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The elastic constants of sodium chloride and rubidium chloride in the range 140–300 K

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Abstract. Measurements of the adiabatic elastic constants of NaCl from 190 to 300 K and of RbCl from 140 to 300 K have been made using the 'sing-around' system to an absolute accuracy of $\pm 0.2\%$ and with a relative accuracy of $\pm 0.05\%$. The elastic constants of NaCl agree with other recent measurements. The measurements of the elastic constants of RbCl agree with other work for C_{11} and C_{44} but the variation of C_{12} with temperature disagrees with the other measurements. The decrease in C_{12} of RbCl with decreasing temperature is in accordance with the variation of C_{12} in other alkali chlorides. The reasons for the disagreement of the variation of C_{12} are examined and it is shown that the observed decrease is real.

1. Introduction

A valuable insight into the fundamental nature of solids is provided by measurements of the elastic constants. Extension of these measurements to include the dependencies upon both temperature and pressure provides information of the anharmonicity of the solid and permits further correlation with complementary data obtained from measurements of thermal expansion and specific heat. Accurate determinations of elastic constants may be obtained from measurements of appropriate wave velocities for which present techniques offer relative accuracies of a few parts per million. In such studies the alkali halides have featured prominently, although in the cases of certain materials, no complete set of experimental data is available. For example there are, as yet, no experimental values of the thermal expansion of rubidium chloride below room temperature. However, the elastic constants have been calculated from experimental measurements by Marshall *et al.* (1967) and by Lewis *et al.* (1967) using estimated thermal expansion data. In this paper we describe a method of estimating the thermal expansion based upon the Hanneman-Gatos relationship used by Sirdeshmukh (1967) and use it together with new measurements of the appropriate transit times to calculate the elastic constants of rubidium chloride.

2. Experimental details

2.1. Propagation of ultrasonic waves

The propagation of ultrasonic waves has long been used in the experimental investigation of the properties of solids. It may be shown that the wave velocities calculated from the measured transit times are related to the second-order elastic constants (Kolsky 1953, Fedorov 1968). In materials with cubic symmetry there are three independent second-order elastic constants C_{11} , C_{12} and C_{44} . Considering wave propagation along the $\langle 100 \rangle$ direction these elastic constants are related to the ultrasonic wave velocities by the equations

$$\rho V_1^2 = C_{11} \quad (1)$$

$$\rho V_2^2 = \rho V_3^2 = C_{44} \quad (2)$$

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where ρ is the density of the material. Examination of the elastic constants involved shows that V_1 is a longitudinal wave velocity and that V_2 and V_3 are transverse wave velocities. In the $\langle 110 \rangle$ direction the equations for the wave velocities are

$$\rho V_4^2 = \frac{1}{2}(C_{11} + C_{12}) + C_{44} \quad (3)$$

$$\rho V_5^2 = C_{44} \quad (4)$$

$$\rho V_6^2 = \frac{1}{2}(C_{11} - C_{12}). \quad (5)$$

In this direction V_4 is a longitudinal wave, V_5 is a transverse wave with particle motion in the direction $\langle 001 \rangle$ and V_6 is a transverse wave with the particle motion in the direction $\langle 1\bar{1}0 \rangle$. In the present investigation the velocities V_1 , V_5 and V_6 were obtained from the measured transit times σ_1 , σ_5 and σ_6 . The elastic constants were then determined from the equations

$$C_{11} = \rho V_1^2 \quad (6)$$

$$C_{44} = \rho V_5^2 \quad (7)$$

$$C_{12} = \rho V_1^2 - 2\rho V_6^2. \quad (8)$$

2.2. Transit time measurement

It was decided to use the ultrasonic sing-around system described by Forgacs (1960) for the measurement of the transit time of pulses of ultrasonic waves. This system was chosen because it is capable of:

- (i) The measurement of environmentally induced velocity changes of 1 part in 10^6 .
- (ii) The measurement of absolute velocities for the subsequent evaluation of the second-order elastic constants.

On the grounds of sensitivity only some of the available high frequency techniques meet the first requirement, namely:

- (i) The balanced demodulator system developed by Mason and Bommell (1956).
- (ii) The pulse superposition system described by McSkimin (1961, 1965).
- (iii) The improved sing-around system.

