements to the higher temperature range; the results reported by Suski and Zuca, with Standards Quality NaCl, are illustrated in figure 5. Inspection shows that the results fall within the overall limits of accuracy ( $\pm 1.5\%$ ); the uncertainty limits assigned to the maximum bubble technique as part of the earlier NSRDS recommendations, i.e.,  $\pm 0.1\%$ ; appear overly optimistic and must be revised. Some questions are also raised on the temperature dependence of the NaCl surface tensions values; resolution of this aspect is not possible without additional data.

### 5.3. Electrical Conductance Techniques

Experimental aspects of molten salts cell design and measurement techniques are discussed in [4-7,9]. The ac bridge technique is almost exclusively used ( $\sim 97\%$ ). For cell calibration techniques, the critical assessment by Janz and Tomkins [7,28] is informative. The specific conductivity data for aqueous KCl are the generally accepted calibration standards, with the three (Jones and Bradshaw) demal concentrations as the standard reference points. In addition, four fitting-equations have been advanced, the Lind-Zwolenik-Fuoss, the Fuoss-Hsia, the Justice, and the Ebeling equations [see 28]; the use of these by-passes the more laborious procedure of exactly matching the weights of KCl and water to attain the very precisely specified demal concentrations. A significant result of the Ebeling study is the extension of the fitting-equation technique up to 1 molar concentration limit.

The importance of properly designed cells was stressed by Jones and Bollinger [29]; if the filling arms and electrode leads are spaced too closely, a cell calibration anomaly is observed, namely that the "cell constant" is apparently not really constant but varies with the resistance measured; this anomaly (termed the "Parker Effect" to distinguish it from polarization) can be corrected by cell re-design.

A further point of concern relative to the use of the specific conductivity of aqueous KCl as calibration standards is an error in a 1970 textbook of physical chemistry [30]. The composition of the 1 demal KCl solution is given as 74.1352 g/1000 g of solution; the accepted Jones and Bradshaw value is 71.1352 g. Post-1970 conductance data should be double checked for this as possible error source.

The problem of polarization errors in conductance measurements has been re-examined by Tomkins, Janz and Andalaft [31]. With ambient temperature electrolyte solutions, and precision type conductance cell/bridge techniques, it was shown that the uncorrected values for specific conductivity may be as much as 1.2% and 0.7% too low at 3 kHz and 10 kHz frequencies, respectively, relative to the polarization free value (by extrapolation to  $f = \infty$ ). Both the  $1/\sqrt{f}$  and  $1/f$  graphical extrapolation methods

appear valid procedures for the polarization correction. The magnitude of this correction must be assessed empirically in each study, since it is a function of cell constant, the electrolyte specific conductivity, and the bridge techniques (signal frequencies; applied potential, etc.). Because of the relatively high specific conductivities of molten salts (e.g., molten  $\text{KNO}_3$ ,  $\kappa \sim 1.0$ ; molten  $\text{NaCl}$ ,  $\kappa \sim 4.0$ ; aqueous 1M  $\text{KCl}$ ,  $\sim 0.11$ ), and the correspondingly larger cell constants (molten salts, 200–2500  $\text{cm}^{-1}$ ; aqueous 1N  $\text{KCl}$ ,  $\sim 16 \text{ cm}^{-1}$ ), higher signal frequencies are generally used in molten salts conductance measurements so as to minimize polarization errors. The data-sets of the Standards Program were examined from the preceding viewpoints; i.e. cell design (Parker Effects), and the polarization correction. The results may be summarized as follows:

Figures 6 and 7 illustrate comparisons of the data sets for measurements with Standards-Quality molten  $\text{KNO}_3$  and  $\text{NaCl}$ , respectively, relative to the NSRDS recommended values as reference base; some details of the measurement techniques are summarized in table 9. The Ketelaar-Maenaut studies, although pre-Standards Program, are included for completeness.

Relative to the polarization corrections, Ketelaar and Maenaut [32] noted that 10 KHz was satisfactory with a cell constant of  $\sim 2500 \text{ cm}^{-1}$ ; the uncorrected conductivities were in error by as much as 2% at frequencies of 1 kHz. Zuca and Olteanu [33] found 17 kHz satisfactory (cell constant,  $\sim 520 \text{ cm}^{-1}$ ); at 1.7 kHz the conductivities were uniformly  $\sim 1.5\%$

lower. Ejima, Shimakage and Sato [34] reported that the polarization correction was  $\sim 4\%$  for measurements at 1.1 kHz. Emons and Brautigam [35] found 20 kHz satisfactory. The cell of Suski, Gadek, and Stachowicz [36,37] was designed for relative measurements, i.e., various compositions of the same multi-component systems. Suski et al. observed a pronounced "Parker Effect" in determining the cell constant with a range of calibration solutions (concentrated aqueous electrolytes). Correcting for this by an empirical curve-fitting technique leads to a limited improvement in accuracy (see Suski et al. A and B in figs. 6 and 7, respectively). The consistently lower values of the uncorrected conductivities at higher temperatures (figs. 6, 7, Suski, B) are as would be predicted for significant polarization errors in this data set (cell constant,  $\sim 37$  frequency,  $\sim 1 \text{ kHz}$ ). The results, nevertheless, are qualitatively in accord with the observations that the NSRDS reference data set for  $\text{NaCl}$  (based on the earlier measurements by Van Artsdalen and Yaffe [2.6.38]) is as much as 6% too high at  $\sim 1000^\circ\text{C}$ . The error, first recognized by Ketelaar and Maenaut in 1972, is due to onset of parasitic conductance paths through the walls of the quartz dipping capillaries of the conductance cells. The close agreements of the data sets of Ejima, Emons, Zuca, and Ketelaar indicate that the quartz type dipping capillary conductance cells, nevertheless, may be used with high accuracy in this temperature range with due attention to cell design [35,39,40] and polarization corrections.

Table 6. Drying treatments and moisture contents of  $\text{KNO}_3$  and  $\text{NaCl}$

Drying Treatment			H <sub>2</sub> O content
Temp.	Time	Atmosphere	(ppm)
Sample: KNO <sub>3</sub> : Standards Quality; ~ 50 gm			
(a) 25°C	fresh from bottle		~ 200
(b) 135°C	60 hrs.	H <sub>2</sub> (1 atm)	60
(c) 25°C	24 hrs.	10 <sup>-4</sup> mm (vacuo)	84
(d) 25°C	24 hrs.	10 <sup>-6</sup> mm (vacuo)	< 26
(e) 25°C	48 hrs.	10 <sup>-6</sup> mm (vacuo)	~ 13

Sample: NaCl: Standards Quality; ~ 50 gm			
(a) 25°C	fresh from bottle		~ 40
(b) 25°C	from open bottle, 24 hrs.		~ 300
(c) 500°C	24 hrs.	10 <sup>-6</sup> vacuo	~ 20
(d) 25°C	first dried as in (c) above, and then stored 48 hrs. in "dry chamber"		~ 40

Table 7. Comparison of viscosities of Standards-Quality and Reagent Grade  $\text{KI}$

Investigation	Technique	$\text{KNO}_3$	Viscosity (cp)	
			$361^\circ\text{C}$	$376^\circ\text{C}$
Zuca and Costin [47]	capillary	Standards-Quality	2.578	2.379
van Os and Ketelaar [50]	capillary	Reagent Grade	2.585	2.380
Timidei, Lederman, and Janz [49]	capillary	Reagent Grade	2.575	2.376



Table 8 Comparisons of Melting Point Measurements

Investigation	Measurement	KNO <sub>3</sub> : $t_m$ (°C)	NaCl: $t_m$ (°C)
Standards Quality Grade		$335^b \pm 2^\circ\text{C}$	$800^\circ \pm 2^\circ\text{C}$
Janz, et al. [11]	DSC-II <sup>a</sup>	335.0°C <sup>b</sup> 334.6°C <sup>c</sup> 334.1°C <sup>d</sup>	
Matiasovsky, Votova [44]	-	334.7°C 334.1°C	800.6°C
Ejima, Yoko [43]	-	333.2°C	799°C
Zuca, et al. [33]	-	337°C	800°C
Emons, Brautigam [35]	cryoscopy	$337 \pm 0.2^\circ\text{C}$	$800^\circ \pm 0.4^\circ\text{C}$

(a) DSC-II, differential scanning calorimetry

(b) sample, as received from bottle

(c) sample, after recommended pretreatment

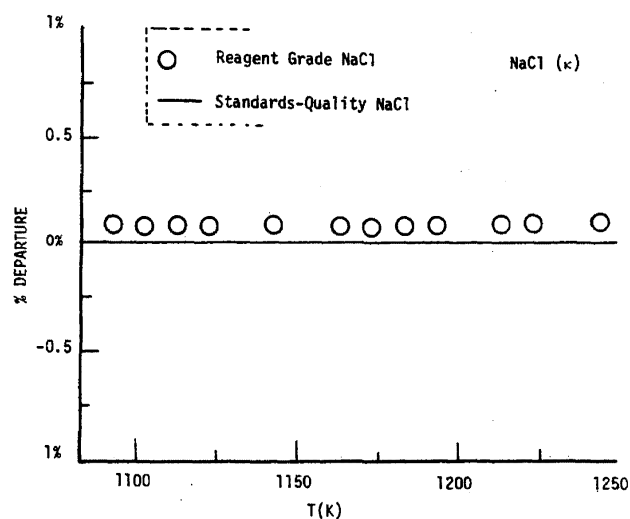
(d) sample, heated at 450°C, 24 hrs., under dynamic vacuum ( $10^{-4}$  mm Hg)

Figure 1 Comparison of the specific conductance of Standards-Quality and Reagent Grade NaCl

The specific conductance of Standards-Quality NaCl was used as reference base for the Percent Departure analysis of the reagent grade NaCl [33]

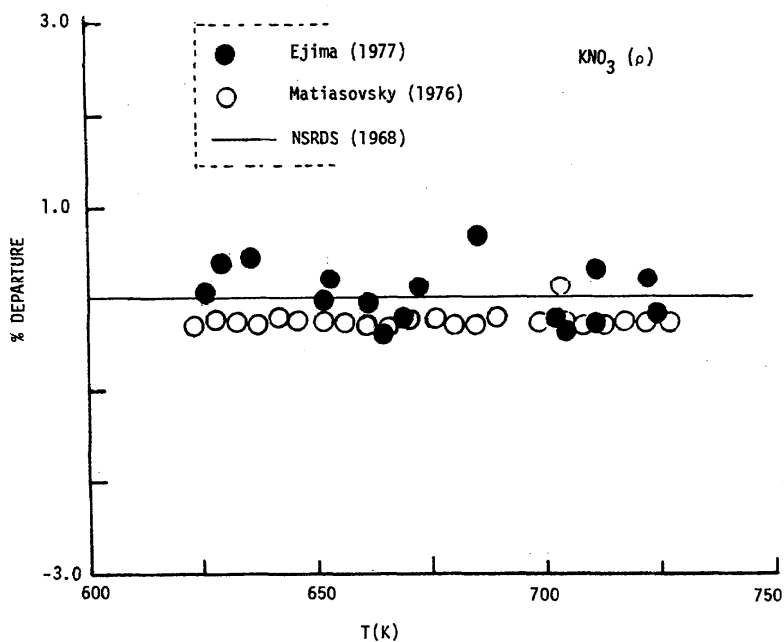


Figure 2 Comparison of  $\text{KNO}_3$  density data from the Standards Program with the NSRDS (1968) recommendations

A Percent Departure analysis is shown with the NSRDS (1968) recommendation [Bloom et. al data base]:  $\rho = 2.315 - 7.29 \times 10^{-4}T$ , as comparison reference.

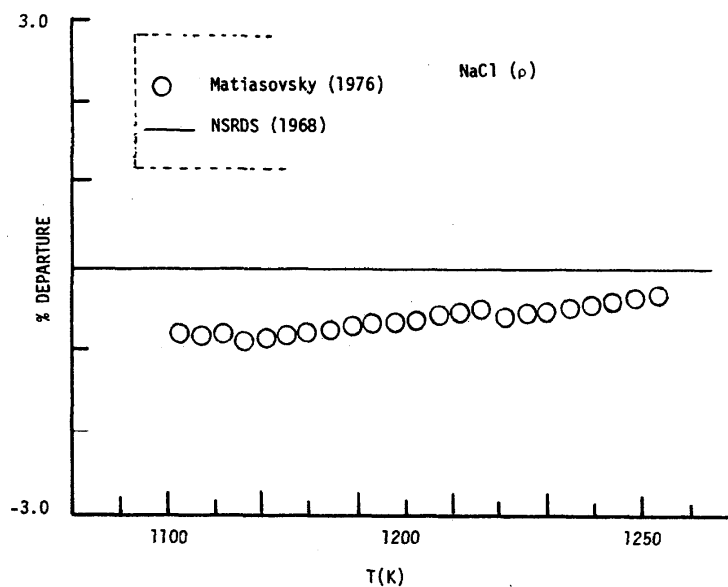


Figure 3 Comparison of  $\text{NaCl}$  density data from the Standards Program with the NSRDS (1968) recommendation.

A Percent Departure analysis is illustrated, with the NSRDS (1968) recommendation [Van Artsdalen and Yaffe data base]:  $\rho = 2.1393 - 5.430 \times 10^{-4}T$ , as comparison reference.

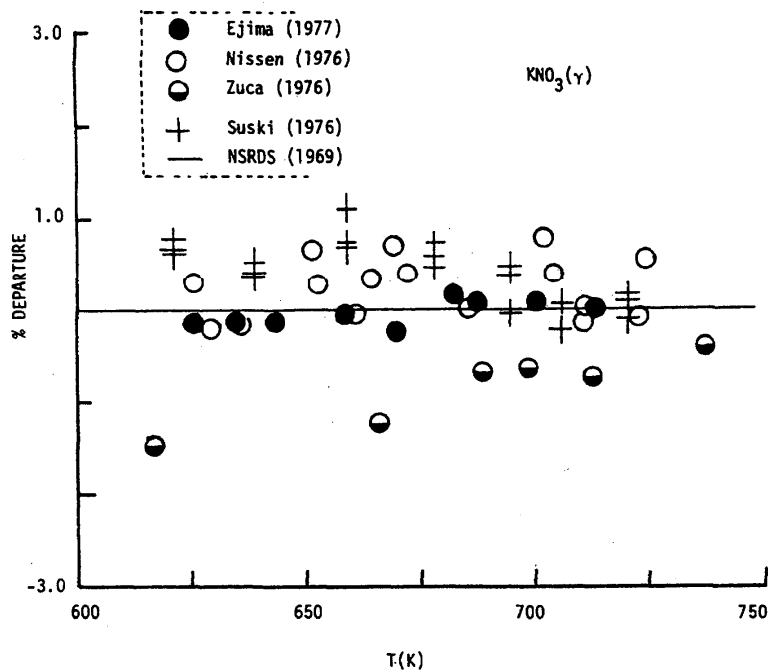


Figure 4 Comparison of  $\text{KNO}_3$  surface tension data from the Standards Program with the NSRDS (1969) recommendation.

A Percent Departure analysis is illustrated with the NSRDS (1969) recommendation [Janz and Lorenz data base]:  $\gamma = 148.4337 - 49.7486 \times 10^{-3}T - 18.6207 \times 10^{-6}T^2$  as comparison reference.

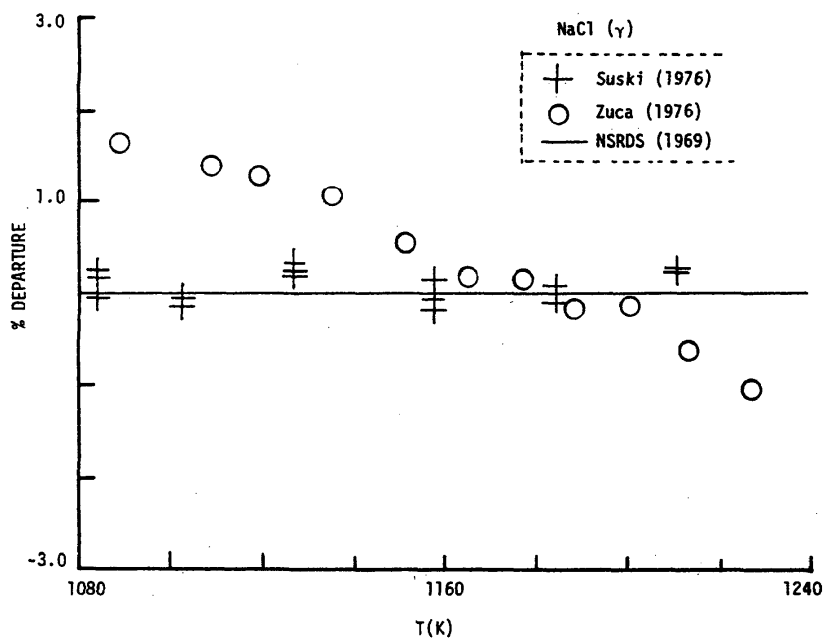


Figure 5 Comparison of  $\text{NaCl}$  surface tension data from the Standards Program with the NSRDS (1969) recommendation

A Percent Departure analysis is illustrated with the NSRDS (1969) recommendation [Sokolova and Voskresenskaya data base]:  $\gamma = 191.1 - 71.90 \times 10^{-3}T$ , as comparison reference.

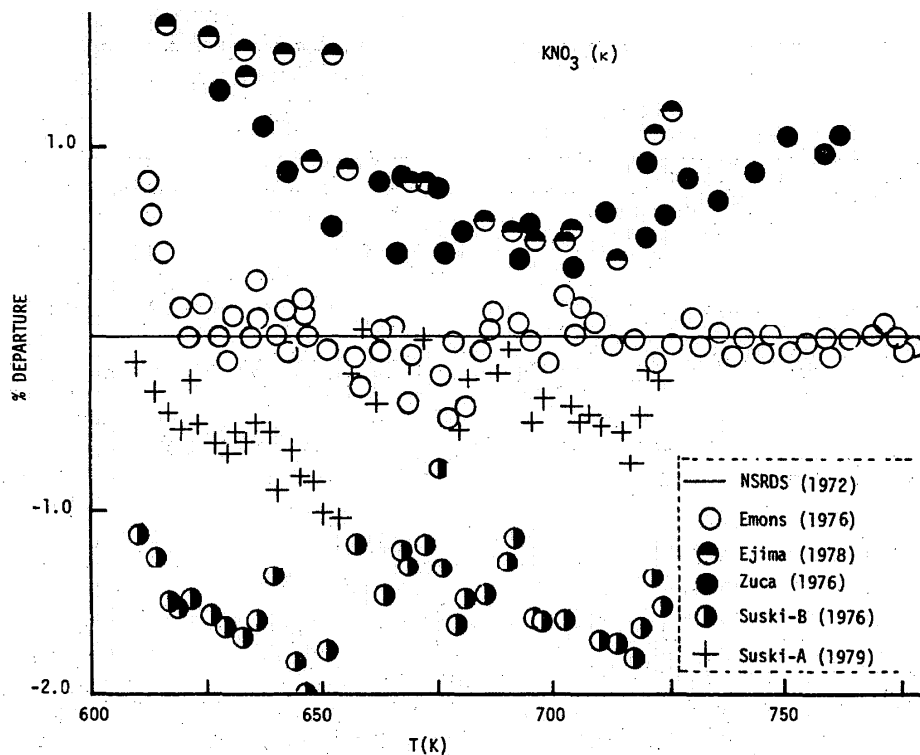


Figure 6 Comparison of  $\text{KNO}_3$  specific conductance data from the Standards Program with the NSRDS (1972) recommendation  
A Percent Departure analysis is illustrated with the NSRDS (1972) recommendation [Robins and Braunstein data base]:  $\kappa = -2.1230 + 5.7252 \times 10^{-3}T - 2.0221 \times 10^{-6}T^2$ , as comparison reference.

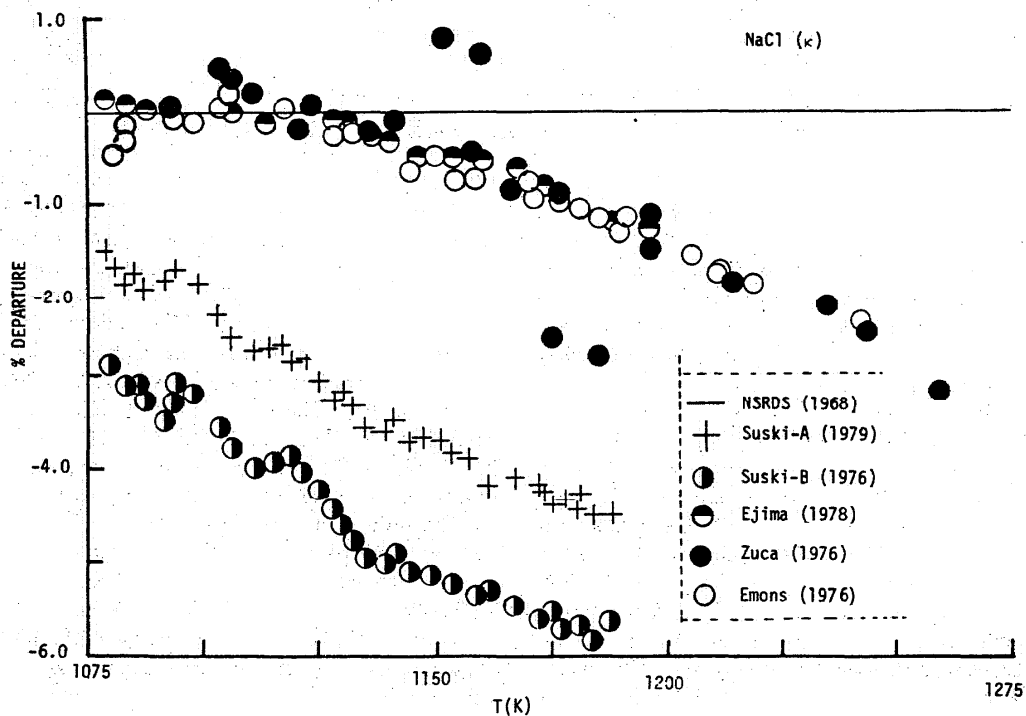


Figure 7 Comparison of  $\text{NaCl}$  specific conductance data from the Standards Program with the NSRDS (1968) recommendation  
A Percent Departure analysis is illustrated with the NSRDS (1968) recommendation [van Artsdalen and Yaffe data base]:  $\kappa = -2.4975 + 8.0431 \times 10^{-3}T - 2.227 \times 10^{-6}T^2$ , as comparison reference.

Table 9 Molten salts conductance techniques (Standards Program)

Investigation Details	Ejima [34]	Emons [35]	Suski [36]	Zuca [33]	Ketelaar [32] (d)
Bridge	hi-precision ac	hi-precision ac	hi-precision ac	hi-precision ac	hi-precision ac
f (kHz)	1 - 10	1 - 25	1.0	1 - 17	1 - 10
Extrap <sup>n</sup>	(1/f <sup>2</sup> )	-	-	-	-
R used at	f = ∞	f = 20 kHz	f = 1.0 kHz	f = 17 kHz	f = 10 kHz
Cell constant (cm <sup>-1</sup> )	227	188	~ 133 (b)	500 - 700	1500; 2500
R <sub>obs'd</sub>					
KNO <sub>3</sub>	~ 300 Ω	~ 150 Ω	~ 145 Ω	~ 700 Ω	-
NaCl	~ 60 Ω	~ 150 Ω	~ 37 Ω	~ 170 Ω	~ 700
Cell Design	[34]	(a)	[36,37]	[33]	[32]
quartz capillary	✓	✓	✓	✓	✓
Dipping type	✓	✓	-	-	-
U design	-	-	✓	✓	✓
electrodes	Pt	Pt	(c)	Pt	Pt

(a) cell design similar in principle to van Artsdalen and Yaffe [38]

(b) cell constant not constant but varied resistance measured

(c) for KNO<sub>3</sub>, Pt; for NaCl, Mo

(d) measurements were completed pre-Standards Program project; NaCl was Analytical Reagent Grade Quality

#### 5.4. Viscosity

For experimental techniques see [4-6,9]. Within the molten salts Standards Program intercomparisons of the capillary and torsional pendulum techniques have been possible [i.e., the two salient molten salts viscosity measurements methods]. The "round-robin" Standards Quality KNO<sub>3</sub> and NaCl samples were used at each participating laboratory to normalize sample quality; thus attention could be focussed on technique error analysis. For the comparisons, the 1968 NSRDS recommended data set was selected as the reference base. For KNO<sub>3</sub>, this data set is based on the results of Dantuma [41] while for NaCl, on the results of Murgulescu and Zuca [42]. The Dantuma KNO<sub>3</sub> data and the Murgulescu and Zuca NaCl data have been widely cited in scientific literature and are thus a practical "touch-stones" for the present intercomparisons. The results are thus illustrated in figures 8 and 9 for the measurements under the Standards Program completed by Ejima and Yoko [43], Matiasovsky and Votova [44], Suski, Galka, and Stachowicz [45], Tørklep and Øye [46], and Zuca, Costin, and Borcan [47,48].

Capillary technique: The results for Standards Quality molten KNO<sub>3</sub> by Zuca and Costin [47] and Suski, Galka and Stachowicz [45] are illustrated again in figure 10. Also illustrated are the results by Timidei, Lederman, and Janz [49] and van Os and Ketelaar [50], both utilizing with new designs of closed-type molten salts capillary viscometers. The latter two investigations were pre-Standards Program; the KNO<sub>3</sub> was a Reagent Grade quality (refer: tables 4,7). The results are in close agreement (<0.2%) with Standards Quality KNO<sub>3</sub>. The viscosity is, apparently, not sensitive to the trace impurities (mainly nitrite) providing the KNO<sub>3</sub> is carefully dried. Zuca and Costin used the Ubbelohde-Bingam capillary-type viscometer [51], while Suski et al., used a modified Ostwald technique for relatively short capillaries [52]. The accuracy limits of the classical capillary-type viscometers [49-51] in this temperature range (340°-500 °C) thus appears to be ±1.0% for routine measurements, and this can be improved to ±0.25% with great care. By comparison, the greater scatter of the data from the short capillary-type viscometer, and the differences with the preceding three data sets, fix the accuracy limits of this modification to be about ±2%, i.e., somewhat larger.

For molten NaCl, capillary viscosity measurements with standards Quality samples have also been completed by Zuca and Costin [47] and Suski et al. [45] using the classical single capillary and the modified technique with short capillary, respectively. The results together with those from the torsional pendulum techniques, are illustrated in figure 9. It is sufficient to note that with the classical single capillary technique, the results are in agreement (within experimental limits of accuracies) with the data of the torsional pendulum techniques. It follows that the capillary technique is applicable, with good accuracy, in the higher temperature range (800°–1000 °C).

Additional support for this is found in the work of van Os and Ketelaar with molten KCl [50]. It was observed that the viscosities were in good agreement with data from the torsional pendulum measurements of Brockner et al. [53] and Zuca and Borcan [48]. In the presence of water (as impurity) etching of the quartz capillary is observed; however no etching was observed with carefully dehydrated salts. The accuracy limits of the classical capillary technique at molten NaCl temperatures (800°–1000°) thus may be fixed at  $\sim \pm 1.5\%$ . The short capillary technique does not appear satisfactory for this temperature range; the data-set falls close to torsional measurements in which a pendulum design was used that induced secondary flow effects (see later, this part).

**Torsional technique:** The torsional technique has been and is the most widely used technique in molten salts viscometry. Advantages are the relatively small temperature "flat-zone" required, and the wider choice of container materials than possible for the capillary methods. A disadvantage would appear to be the mathematical complexity of the theory in the derivation of solutions leading to working equations for absolute viscosity measurements; the advent of computer-assisted techniques has eased this consideration. Some of the practical features relative to accuracy of measurements have been re-examined as part of the Standards Program; particularly design features re: torsion wire; torsion pendulum; immersed spherical oscillator; immersed cylindrical oscillator; on-line computer assisted data acquisition techniques; and advances in mathematical theory/computational techniques. Some of the observations are as follows.

Relative to torsion wires, the "Kestin" alloy (Pt-8% W) appears superior [54,55]. This alloy has a low internal friction and a highly stable elastic constant; these properties are important for minimizing errors due to non-ideality of such wires. It, accordingly, was used for the Standards Program measurements at Trondheim, Bucharest, and Bratislava. At Sendai, molybdenum was used (conventional practice). It is difficult to ascribe the fraction of the total error due to the properties of the torsion wire as error sources; it is estimated [45,56,57] that the Kestin alloy significantly reduces the error source.

The departures of the results of Ejima et al. from those of Tørklep and Øye for NaCl (see fig. 9) may be, in large part, due to this factor. Change of the torsion wire to the Kestin alloy, and re-design of the pendulum to remove an inertia mass (in the hot zone) eliminated the factors apparently contributing to the very significant errors in the NaCl

temperature range at Bratislava; see fig. 9 (800°,  $\sim 40\%$  too high, 1000°C,  $\sim 15\%$  too high).

The design of the torsion initiators ranged from the classical manual techniques (Bucharest [42,59], Sendai [43]), to electrically assisted techniques (Trondheim [53,57], Bratislava [58]). Pendulum designs ranged from those without damping weights [48,59] to various inertia masses [43,53,57,58,60]. The large differences in the earlier and later data sets for NaCl from Bratislava [4] (see fig. 9, 1976; 1979) may be attributed, in large part, to the pendulum design. In the earlier designs, the pendulum had a disc-like inertia mass in the hot zone of the furnace, much as in [58]. The possible "sail-like" effect of this damping design undoubtedly imposed additional pendulum movements (leading to a precession of the axis of rotation), and this would be realized as secondary flow effects in the damping action of the torsional oscillation by the fluid(s). As noted earlier, such error sources lead to observed viscosities that are too high.

It is sufficient to note here, that while the torsional techniques may be in good accord at molten KNO<sub>3</sub> temperatures, one cannot necessarily assume that this agreement will carry through to the molten NaCl temperature range (800°–1000 °C). The Bratislava torsional assembly with the weighted pendulum and molybdenum torsion wire which gave the greatly discrepant results for NaCl (fig. 9) nevertheless yielded results for KNO<sub>3</sub> that were in general accord with the capillary technique within the limits of accuracy. This generalization also applies to other viscosity techniques. For example, with the Krakow short capillary technique, the results for molten KNO<sub>3</sub> are basically in moderately good agreement, i.e.,  $\sim \pm 4\%$  (fig. 7), but at NaCl temperatures, the results differ seriously from the correct values (fig. 9, Øye and Tørklep).

In addition to the preceding, experimental errors in the torsional technique can be attributed to sample quality, irregularities in the shape of the immersed oscillator, and the computational approach. In the present program, there has been an opportunity to re-examine these critically.

At Bucharest [48,60] a careful error analysis of the immersed sphere technique and a re-examination of the use of the Verschaffelt Equations in the computations was undertaken. A computational procedure based on the solution of two Verschaffelt equations simultaneously (TESS computation procedure) has been found to be superior to the more conventional approach (requiring experimental period measurement, i.e., the EPM computation procedure). In the EPM method, the usual experimental approach is the use of a pendulum weighted with discs for greater pendulum stability (through an increased moment of inertia); this practice introduces a possible error source (as additional pendulum movements), and thus, departures from true torsional oscillations, as already noted. With the TESS computational approach, the simplest pendulum may be used in which damping weights are not used; most of the mass is thus concentrated in the immersed sphere. This was the design used in Bucharest [42,48,59]. A further significant result of this analysis shows that the absolute limits of accuracy of the torsional immersed

sphere technique are  $\pm 2\%$  at the very best. The determining factor to the absolute limits appears to be the accuracy to which the "sphericity" of the sphere can be established. Thus for a sphere of radius 1.022 cm, an uncertainty in radius of  $\pm 0.004$  cm imposes a limit of accuracy of  $\pm 2-3\%$  for the torsional technique. In molten salts measurements, the noble metals (platinum, gold, and alloys of these) are generally used for such spheres; the inherent softness of these metals limits the spherical machineability tolerances to approx.  $\pm 4 \times 10^{-3}$  cm. In Trondheim and Bucharest measurements to these limits of accuracy have been achieved with the torsional techniques (immersed sphere) [8,56]. A comparison of the viscosity data from the torsional technique as percent departure relative to capillary technique results, both with Standards Quality  $\text{KNO}_3$ , from measurements completed at Bucharest [60] is illustrated in figure 11.

The preceding considerations of absolute limits, led to the change from the sphere to the immersed cylinder technique at Trondheim, i.e., cylinders can be machined to more exact tolerances than spherical shapes [46,57]. Absolute viscosities are calculated from two working equations (imaginary and real parts) derived by Tørklep and Øye from the general solution for cylindrical shapes given by Azpeitia and Newell [61]. This computational advance enables fullest utilization of the more exact tolerance limits of cylinders. For a full description of the Trondheim viscometer, re-designed for oscillating cylindrical shapes, together with the computer coupled system and ancillary software programs for on-line

viscosity computations see [57]. This viscometer is operated at two levels of accuracy. The accuracies associated with the optimized procedures are 0.1% for water and 0.2% for reference molten salts (68% confidence level); the accuracy limits for routine measurements are, by comparison, 0.5-1%.

Attention is directed to the work at Grouvel [62]. It is shown that the mathematically exact first order solution to the theory of the oscillating hollow cylinder (cup) viscometer is adequate for very precise measurements under conditions where the effects of secondary flow have been successfully eliminated (e.g., due to additional pendulum motions and related effects, see earlier, this discussion). The working equations of Roscoe [63] and Beckwith and Newell [64] will yield viscosities with an accuracy of  $\pm 1.0\%$  under such conditions. The equations by Shvidovskii [65], Reeves and Janz [66] and Hopkins [67] are much less exact.

With reference to the method of calculating viscosities for the oscillating cylindrical method after Roscoe [63] and the working equations of Thresh [68], Ejima has noted an error in the expansion of the  $a_0$  term; re-derivation of the working equation shows that the coefficient of the 2nd term in this series expansion is not 1/2 but 3/2. With this correction, the series convergence is markedly improved.

The Standards Program data sets (illustrated in figs. 8 and 9) demonstrate some of the difficulties encountered in extending such measurements from moderately high temperatures ( $360^\circ\text{--}500^\circ\text{C}$ ) to the higher temperature ranges ( $800\text{--}1000^\circ\text{C}$ ).

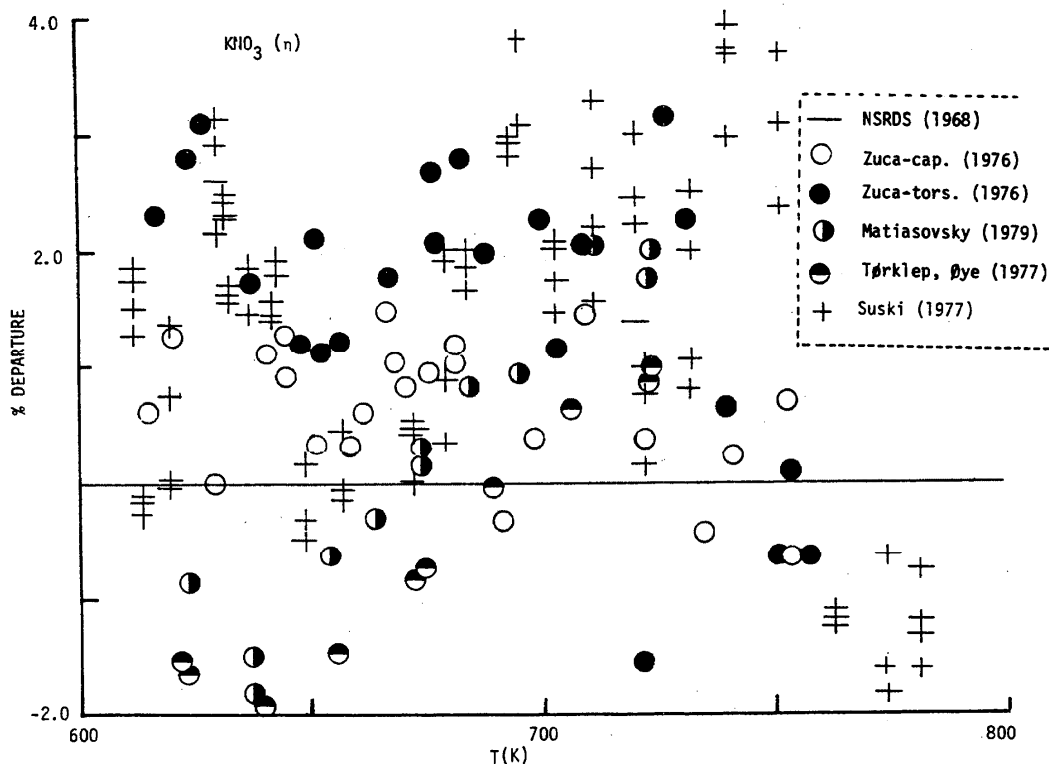


Figure 8 Comparison of  $\text{KNO}_3$  viscosity data from the Standards Program with the NSRDS (1968) recommendation  
A Percent Departure analysis is illustrated with the NSRDS (1968) recommendation [Dantuma data base]:  $\eta = -50.1676 - 164.572 \times 10^{-3}T + 186.335 \times 10^{-6}T^2 - 71.2497 \times 10^{-9}T^3$  as comparison base.

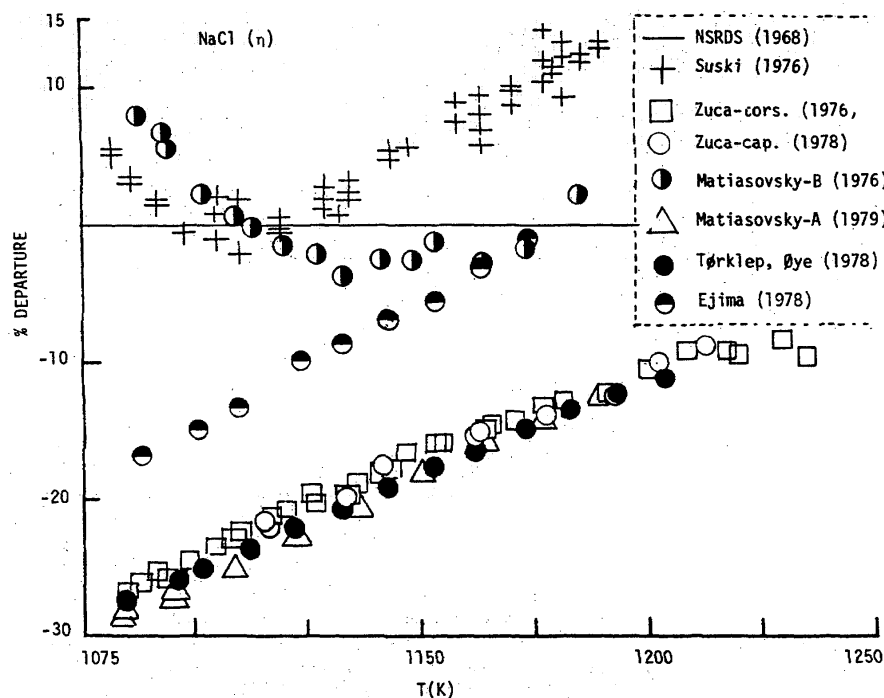


Figure 9 Comparison of NaCl viscosity data from the Standards Program with the NSRDS (1968) recommendation

A Percent Departure analysis is illustrated with the NSRDS (1968) recommendation [Murgulescu and Zuca]:  $\eta = 24.14537 - 36.0015 \times 10^{-3}T + 13.86822 \times 10^{-6}T^2$  as comparison reference.

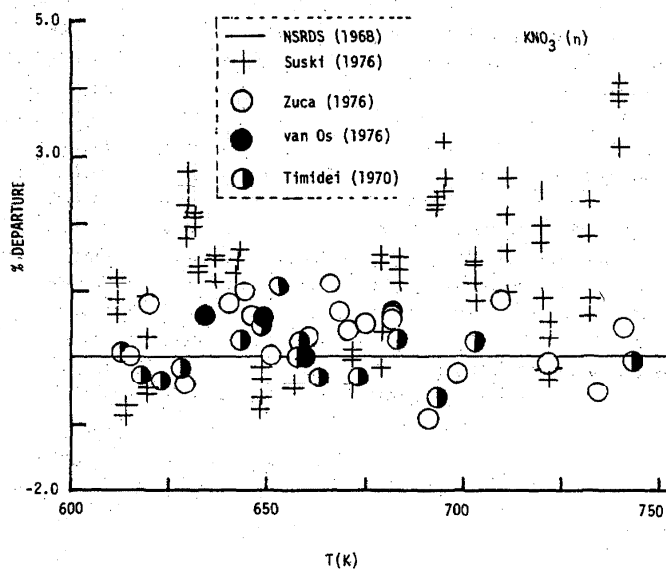


Figure 10 Comparison of KNO<sub>3</sub> viscosity data from capillary techniques

The results of Zuca and Suski are with Standards-Quality KNO<sub>3</sub>, but with the classical and modified short capillary-type viscometers; Timidei and van Os used Reagent-Grade KNO<sub>3</sub> and classical capillary viscometers of differing designs. The results are illustrated as a Percent Departure analysis relative to the NSRDS-1968 recommendations [Dantuma data base] as comparison reference, i.e.,:  $\eta = 50.1676 - 0.164572T + 1.86335 \times 10^{-4}T^2 - 7.12497 \times 10^{-8}T^3$



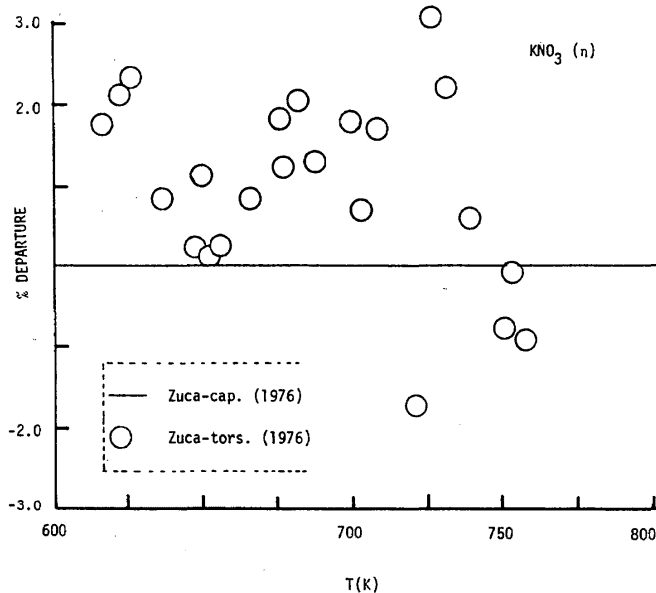


Figure 11 Comparison of the  $\text{KNO}_3$  viscosity data from the torsional and capillary techniques

The results are illustrated as a Percent Departure analysis of the torsional data relative to the capillary results:  
 $\eta = 27.46798 - 64.57701 \times 10^{-3}T + 39.941335 \times 10^{-6}T^2$  as comparison reference. Both from measurements at Bucharest [47,48] with Standards-Quality  $\text{KNO}_3$ .

In a private communication [198] Tørklep and Øye have pointed to an additional error source; this is the possible adherence of gas bubbles to the oscillating body. This was not fully realized during their  $\text{KNO}_3$  measurements in the Standards Program and it is suggested that the poorer reproducibility of this data set relative to  $\text{NaCl}$  is probably due to the gas bubble adherence effect. A small decomposition of the nitrate with resulting gas, adherence would account for the poorer reproducibility. In recent redeterminations of the alkali chlorides at Trondheim [199], the gas bubble problem occurred, especially in cooling (where the gas solubility decreases). This error source is less serious if the viscosity measurements are carried out at a sequence of increasing temperatures. If adherence occurs, the remedy is simply to move the oscillating body up and down until the gas bubbles are dislodged.

Damage to the laboratory by the Sendai earthquake (1978) interrupted the measurements projected with Standards Quality  $\text{KNO}_3$ , so that this part of the program could not be completed at Sendai.

## 6. Numerical Data

For each of the two compounds,  $\text{KNO}_3$  and  $\text{NaCl}$ , the earlier studies of densities, surface tensions, electrical conductances, and viscosities have been re-examined in light of the results from the Standards Program project. In addition the numerical values for each property were calculated from the recommended data base and have been tabulated. In this section, accordingly, the results of these evaluations are presented as follows.

(a) Investigations critically examined. The results are summarized both as a table and in graphical format. The former lists the reference numbers, authors, dates, together with the minimum and maximum deviations (as percent departures) from the recommended data set. Point by point comparisons with the recommended data set for each study are illustrated in the figures, also as percent departures.

(b) Recommended numerical values. The best equation for the data set selected as the reference base was used to calculate numerical values at rounded values of temperature. The numerical values are summarized in tables, together with the equations, and precisions. The accuracy estimate was based on the considerations of the experimental features, as already discussed. The coefficients of the equations are listed to more significant figures than justified by the accuracy for internal consistency with the limits of precision.

### 6.1. Potassium Nitrate: $\text{KNO}_3$

6.1.1.  $\text{KNO}_3$  density ( $\rho$ ): The measurements of Matiasovsky and Votova with Standards Quality  $\text{KNO}_3$  [12] are selected to supersede the 1968 NSRDS density recommendations [2]. The reference data base thus is gently shifted to lower values by  $\sim 0.25\%$  (see fig. 2).

Measurement Method: Archimedeian technique [12]

Density equations:

$$\rho(\text{gcm}^{-3}) = a + bT \quad (6.1.1a)$$

$$\rho(\text{gcm}^{-3}) = \alpha + \beta t \quad (6.1.1b)$$

precision:  $\sim \pm 0.1\%$  accuracy:  $\sim \pm 0.25\%$

The parameters for eqs (6.1.1a,1b) and the numerical values for the densities are in tables 10.1 and 10.2, respectively. The results of critical comparisons of the density measurements for the period 1907-1976 for  $\text{KNO}_3$  are summarized in table 11 and illustrated in figures 12 and 13.

6.1.2.  $\text{KNO}_3$  surface tension ( $\gamma$ ): The measurements of Nissen with Standard Quality  $\text{KNO}_3$  [15] are selected as the recommended reference data set. This is a shift from the data set selected in 1969 [3] but inspection of figure 3 shows that the two investigations agree within  $\pm 0.25\%$ , i.e., well within the limits of experimental accuracies.

Measurement method: maximum bubble pressure [15]

Surface tension equations:

$$\gamma(\text{mN m}^{-1}) = a + bT \quad (6.1.2a)$$

$$\gamma(\text{mN m}^{-1}) = \alpha + \beta t \quad (6.1.2b)$$

precision:  $\sim \pm 0.1\%$  accuracy:  $\sim \pm 0.5\%$

The parameters for eqs (6.1.2a,2b) and the numerical values of the surface tensions are in tables 12.1 and 12.2, respectively. The results of critical comparisons of the surface tension data for the period 1958-1976 for  $\text{KNO}_3$  are summarized in table 13 and illustrated in figure 14.

6.1.3.  $\text{KNO}_3$  electrical conductance ( $\kappa$ ): The measurements of Emons and Brautigam with Standards Quality  $\text{KNO}_3$  [35] are selected as the recommended reference data set. The NSRDS  $\text{KNO}_3$  recommendations, first advanced in 1968, were revised and updated in 1972. The data set of Emons and Brautigam (1975) is virtually in exact agreement with the 1972 NSRDS data base, well within experimental limits of accuracies,  $\pm 0.5\%$  (see fig. 6).

Measurement method: ac conductance technique [35]

Electrical conductance equations:

$$\kappa(\Omega^{-1} \text{cm}^{-1}) = a + bT + cT^2 \quad (6.1.3a)$$

$$\kappa(\Omega^{-1} \text{cm}^{-1}) = \alpha + \beta t + \gamma t^2 \quad (6.1.3b)$$

precision:  $\sim \pm 0.1\%$  accuracy:  $\sim \pm 0.5\%$

The parameters for eqs (6.1.3a,3b) and the numerical values of the electrical conductances are in tables 14.1 and 14.2, respectively. The results of critical comparisons of the conductivity data for the period 1917-1979 for  $\text{KNO}_3$  are summarized in table 15 and illustrated in figures 15 and 16.

6.1.4.  $\text{KNO}_3$  viscosity ( $\eta$ ): The measurements of Zuca and Costin with Standards Quality  $\text{KNO}_3$  [47] are selected as

the recommended reference data set, and this data set supersedes the 1969 NSRDS recommendations. Inspection of figure 8 shows that this gently shifts the reference data base  $\sim 1\%$  to higher values.

Measurement method: capillary technique [47]

Viscosity equations:

$$\eta(\text{mN s m}^{-2}) = a + bT + cT^2 \quad (6.1.4a)$$

$$\eta(\text{mN s m}^{-2}) = \alpha + \beta t + \gamma t^2 \quad (6.1.4b)$$

precision:  $\sim \pm 0.6\%$  accuracy:  $\sim \pm 2.0\%$

The parameters for eqs (6.1.4a,4b) and the numerical values for the viscosities are in tables 16.1 and 16.2, respectively. The results of critical comparisons of the viscosity data for  $\text{KNO}_3$  for the period 1907-1978 are summarized in table 17 and illustrated in figures 17 and 18.

Table 10.1 Parameters of density equations (6.1.1a,b)

	a (g cm <sup>-3</sup> )	-b x 10 <sup>3</sup> (g cm <sup>-3</sup> K <sup>-1</sup> )	temp. range
Kelvin	2.3063	0.7235	620 - 730 K
	$\alpha$ (g cm <sup>-3</sup> )	$-\beta \times 10^3$ (g cm <sup>-3</sup> deg <sup>-1</sup> )	
Celsius	2.1087	0.7235	345 - 455 C

Table 10.2  $\text{KNO}_3$  Densities from Kelvin equation (6.1a)

T (K)	$\rho$ (g cm <sup>-3</sup> )	T (K)	$\rho$ (g cm <sup>-3</sup> )
620	1.858	680	1.814
625	1.854	685	1.811
630	1.851	690	1.807
635	1.847	695	1.803
640	1.843	700	1.800
645	1.840	705	1.796
650	1.836	710	1.793
655	1.832	715	1.789
660	1.829	720	1.785
665	1.825	725	1.782
670	1.822	730	1.770
675	1.818		

Table 11  $\text{KNO}_3$  density studies critically examined and comparisons with [12] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
12	Matiasovsky, Votova (1975)	0.00%	0.00%
69	Slavyanski (1947)	graphical	
70	Smith, Van Artsdalen (1961)	0.20% (725 K)	0.43% (625 K)
71	Murgulescu, Zuca (1959)	0.109% (673 K)	0.73% (623 K)
72	Protsenko, Malakhova (1961)	0.04% (673 K)	0.20% (633 K)
73	Dantuma (1928)	0.00% (660 K)	0.16% (643 K)
74	Lorenz, Frei, Jabs (1908)	-0.04% (733 K)	-0.57% (723 K)
75	Polyakov, Beral (1955)	0.01% (733 K)	0.15% (633 K)
76	Jaeger, Kapma (1920)	1.33% (623 K)	1.73% (735 K)
77	Bloom, Knaggs, Molloy, Welch (1953)	0.32% (725 K)	0.34% (625 K)
78	Polyakov (1955)	0.04% (673 K)	-0.29% (623 K)
79	Goodwin, Mailey (1907)	0.43% (793 K)	0.64% (633 K)
80	Fawsitt (1908)	0.04% (680 K)	0.37% (622 K)
81	McAuley, Rhodes, Ubbelohde (1966)	-0.01% (640 K)	-0.12% (690 K)
82	Sandonnini (1920)	1.63% (673 K)	1.63% (673 K)
83	Smith, Petersen (1961)	-0.12% (620 K)	0.43% (737 K)
84	Bockris, Calandra, Solomons (1969)	0.21% (716 K)	0.47% (629 K)
85	Murgulescu, Zuca (1969)	0.19% (750 K)	0.40% (630 K)
86	Layborn, Madgin (1934)	0.07% (693 K)	0.13% (627 K)
13	Ejima, Shimakage, Hoshi (1976)	0.00% (703 K)	0.95% (686 K)

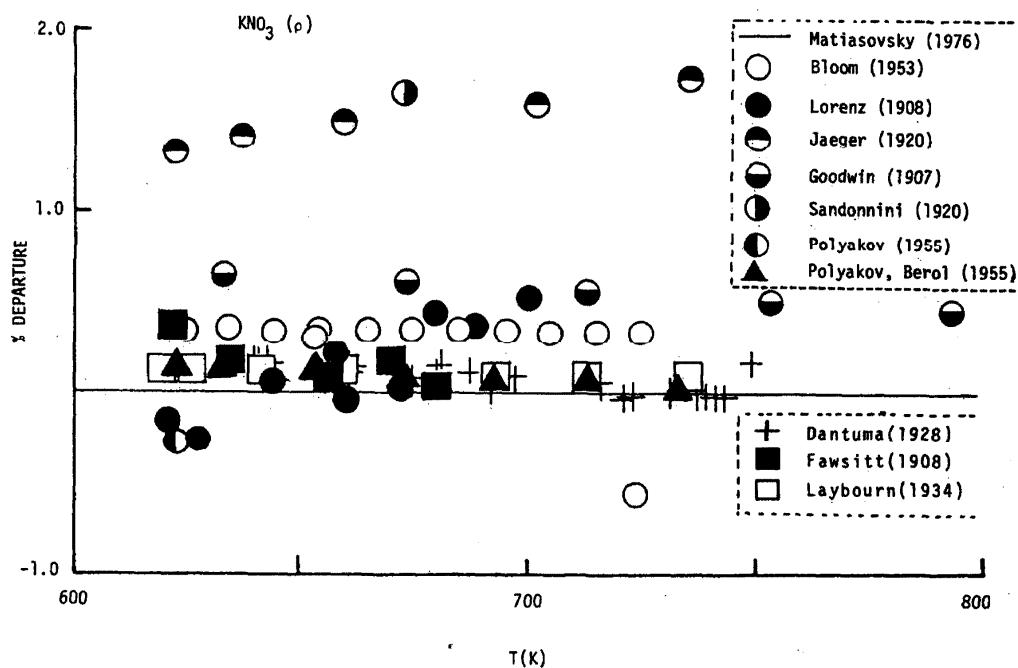


Figure 12  $\text{KNO}_3$  density data for the period 1907-1955, shown as a percent departure analysis with the Matiasovsky and Votova data base (1976) as comparison reference standard.

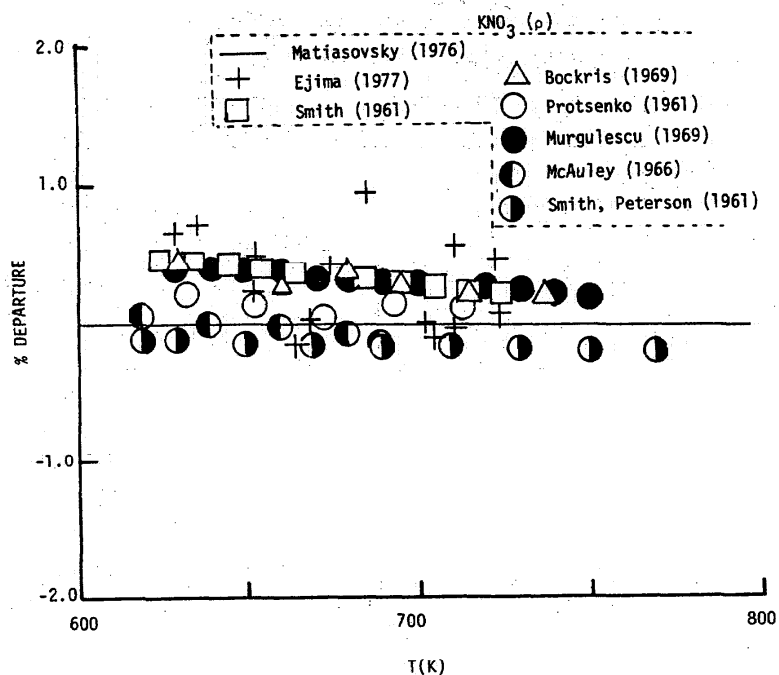


Figure 13  $\text{KNO}_3$  density data for the period 1955-1976, shown as percent departure analysis with the Matiasovsky and Votova data base (1976) as comparison reference standard.

Table 12.1 Parameters of  $\text{KNO}_3$  surface tension equations (6.1.2a,b)

kelvin	a ( $\text{mN m}^{-1}$ )	$-b \times 10^3$ ( $\text{mN m}^{-1}\text{K}^{-1}$ )	temp. range
	154.715	71.7080	620 - 760 K
Celsius	$\alpha$ ( $\text{mN m}^{-1}$ )	$-\beta \times 10^3$ ( $\text{mN m}^{-1}\text{deg}^{-1}$ )	
	135.128	71.7080	345 - 490°C

Table 12.2  $\text{KNO}_3$  surface tensions<sup>a</sup> from kelvin equation (6.1.2a)

T (K)	$\gamma$ ( $\text{mN m}^{-1}$ )	T (K)	$\gamma$ ( $\text{mN m}^{-1}$ )
620	110.3	695	104.9
625	109.9	700	104.5
630	109.5	705	104.2
635	109.2	710	103.8
640	108.8	715	103.4
645	108.5	720	103.1
650	108.1	725	102.7
655	107.7	730	102.4
660	107.4	735	102.0
665	107.0	740	101.6
670	106.7	745	101.3
675	106.3	750	100.9
680	105.9	755	100.6
685	105.6	760	100.2
690	105.2		

(a)  $1 \text{ mN m}^{-1} = 1 \text{ dyn cm}^{-1}$ ; (see table 2)

Table 13.  $\text{KNO}_3$  surface tension studies critically examined and Comparisons with [15] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
87	Janz, Lorenz (1960)	-0.04% (698 K)	-0.20% (738 K)
88	Bloom et al. (1960)	-0.71% (710 K)	-1.24% (630 K)
89	Dahl, Duke (1958)	1.59% (727 K)	2.83% (671 K)
90	Addison, Coldrey (1961)	-0.48% (630 K)	-0.61% (710 K)
91	Bertozzi, Sternheim (1964)	0.57% (710 K)	1.22% (630 K)
92	Jaeger (1917)	-0.20% (679 K)	-4.50% (687 K)
93	Eliseeva et. al. (1969)	-1.21% (630 K)	-1.38% (710 K)
16	Ejima, Yamamura (1977)	0.01% (661 K)	0.75% (652 K)
17	Suski, Tomczyk (1975)	-0.01% (706 K)	1.19% (659 K)
18	Zuca, Terzi (1975)	-0.65% (738 K)	-1.32% (617 K)
15	Nissen (1975)	0.00%	0.00%

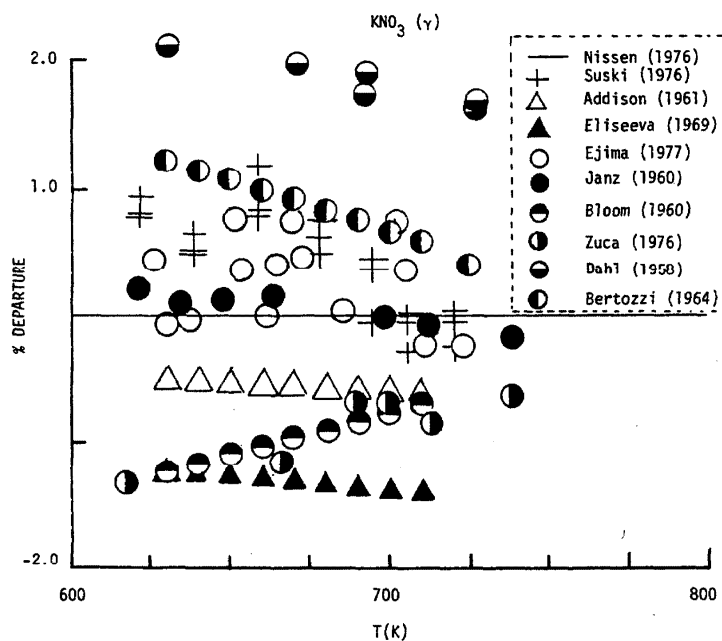


Figure 14  $\text{KNO}_3$  surface tension data for the period 1958-1976, shown as a percent departure analysis with the Nissen data base (1976) as comparison reference standard.

Table 14.1 Parameters of the  $\text{KNO}_3$  conductance equations (6.1.3a,b)

	-a ( $\Omega^{-1} \text{ cm}^{-1}$ )	b x 10 <sup>3</sup> ( $\Omega^{-1} \text{ cm}^{-1} \text{ K}^{-1}$ )	-c x 10 <sup>6</sup> ( $\Omega^{-1} \text{ cm}^{-1} \text{ K}^{-2}$ )	temp. range
kelvin	2.1250	5.7332	2.0301	615 - 789 K
	-a ( $\Omega^{-1} \text{ cm}^{-1}$ )	$\beta \times 10^3$ ( $\Omega^{-1} \text{ cm}^{-1} \text{ deg}^{-1}$ )	- $\gamma \times 10^6$ ( $\Omega^{-1} \text{ cm}^{-1} \text{ deg}^{-2}$ )	
Celsius	0.7109	4.6242	2.0301	340° - 505°C

Table 14.2  $\text{KNO}_3$  Electrical conductances from Kelvin equations (6.1.3a)

T (K)	( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	T (K)	( $\text{ohm}^{-1} \text{ cm}^{-1}$ )
615	0.633	705	0.908
620	0.649	710	0.922
625	0.665	715	0.936
630	0.681	720	0.951
635	0.697	725	0.965
640	0.713	730	0.978
645	0.728	735	0.992
650	0.744	740	1.006
655	0.759	745	1.019
660	0.775	750	1.033
665	0.790	755	1.046
670	0.805	760	1.060
675	0.820	765	1.073
680	0.835	770	1.086
685	0.850	775	1.099
690	0.864	780	1.112
695	0.879		
700	0.894		

Table 15.  $\text{KNO}_3$  electrical conductance studies critically examined and comparisons with [35] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
94	Lorenz, Kalmus (1907)	0.02% (616 K)	-1.65% (606 K)
95	Aten (1912)	0.13% (623 K)	1.41% (693 K)
96	Jaeger, Kapma (1920)	-0.20% (619 K)	1.30% (691 K)
97	Sandonnini (1920)	0.72% (623 K)	5.46% (673 K)
98	Smith, Van Artsdalen (1961)	0.0% ((650 K)	1.04% (610 K)
99	Bloom, Knaggs, Molloy, Welch (1953)	-0.12% (840 K)	-0.15% (800 K)
100	King, Duke (1964)	0.18% (627 K)	1.62% (671 K)
101	Murgulescu, Zuca (1959)	1.11% (623 K)	3.80% (673 K)
102	Angell (1965)	1.12% (626 K)	-1.66% (658 K)
103	DeNoijer, Ketelaar (1967)	0.34% (678 K)	-2.80% (613 K)
104	Robbins, Braunstein (1969)	0.00% (770 K)	-0.48% (630 K)
105	Rhodes, Smith, Ubbelohde (1965)	0.51% (620 K)	2.34% (660 K)
106	Papaioannou, Harrington (1964)	0.04% (660 K)	-0.98% (730 K)
107	Bizouard (1961)	-0.10% (606 K)	2.43% (723 K)
108	Bergman, Chagin (1940)	0.06% (623 K)	0.84% (673 K)
109	Brillant (1967)		graphical
110	Cowen, Axon		graphical
111	Protsenko, Papovskaya (1954)		graphical
112	Protsenko, Papovskaya (1961)		graphical
113	Barton, Cleaver, Hills (1968)		graphical
114	Goodwin, Mailey (1907)		graphical
115	Protsenko, Malakhova (1960)		graphical
33	Zuca, Olteanu (1975)	0.57% (628 K)	1.27% (720 K)

Table 15.  $\text{KNO}_3$  electrical conductance studies critically examined and comparisons with [35] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
35	Emons, Brautigam (1975)	0.00%	0.00%
34	Ejima, Shimakage, Sato (1976)	0.35% (616 K)	1.60% (652 K)
36	Suski, Gadek, Stachowitz (1979)	0.02% (706 K)	-1.81% (610 K)

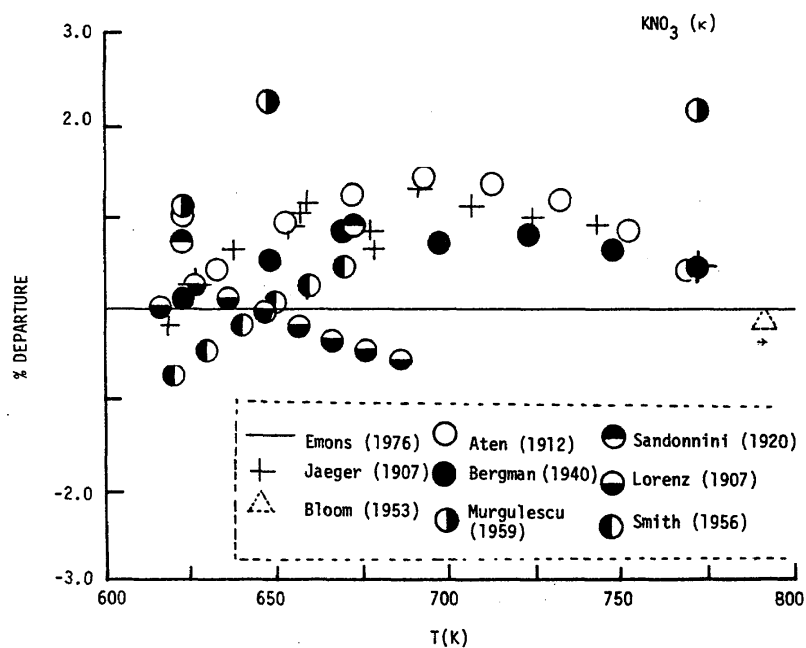


Figure 15  $\text{KNO}_3$  conductance data for the period 1917-1960, shown as a percent departure analysis with Emons and Brautigam data base (1976) as comparison reference standard.

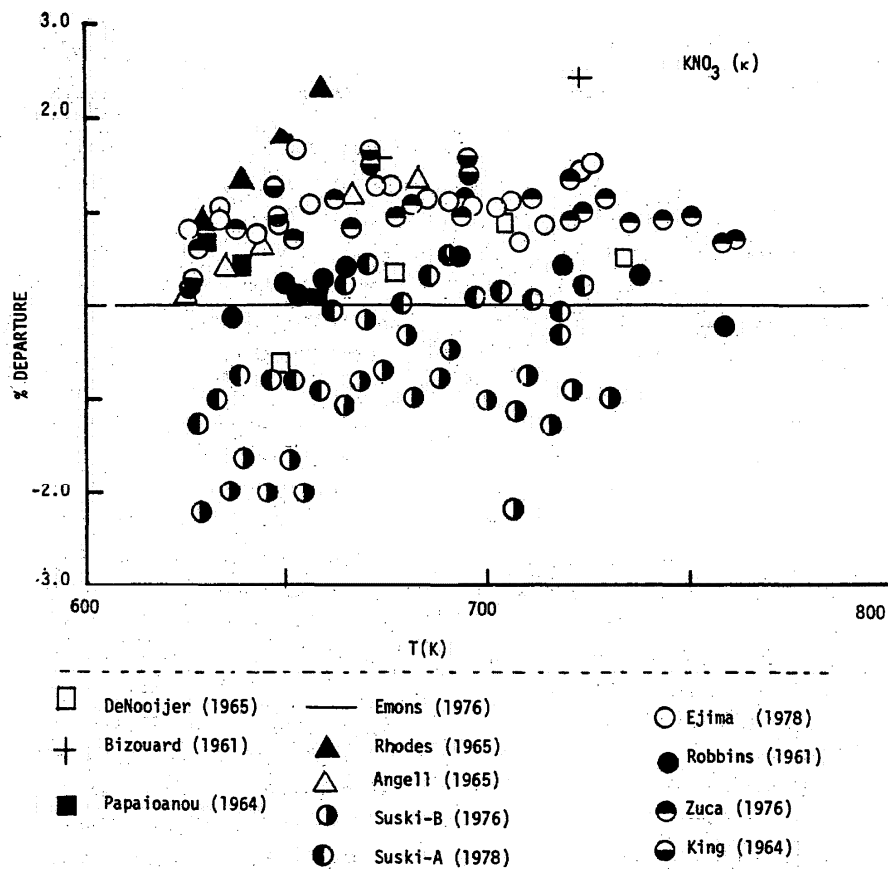


Figure 16  $\text{KNO}_3$  conductance data for the period 1960-1979, shown as a percent departure analysis with the Emons and Brautigam data base (1976) as comparison reference standard.

Table 16.1 Parameters for  $\text{KNO}_3$  viscosity equations (6.1.4a,b)

	a	$-b \times 10^3$	$c \times 10^6$	temp. range
kelvin	( $\text{mN s m}^{-2}$ )	( $\text{mN s m}^{-2} \text{K}^{-1}$ )	( $\text{mN s m}^{-2} \text{K}^{-2}$ )	
	29.7085	71.1208	44.7023	615 - 760 K
Celsius	a	$-B \times 10^3$	$\gamma \times 10^6$	
	( $\text{mN s m}^{-2}$ )	( $\text{mN s m}^{-2} \text{deg}^{-1}$ )	( $\text{mN s m}^{-2} \text{deg}^{-2}$ )	
	13.6171	46.6999	44.7023	340° - 490°C

Table 16.2  $\text{KNO}_3$  viscosities calculated from Kelvin equation (6.1.4)

T (K)	$\eta$ (cp)	T (K)	$\eta$ (cp)
615	2.877	690	1.918
620	2.797	695	1.872
625	2.720	700	1.828
630	2.645	705	1.786
635	2.572	710	1.747
640	2.501	715	1.710
645	2.433	720	1.675
650	2.367	725	1.643
655	2.303	730	1.612
660	2.241	735	1.584
665	2.181	740	1.558
670	2.124	745	1.534
675	2.069	750	1.513
680	2.017	755	1.494
685	1.966	760	1.477



Table 17.  $\text{KNO}_3$  viscosity studies critically examined and comparisons with [47] as the recommended reference data base

Ref.	Author	Min. departure	Max. departure
116	Goodwin, Mailey (1908)	-0.07% (669 K)	-3.90% (644 K)
117	Lorenz, Kalmus (1907)	-0.01% (626 K)	2.41% (686 K)
118	Fawsitt (1908)	0.50% (670 K)	-6.52% (635 K)
119	Janz, Saegusa (1963)	-0.25% (674 K)	-2.83% (764 K)
120	Murgulescu, Zuca (1961)	0.60% (716 K)	-21.1% (630 K)
121	Protzenko, Razumovskaya (1965)	-0.03% (681 K)	-2.83% (764 K)
41	Dantuma (1928)	0.03% (741 K)	-16.95% (815 K)
122	Ogawa (1950)	graphical	
123	Murgulescu (1969)	-0.35% (698 K)	-2.63% (748 K)
124	Murgulescu, Zuca (1966)	-0.35% (698 K)	-1.86% (648 K)
125	Rhodes, Smith, Ubbelohde (1965)	-0.41% (628 K)	-1.30% (658 K)
126	Zuca, Borcan (1970)	-1.66% (630 K)	-2.08% (670 K)
49	Timidei, Janz, Lederman (1970)	0.02% (683 K)	-0.87% (663 K)
127	Dumas, Grojtheim, Hogdahl, Øye (1970)	-2.23% (655 K)	-5.40% (615 K)
128	Kleinschmidt (1968)	-1.17% (675 K)	-8.98% (630 K)
129	Willman, DeWitt, Ellis (1966)	0.04% (765 K)	3.24% (712 K)
47	Zuca, Costin (1975)	0.00%	0.00%
46	Tørklep, Øye (1977)	0.27% (706 K)	-2.80% (640 K)
45	Suski, Galka, Stachowicz (1975)	0.02% (722 K)	4.10% (751 K)
50	Ketelaar and Van Os (1973)	-0.07% (649 K)	-1.18% (621 K)
44	Matiasovsky, Votova (1976)	0.07% (624 K)	6.27% (723 K)
48	Zuca and Borcan (1975)	0.08% (655 K)	3.05% (740 K)

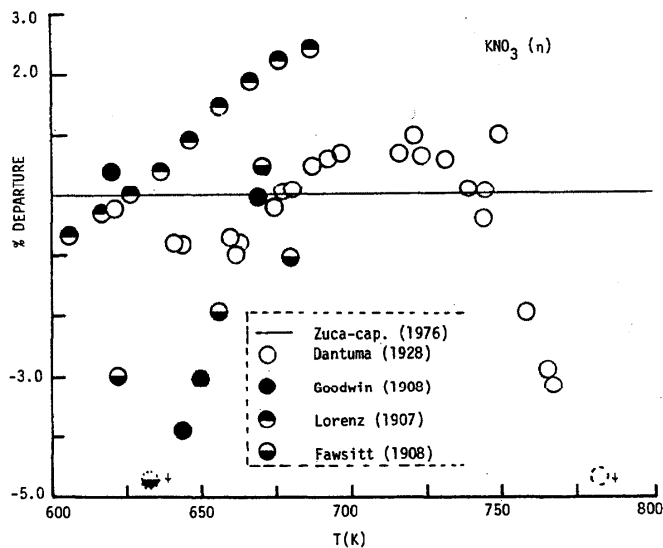


Figure 17  $\text{KNO}_3$  viscosity data for the period 1907-1968, shown as a percent departure analysis with the Zuca and Costin data base [1976] as comparison reference standard.

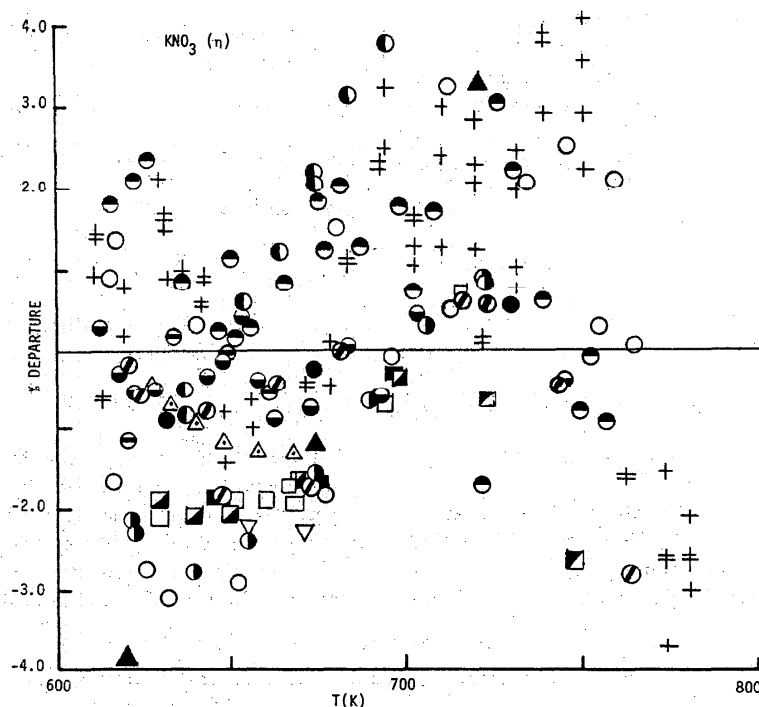
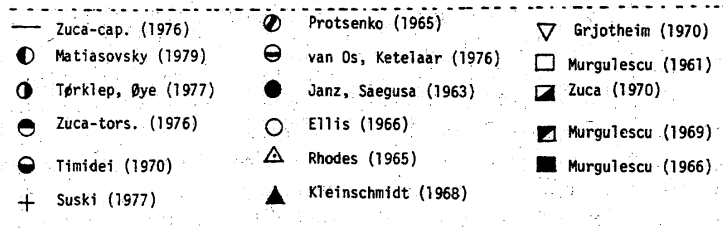


Figure 18  $\text{KNO}_3$  viscosity data for the period 1960-1978 as comparison reference standard with the Zuca and Costin data base (1976) as comparison reference standard.

## 6.2. Sodium Chloride: NaCl

6.2.1. NaCl density ( $\rho$ ): The 1968 NSRDS density recommendations [2] are based on the 1955 determinations of van Artsdalen and Yaffe [38]. Inspection of figure 3 shows that the measurements of Matiasovsky and Votova with Standards Quality NaCl [12] support the 1968 recommendations well within the limits of accuracy of the experimental techniques ( $\sim \pm 1.0\%$ ). These are as follows:

Measurement method: Archimedean technique [38]

Density equations:

$$\rho(\text{gcm}^{-3}) = a + bT \quad (6.2.1a)$$

$$\rho(\text{gcm}^{-3}) = \alpha + \beta t \quad (6.2.1b)$$

precision:  $\sim \pm 0.1\%$  accuracy:  $\sim \pm 1.0\%$

The parameters for eqs (6.2.1a,1b) and the numerical density values are in tables 18.1 and 18.2, respectively. The results of critical comparisons of the density data for NaCl for the period 1904-1975 are summarized in table 19 and illustrated in figures 19 and 20.

6.2.2. NaCl surface tension ( $\gamma$ ): The 1969 NSRDS surface tension recommendations [3] are based on the 1962 determinations of Sokolova and Voskresenskaya [151]. Figure 5 shows a comparison of the measurements of Zuca and Terzi [18] and Suski and Tomczyk [17], both with Standards Quality NaCl. The deviations of the Zuca, Terzi data set intuitively suggest undetected temperature gradients, but this is at best speculative and further discussion is deferred. Without additional data, the accuracy limits for NaCl must be set at  $\sim \pm 1.5\%$ , rather than the limits of  $\sim 0.1\%$  as estimated in the earlier work. Within these limits, the results of the Standards Program support the 1969 NSRDS recommendations. These are as follows:

Measurement method: maximum bubble pressure [151]

Surface tension equations:

$$\gamma(\text{mN m}^{-1}) = a + bT \quad (6.2.2a)$$

$$\gamma(\text{mN m}^{-1}) = \alpha + \beta t \quad (6.2.2b)$$

precision:  $\sim \pm 0.2\%$  accuracy:  $\sim \pm 1.5\%$

The parameters for eqs (6.2.2a,2b) and the numerical surface tension values are in tables 20.1 and 20.2, respectively. The results of critical comparisons of the surface tension data for NaCl for the period 1917–1975 are summarized in table 21 and illustrated in figures 21 and 22.

6.2.3. NaCl electrical conductance ( $\kappa$ ): The measurements of Emons and Brautigam with Standards Quality NaCl [35] are selected as the recommended reference data set. Inspection of figure 7 shows that whereas the 1968 NSRDS recommendations [2] are virtually in exact agreement with this new data set in the range 800–850 °C, the 1968 recommendations are uniformly higher with increasing temperatures, the departure increasing to a maximum of ~3.5% at the upper limit of measurements (~1020 °C). Through the Standards Program measurements the quality of the NaCl specific conductance data has been upgraded to calibration-quality data.

Measurement method: ac conductance technique [35]

Electrical conductance equations:

$$\kappa(\Omega^{-1} \text{ cm}^{-1}) = a + bT + cT^2 \quad (6.2.3a)$$

$$\kappa(\Omega^{-1} \text{ cm}^{-1}) = \alpha + \beta t + \gamma t^2 \quad (6.2.3b)$$

precision:  $\sim \pm 0.15\%$  accuracy:  $\sim \pm 1\%$

The parameters for eqs (6.2.3a,3b) and the numerical values of the NaCl electrical conductivities are in tables 22.1 and 22.2, respectively. The results of critical comparisons of the NaCl conductance data for the period 1920–1979 are

summarized in table 23 and illustrated in figures 23 and 24.

6.2.4. NaCl viscosity ( $\eta$ ): The measurements of Tørklep and Øye with Standards Quality NaCl [46,57] are selected as the recommended reference data set. The values advanced in the 1968 NSRDS recommendations are superseded with this new data set. Inspection of figure 9, and reference to the error analyses (see 5.4), justifies this shift of the reference data base to the Tørklep and Øye data set.

Measurement method: torsional technique; immersed cylinder [46,57]

Viscosity equations.

$$\eta(\text{mN s m}^{-2}) = A \exp[E/RT] \quad (6.2.4a)$$

$$\eta(\text{mN s m}^{-2}) = a + bT + cT^2 \quad (6.2.4b)$$

$$\eta(\text{mN s m}^{-2}) = \alpha + \beta t + \gamma t^2 \quad (6.2.4c)$$

precisions: (6.2.4a):  $\sim \pm 0.05\%$  accuracy:  $\sim \pm 0.2\%$

(6.2.4b):  $\sim \pm 0.4\%$

(6.2.4c):  $\sim \pm 0.4\%$

The parameters of eqs (6.2.4a,4b,4c) and the numerical values for NaCl viscosities are in tables 24.2 and 24.1, respectively. The confidence level for the accuracy estimate is given as 99%. The results of critical comparisons of the NaCl viscosity data for the period 1908–1979 are summarized in table 25 and illustrated in figure 25.

Table 18.1 Parameters for NaCl density equations (6.2.1a,b)

kelvin	a	$-b \times 10^3$	temp. range
	(g cm <sup>-3</sup> )	(g cm <sup>-3</sup> K <sup>-1</sup> )	
	2.1389	0.5426	1080 – 1300 K
Celsius	$\alpha$	$-\beta \times 10^3$	
	(g cm <sup>-3</sup> )	(g cm <sup>-3</sup> deg <sup>-1</sup> )	
	1.9907	0.5426	805° – 1030°C

Table 18.2 NaCl densities calculated from Kelvin equation (6.2.1a)

T (K)	$\rho$ (g cm <sup>-3</sup> )	T (K)	$\rho$ (g cm <sup>-3</sup> )
1080	1.553	1195	1.490
1085	1.550	1200	1.488
1090	1.547	1205	1.485
1095	1.545	1210	1.482
1100	1.542	1215	1.480
1105	1.539	1220	1.477
1110	1.537	1225	1.474
1115	1.534	1230	1.471
1120	1.531	1235	1.469
1125	1.528	1240	1.466
1130	1.526	1245	1.463
1135	1.523	1250	1.461
1140	1.520	1255	1.458
1145	1.518	1260	1.455
1150	1.515	1265	1.452
1155	1.512	1270	1.450
1160	1.509	1275	1.447
1165	1.507	1280	1.444
1170	1.504	1285	1.442
1175	1.501	1290	1.439
1180	1.499	1295	1.436
1185	1.496	1300	1.433
1190	1.493		

Table 19. NaCl density studies and comparisons with [38]  
as the reference data base

Ref.	Authors	Min. departure	Max. departure
38	Van Artsdalen, Yaffe (1955)	0.00%	0.00%
130	Brunner (1904)	0.02% (1283 K)	0.84% (1082 K)
92	Jaeger (1917)	-0.53% (10 K)	-1.62% (1268 K)
131	Edwards, et al. (1952)	0.19% (1300 K)	-0.41% (1110 K)
77	Bloom, et al. (1953)	-0.01% (1121 K)	-0.08% (1120 K)
132	Kirshenbaum, et al. (1962)	-0.01% (1208 K)	-0.35% (1162 K)
133	Bukhalova, Yaguby'an (1968)		graphical
134	Emons, Brautigam, Vogt (1970)		graphical
135	Vereshchetina, Luzhnaya (1954)	-1.29% (1098 K)	-1.46% (1128 K)
136	Grjotheim, Lillebuen, Holm (1971)	0.22% (1073 K)	
82	Sandonnini (1920)	-1.13% (1123 K)	-1.13% (1123 K)
137	Chesnokov, Strelets (1970)		graphical
138	Zuca, Murgulescu (1962)	-0.58% (1085 K)	-1.68% (1165 K)
139	Jaeger, Kahn (1916)	-0.57% (1096 K)	-0.96% (1158 K)
140	Sackur (1913)	-0.34% (1120 K)	-0.44% (1080 K)
141	Zuca, Olteanu (1970)	0.12% (1090 K)	-0.13% (1170 K)
142	Bukhalova, Yagub'yan (1965)	2.72% (1080 K)	-4.27% (1270 K)
143	Barzakovskii (1940)	-2.76% (1080 K)	-3.71% (1270 K)
144	Luzhnaya, Evsseva, Vereskhetina (1956)		graphical
145	Markov, Prisyazhnyi (1963)		graphical
146	Lillebuen (1969)	0.40% (1087 K)	0.53% (1138 K)
147	Perks, Pozodnyokov, Sheiko (1965)	-0.53% (1080 K)	-1.07% (1170 K)
148	Zuca, Ionescu-Vasu (1967)	-0.12% (1080 K)	-0.14% (1200 K)
149	Yamada, Yoshida, Kunoda (1969)	-0.20% (1170 K)	-0.44% (1080 K)
150	Fuseya, Ouchi (1949)	0.46% (1085 K)	-0.47% (1080 K)
12	Mariasovsky, Votova (1975)	-0.02% (1280 K)	-1.04% (1080 K)

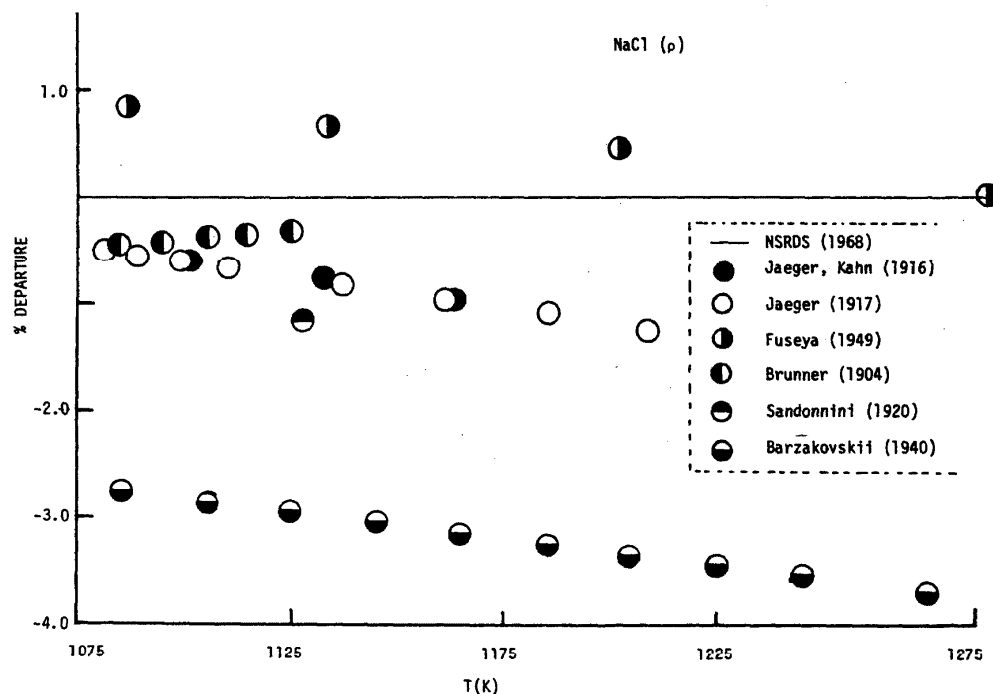


Figure 19 NaCl density data for the period 1904-1940, shown as a percent departure analysis with the 1968 NSRDS recommended data base (van Artsdalen and Yaffe [38]) as comparison standard.

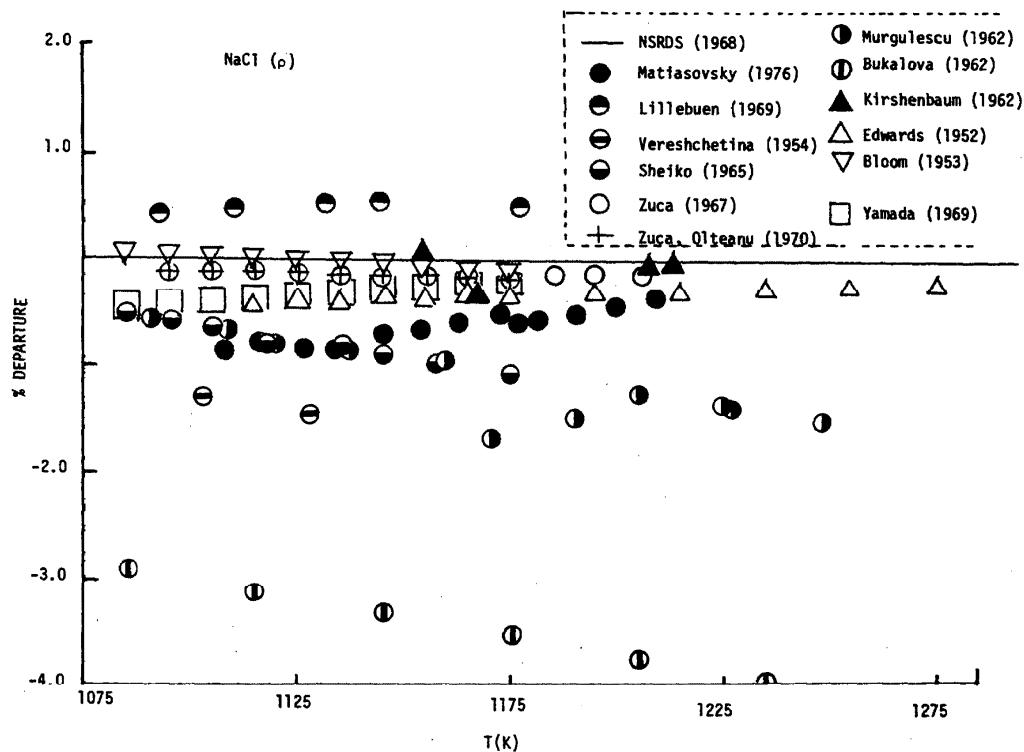


Figure 20 NaCl density data for the period 1940-1975, shown as a percent departure analysis with the 1968 NSRDS recommended data base (van Artsdalen and Yaffe [38]) as comparison standard.

Table 20.1 Parameters for the NaCl surface tension equations (6.2.2a,b)

kelvin	a (mN m <sup>-1</sup> )	-b x 10 <sup>3</sup> (mN m <sup>-1</sup> K <sup>-1</sup> )	temp. range
	191.1600	71.8800	1080 - 1240 K
Celsius	α (mN m <sup>-1</sup> )	-β x 10 <sup>3</sup> (mN m <sup>-1</sup> deg <sup>-1</sup> )	
	171.5260	71.8800	805° - 970°C

Table 20.2 NaCl surface tensions<sup>a</sup> calculated from kelvin equation (6.2.2a)

T (K)	γ (mN m <sup>-1</sup> )	T (K)	γ (mN m <sup>-1</sup> )
1080	113.5	1165	107.4
1085	113.2	1170	107.1
1090	112.8	1175	106.7
1095	112.4	1180	106.3
1100	112.1	1185	106.0
1105	111.7	1190	105.6
1110	111.4	1195	105.3
1115	111.0	1200	104.9
1120	110.6	1205	104.5
1125	110.3	1210	104.2
1130	109.9	1215	103.8
1135	109.6	1220	103.5
1140	109.2	1225	103.1
1145	108.9	1230	102.7
1150	108.5	1235	102.4
1155	108.1	1240	102.0
1160	107.8		

(a) 1 mN m<sup>-1</sup> = 1 dyn cm<sup>-1</sup>; (see table 2)

Table 21. NaCl surface tension studies critically examined and comparisons with [3,151] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
88	Bloom, Davis, James (1960)	-0.09% (1190 K)	1.96% (1080 K)
152	Semenchenko, Shikobalova (1947)	2.03% (1173 K)	2.67% (1223 K)
153	Lantratov (1961)	0.01% (1140 K)	0.32% (1080 K)
92	Jaeger (1917)	-0.03% (1203 K)	0.33% (1093 K)
154	Desyatnikov (1956)	0.03% (1080 K)	0.55% (1230 K)
155	Bertozzi (1965)	3.39% (1080 K)	3.42% (1180 K)
156	Barzakovskii (1940)	0.00% (1170 K)	0.01% (1080 K)
151	Sokolva, Voskrensenskaya (1962)	0.00%	0.00%
157	Sternberg, Terzi (1972)	0.02% (1120 K)	1.55% (1210 K)
158	Mizuno, Matsumura, Tenako (1968)	3.06% (1173 K)	3.70% (1103 K)
159	Lutzow, Holm (1971)	1.63% (1168 K)	2.01% (1153 K)
160	Dahl, Duke (1957)	1.19% (1147 K)	2.98% (1099 K)
17	Suski, Tomczyk (1975)	0.05% (1034 K)	0.34% (1127 K)
18	Zuca, Terzi (1975)	-0.10% (1200 K)	1.65% (1089 K)

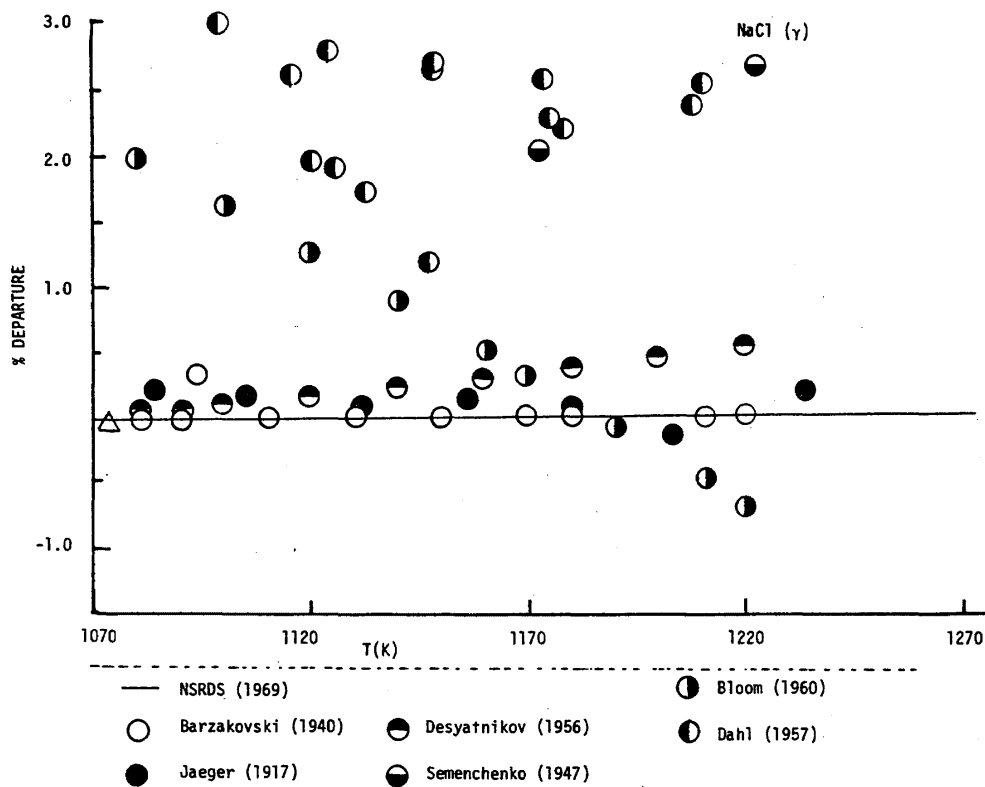


Figure 21 NaCl surface tension data for the period 1917-1960, shown as a percent departure analysis with the 1969 NSRDS recommended data base (Sokolova, Voskrenenskaya [1962]) as comparison standard.

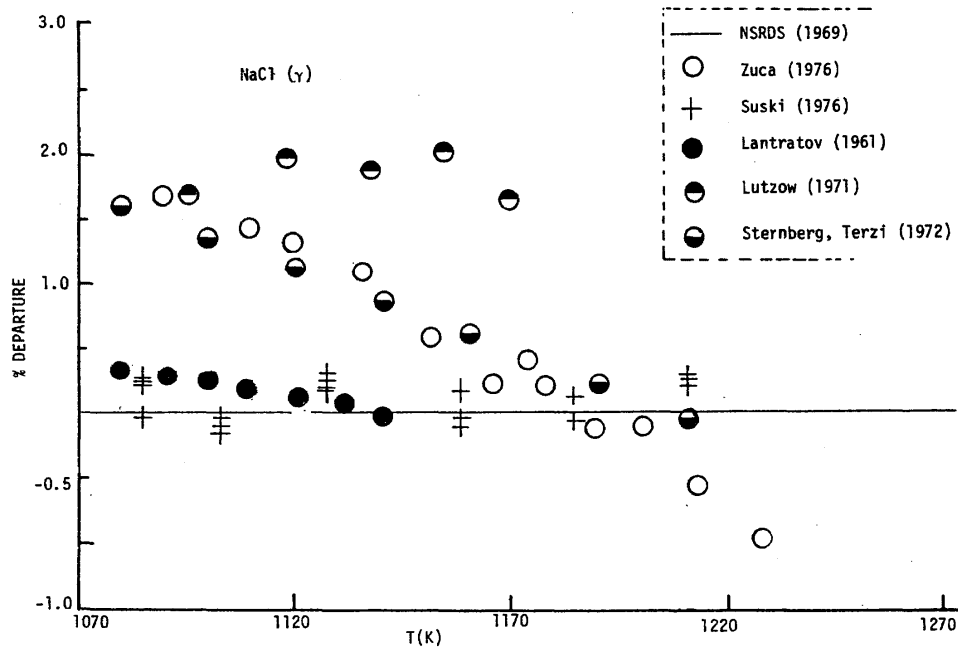


Figure 22 NaCl surface tension data for the period (1961-1975), shown as a percent departure analysis with the 1969 NSRDS recommended data base (Sokolova, Voskrenenskaya [1962]) as comparison standard.

Table 22.1 Parameters of NaCl conductance equations (6.2.3a,b)

kelvin	$-a$ ( $\Omega^{-1} \text{ cm}^{-1}$ )	$b \times 10^3$ ( $\Omega^{-1} \text{ cm}^{-1} \text{ K}^{-1}$ )	$-c \times 10^6$ ( $\Omega^{-1} \text{ cm}^{-1} \text{ K}^{-2}$ )	temp. range
	5.6241	13.9640	5.0245	1080 - 1250 K
Celsius	$-a$ ( $\Omega^{-1} \text{ cm}^{-1}$ )	$8 \times 10^3$ ( $\Omega^{-1} \text{ cm}^{-1} \text{ deg}^{-1}$ )	$-\gamma \times 10^6$ ( $\Omega^{-1} \text{ cm}^{-1} \text{ deg}^{-2}$ )	
	2.1847	11.2191	5.0245	805° - 980°C

Table 22.2 NaCl conductances calculated from Kelvin equation (6.2.3a)

T (K)	$\kappa$ ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	T (K)	$\kappa$ ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )
1080	3.597	1170	3.836
1085	3.612	1175	3.847
1090	3.627	1180	3.857
1095	3.642	1185	3.868
1100	3.657	1190	3.878
1105	3.671	1195	3.888
1110	3.685	1200	3.898
1115	3.699	1205	3.907
1120	3.713	1210	3.916
1125	3.726	1215	3.925
1130	3.740	1220	3.934
1135	3.752	1225	3.942
1140	3.765	1230	3.950
1145	3.778	1235	3.958
1150	3.790	1240	3.966
1155	3.802	1245	3.973
1160	3.813	1250	3.980
1165	3.825		

Table 23. NaCl electrical conductance studies critically examined and comparisons with [35] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
161	Arndt (1906)	no temperature	range overlap
82	Sandonnini (1920)	-1.24% (1223 K)	-3.93% (1123 K)
162	Ryschkewitsch (1933)	1.10% (1213 K)	-8.62% (1103 K)
131	Edwards et al. (1952)	0.22% (1073 K)	7.73% (1373 K)
163	Huber et al. (1952)	-0.07% (1085 K)	-0.64% (1215 K)
135	Vereschetina, Luzhneya (1954)	no temperature	range overlap
164	Winterhager, Werner (1956)	-0.65% (1188 K)	2.62% (1278 K)
38	Van Artsdalen, Yaffe (1955)	0.05% (1100 K)	-3.78% (1075 K)
165	Poincare (1890)	no temperature	range overlap
166	Arndt, Gessler (1908)	no temperature	range overlap
167	Biltz, Klemm (1924)		graphical
168	Grothe (1949)	no temperature	range overlap
145	Markov, Prizyazhnyi (1962)		graphical
32	Ketelaar, Meenaut (1972)	0.02% (1273 K)	1.23% (1103 K)
169	Beloserski, Freidini (1941)	-3.26% (1123 K)	-6.57% (1073 K)
170	Nikolic, Grjotheim, Øye (1970)	0.54% (1135 K)	0.88% (1095 K)
171	Barzakovskii (1940)	0.37% (1179 K)	-0.44% (1197 K)
172	Kamenetzky, Shevlyakova (1962)	-2.77% (1223 K)	-7.13% (1073 K)



Table 23. NaCl electrical conductance studies critically examined and comparisons with [35] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
173	Balakir, Bushuev, Kudryaytsev (1968)	-0.99% (1573 K)	-4.83% (1323 K)
174	Lee, Pearson (1945)	1.31% (1123 K)	3.07% (1223 K)
175	Danek, Matiasovsky, Lillebuen (1971)	1.18% (1198 K)	1.18% (1198 K)
176	Matsumura, Mizuno, Nishihara (1966)	-1.47% (1113 K)	-1.51% (1153 K)
177	Bizouard (1961)	0.36% (1073 K)	0.47% (1123 K)
178	Matsumura, Mizuno, Nishihara (1967)	-0.97% (1166 K)	-1.21% (1106 K)
179	Argyriades (1954)	-2.15% (1146 K)	-5.83% (1083 K)
180	Bondarenko, Strelets (1962)	-2.05% (1123 K)	-2.94% (1073 K)
181	Markov, Sherbakov (1939)	graphical	
182	Batashev (1935)		
183	Matiasovsky, Lillebuen, Danek (1972)	0.79% (1120 K)	3.44% (1275 K)
184	Clarke, Story (1957)	-0.31% (1158 K)	1.59% (1160 K)
201	Carmichael, Flengas (1979)	-0.02%	-0.64%
33	Zuca, Olteanu (1975)	-0.05% (1218 K)	0.37% (1142 K)
35	Emons, Brautigam (1975)	0.00%	0.00%
34	Ejima, Shimakage, Sato (1976)	0.02% (1181 K)	0.25% (1128 K)
36	Suski, Gadek, Stachowitz (1976)	1.47% (1077 K)	3.30% (1180 K)

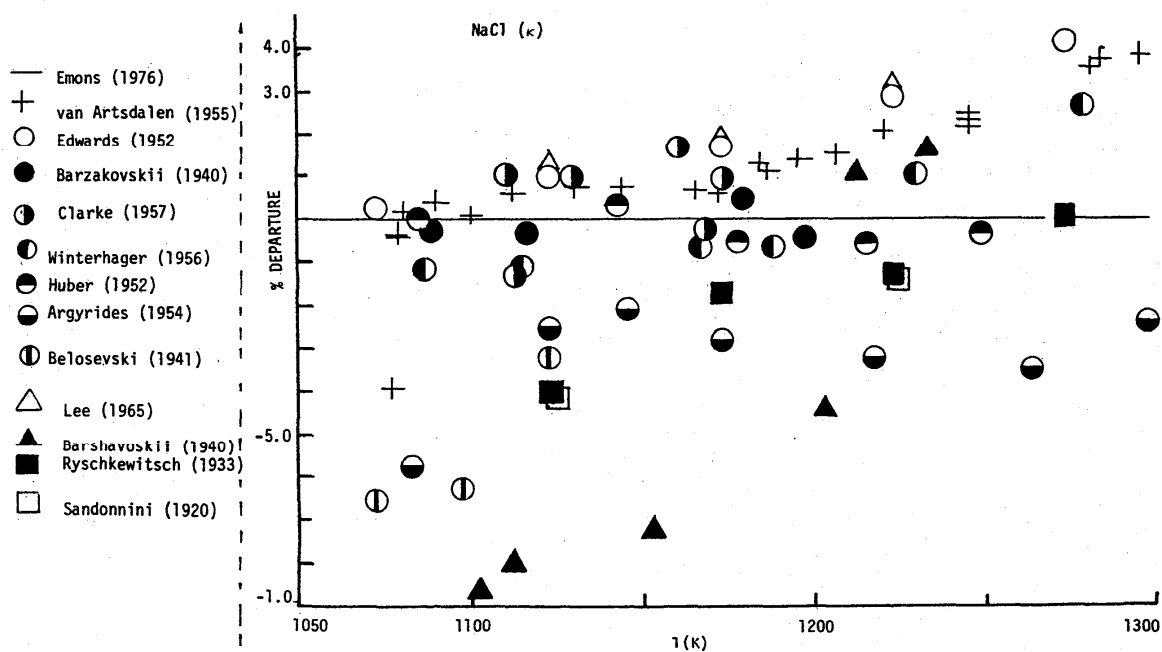


Figure 23 NaCl conductance data for the period 1920-1960, shown as a percent departure analysis with the Emons and Brautigam data base (1976) as comparison standard.

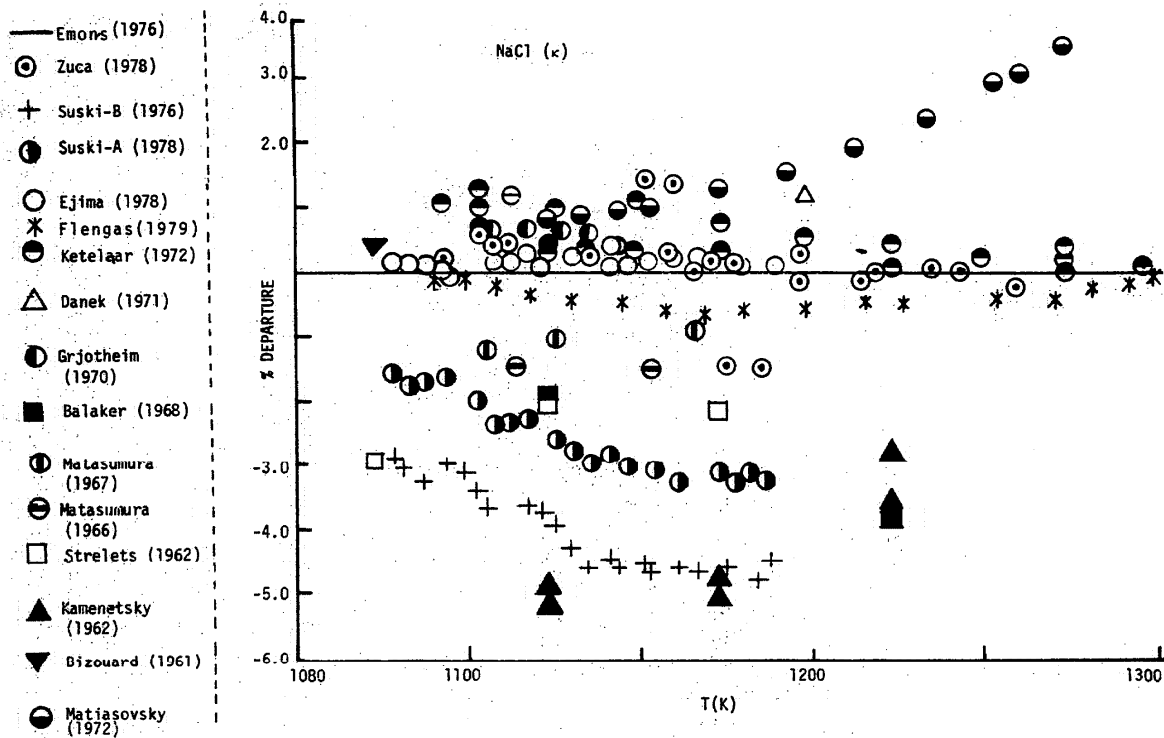


Figure 24 NaCl conductance data for the period 1960-1979, shown as a percent departure analysis with Emons and Brautigan data base (1976) as comparison standard.

Table 24.1. Parameters for NaCl viscosity equations (6.2.4a,b,c)

kelvin expo	$A \times 10^3$ (mN s m <sup>-2</sup> )		E (cal mol <sup>-1</sup> ) <sup>a</sup>	temp. range
	89.272		5248.5	1080 - 1210 K
kelvin power	a (mN s m <sup>-2</sup> )	$-b \times 10^3$ (mN s m <sup>-2</sup> K <sup>-1</sup> )	$c \times 10^6$ (mN s m <sup>-2</sup> K <sup>-2</sup> )	temp. range
	3.4877	2.6966	0.3319	1080 - 1210 K
Celsius	a (mN s m <sup>-2</sup> )	$-b \times 10^3$ (mN s m <sup>-2</sup> deg <sup>-1</sup> )	$\gamma \times 10^6$ (mN s m <sup>-2</sup> deg <sup>-2</sup> )	
	2.7796	2.4880	0.3819	810° - 940° C

(a) E is expressed in units of cal mol<sup>-1</sup> for ease of reference relative to the general practice in this work; 1 cal mol<sup>-1</sup> = 4.184 J mol<sup>-1</sup> (see table 2)

Table 24.2 NaCl viscosities<sup>a</sup> calculated from kelvin equation (6.2.4a)

T (K)	$\eta$ (mN s m <sup>-2</sup> )	T (K)	$\eta$ (mN s m <sup>-2</sup> )
1080	1.030	1150	0.888
1085	1.019	1155	0.879
1090	1.007	1160	0.870
1095	0.996	1165	0.862
1100	0.985	1170	0.854
1105	0.975	1175	0.845
1110	0.964	1180	0.837
1115	0.954	1185	0.829
1120	0.944	1190	0.822
1125	0.934	1195	0.814
1130	0.925	1200	0.807
1135	0.915	1205	0.799
1140	0.906	1210	0.792
1145	0.897		

(a) 1 mN s m<sup>-2</sup> = 1 cp = 1 mPa s; (see table 2)

Table 25. NaCl viscosity studies critically examined and comparisons with [46,57] as the recommended reference data base

Ref.	Authors	Min. departure	Max. departure
185	Fawsitt (1908)	17.86% (1169 K)	35.80% (1114 K)
41	Dantuma (1928)	5.16% (1243 K)	48.85% (1089 K)
42	Murgulescu, Zuca (1963)	12.33% (1243 K)	39.63% (1085 K)
186	Ogawa (1950)	graphical	
187	Bondarenko, Strelets (1965)	0.44% (1186 K)	16.35% (1088 K)
188	Barzakovskii (1940)	3.94% (1273 K)	53.83% (1073 K)
189	Vereshchetina, Luzhnaya (1954)	29.47% (1148 K)	43.88% (1098 K)
190	Murgulescu, Zuca (1965)	17.68% (1173 K)	34.31% (1093 K)
191	Matsumura, Mizuno, Nishihara (1967)	28.31% (1143 K)	34.49% (1113 K)
127	Dumas, Grjotheim, Hogdahl, Øye (1970)	-3.38% (1085 K)	-7.82% (1129 K)
193	Moynihan (1967)	47.73% (1090 K)	47.73% (1090 K)
48	Zuca and Borcan (1977) (torsional)	0.37% (1134 K)	3.44% (1220 K)
47	Zuca and Costin (1978) (capillary)	0.23% (1193 K)	3.01% (1213 K)
43	Ejima, Yoko (1976)	13.92% (1110 K)	16.05% (1174 K)
45	Suski, Galka, Stachowicz (1975)	25.80% (1163 K)	48.18% (1082 K)
46,57	Tørklep and Øye (1978)	0.00%	0.00%
44	Matiasovsky, Votova (1975)	15.24% (1163 K)	49.37% (1088 K)
44	Matiasovsky, Votova (1979)	0.17% (1177 K)	1.70% (1085 K)

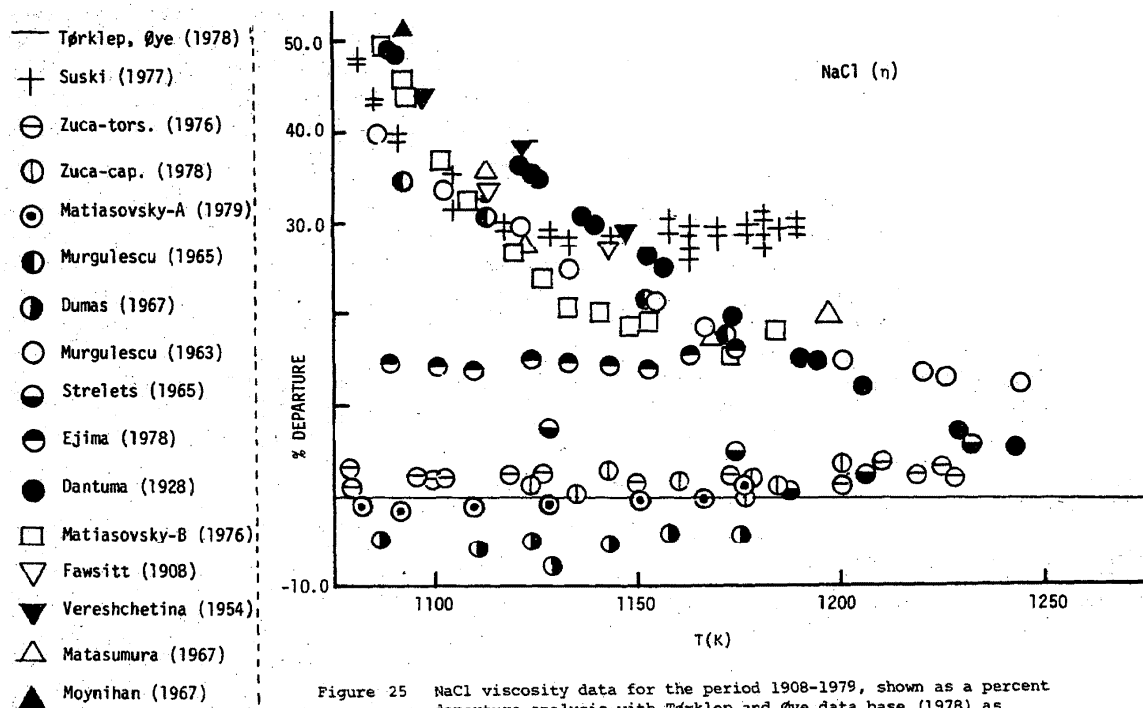


Figure 25 NaCl viscosity data for the period 1908-1979, shown as a percent departure analysis with Tørklep and Øye data base (1978) as comparison standard.

### 6.3. Summary and Recommendations

Based on the preceding, the following summarizes the accomplishments of this program, and the recommendations that may be advanced.

1. Density Data: Molten  $\text{KNO}_3$ : The density data have been upgraded to calibration-quality reference levels for the temperature range of 345°–455 °C. Molten NaCl: The results support the 1968 NSRDS recommendations for the temperature range of 805°–1030 °C within the limits of experimental accuracies.

The Archimedean density technique is recommended for such measurements as the most accurate.

2. Surface Tension Data: Molten  $\text{KNO}_3$ : The surface tension data have been upgraded to calibration-quality reference levels for the temperature range of 345°–490 °C. Molten NaCl: The results indicate that the accuracy limits for this data set must be changed to  $\sim \pm 1.5\%$  rather than the earlier estimate of  $\sim \pm 0.1\%$  advanced with 1969 NSRDS recommendations for the temperature range of 805°–970 °C.

Both the maximum bubble pressure and the flat-pin detachment techniques are recommended as best techniques for highest accuracies.

3. Electrical Conductance Data: Molten  $\text{KNO}_3$ : The 1972 NSRDS recommended values for the temperature range of 340°–505 °C are supported within the limits of experimental accuracies. There are differences in the results from the participating laboratories, and these should be

resolved through additional measurements before the calibration quality of this data set is secure. Molten NaCl: The conductance data have been upgraded to calibration-quality reference levels for the temperature range of 805°–980 °C. The 1968 NSRDS recommendations receive firm support in the range of 805°–850 °C, but are significantly in error at higher temperatures (e.g.,  $\sim 3.5\%$  at  $\sim 1020$  °C) and must be revised.

Classical high precision ac conductance measurement techniques are recommended for best measurements. Both the twin-capillary dipping and U-type capillary designs are recommended for conductance cell designs (to define moderately large cell constants); suitable precautions to minimize electrode polarization errors are essential for measurements of highest accuracies.

4. Viscosity Data: Molten  $\text{KNO}_3$ : The viscosity data have been upgraded, and this gently shifts the 1969 NSRDS recommended values for the temperature range of 340°–490 °C to  $\sim 1\%$  higher values. While the results from the capillary techniques and the torsional pendulum techniques are in agreement within the limits of experimental accuracies, the temperature dependences of the results are different (differing slopes). This point should be resolved to firm up the present recommendations as calibration-quality reference data. For the present, the recommendations for  $\text{KNO}_3$  are based on results from the capillary measurement technique. Molten NaCl: The values advanced in the 1968 NSRDS recommendations have been shown to be in serious error (e.g.,  $\sim 50\%$ , at  $\sim 810$  °C,  $\sim 10\%$  at  $\sim 1000$  °C). This

data set has been upgraded through the present program to calibration-quality levels in the temperature range of 810°–940°C. The present recommendations are based on the torsional pendulum technique with immersed cylindrical shapes.

Both the classical capillary and the torsional pendulum techniques are equally applicable for molten salts viscosity measurements at moderately high temperatures (up to ~500 °C). At higher temperatures the torsional pendulum technique is recommended. With immersed spherical shape, the accuracy limits of this technique appear to be  $\sim \pm 3\%$ . With immersed cylindrical shapes and optimum procedures this technique appears capable of accuracies to  $\sim 0.2\%$  for molten salts. In more routine use, the accuracy limits appear  $\sim 1\%$ . Relative to the torsion wire, the Kestin alloy [92% Pt, 8% W] appears superior and is recommended. The major requirements for precision, as summarized by Kestin [200], are:

Suitable control of pressure and temperature for maintaining a near-equilibrium state

Reducing the effects of unavoidable instabilities to negligible levels, for hydrodynamic stability

A faithful mathematical model of the process and an accurate solution in the form of an adequate working equation and a complete set of corrections

Detection of the major effect with high resolution, and operation at its lowest value consistent with the desired precision and sensitivity of the working equation

Insight on various design parameters and procedural techniques relative to error sources in molten salts viscosity measurements have been significant accomplishments of this program. Some additional points nevertheless remain, and should be noted as areas for further work. For example, at moderately high temperatures (340°–450 °C;  $\text{KNO}_3$ ) the short capillary technique for molten salts viscosities gives results in close accord with the classical capillary techniques and the torsional methods ( $\sim \pm 3\%$ ). By contrast at the higher temperatures (810°–1000 °C;  $\text{NaCl}$ ) the data set from the short capillary technique differs greatly ( $\sim \pm 50\%$ ) from the data sets of the classical capillary techniques and the torsional methods. The resolution of such outstanding points remain a need, and further measurements are recommended in this area of materials science.

## 7. Acknowledgements

The concept of the Standards Program project evolved through discussions with colleagues, particularly Stefania Zuca (Bucharest), Harald A. Øye (Trondheim), and Howard J. White, Jr. (Washington).

The critical analyses of the data and value judgements were part of a program supported at this laboratory by the Office of Standard Reference Data, U.S. Department of Commerce, National Bureau of Standards, Washington, D.C. Much of the work at RPI and at Bucharest on the Standards Program was accomplished in the period of 1973–1976, under the support of a cooperative research program with

Bucharest by the National Science Foundation (USA) and the National Council of Science and Technology (Rumania) under the Inter-Academy of Sciences Agreement. Stefania Zuca and G. J. Janz were co-directors for the Rumanian and U.S. sides, respectively, and R.P.T. Tomkins was co-principal investigator at RPI.

The continued interest of Academician I. G. Murgulescu (Bucharest) throughout this period was greatly appreciated, and discussions with Professor J. Kestin (Brown University), particularly on matters of precise and accurate viscosity measurements were most helpful. I wish to express a special thanks to colleagues at each of the participating centers for the facilities so generously volunteered for these measurements, and the discussions that led to the final results.

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