In any experiment, it is necessary to include a number of correction terms and the accuracy of these determines the final accuracy of the method. In all the above methods the necessary corrections to the measured transit times are due to phase changes on reflections, bond delay, diffraction effects and changes in electronic delays. The variation in diffraction effects and electronic delays are approximately the same in all three systems, except for the temperature dependence of the mercury delay line in the balanced demodulator system. The reflection phase change and the bond delays vary considerably with temperature, unless the transducer is operated at its resonant frequency. These phase changes may be determined by the method given by McSkimin (1961). The only system that does not require the determination of this rather uncertain correction for high accuracy is the improved sing-around system. Whilst using the sing-around for the absolute determination of transit time, difficulty was experienced in deciding on the number of cycles within the echo, from which the trigger pulse was being derived. This led to the adoption of the pulse superposition method for the absolute measurements, as no difficulty was then experienced when determining the transit time. Corrections for phase changes were not applied as the bonds were made very thin and the transducers operated at their resonant frequency. It was found that the absolute transit time could be determined to within better than 0.1% by this method. The sing-around system was then used to measure the temperature variation of the transit time caused by changes in the elastic constants and the dimensions.

2.3. The cryostat

The cryostat used (figure 1) was similar in design to that described by James and Yates (1965 b) except that the large pipe necessary for their optical system was replaced by four tubes to carry the signal leads for the sing-around system. The temperature of the specimen chamber of this cryostat was controlled by the automatic temperature controller described by Gluyas *et al.* (1970). The temperature of the specimen chamber was measured by an indium thermometer similar to that described by James and Yates (1964) and used successfully by James and Yates (1965 a). The thermometer was calibrated against a platinum resistance thermometer in a manner similar to that used by Kirkham and Yates (1968).

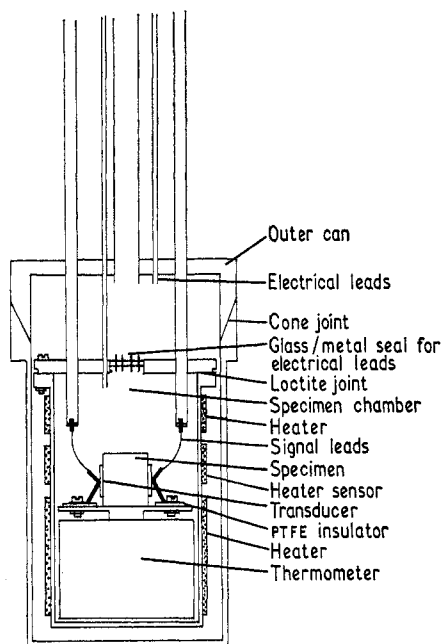


Figure 1. The cryostat. The outer can is immersed in liquid nitrogen or liquid helium.

2.4. Specimens

The specimen of sodium chloride was obtained from Hilger and Watts Ltd in the form of a single crystal and the material from which it was grown had impurities of less than 0.03%. The single-crystal specimens of rubidium chloride were obtained from New Metals and Chemicals Ltd and had a total impurity of less than 22 parts per million. The specimen of sodium chloride was a cleaved single crystal with {100} faces and sides 3 cm, 3 cm and 2 cm. From it a cube with 2 cm sides was cut with two pairs of faces of {110} and one pair of {100} faces. The cutting was performed by means of a tungsten wire loaded with a paste of fine emery powder and oil. Similar faces were required for the rubidium chloride specimen, but here two specimens were used. Both of these were cleaved from a single large crystal along {100} cleavage planes. One specimen was 2 cm long with a square cross section of 1 cm side, whilst the other was slightly longer and had ends cut at 45° to its length to give {110} faces.

After cutting, all the specimens were ground and polished to give flat faces. The faces were aligned to the required axes by taking back reflection, or Laué x-ray photographs of the faces and the degree and direction of misorientation from that required was measured from the photographs using a Gireniger chart. By preferential grinding and polishing and repeated x-ray photographs the specimen faces were aligned to within 0.5° of the required direction. When the first face of each specimen had been aligned to this accuracy,

it was polished to give a bright clear finish. The opposite face was then ground flat and parallel to the first face. Flatness and parallelism were checked using a large dial gauge, with 0.0001 inch divisions, mounted above a surface plate. When this second surface was considered finished, its orientation was checked using the x-ray technique to ensure that there was no twinning within the crystal.

2.5. Transducer bonding

To propagate ultrasonic waves in a specimen, the acoustic energy generated by a piezo-electric transducer must first be coupled into it. This was accomplished by bonding the transducer to the specimen using a variety of materials such as glues, waxes, greases, silicone fluids, etc. since no one material gave satisfactory results over the whole temperature range considered. Main causes of bond failure at high temperatures arise from the diminished viscosity of the above materials failing to support the shear waves, whilst at low temperatures it is due to differential thermal contraction between the specimen and bond, and the bond and transducer. The latter problem is especially critical in the high thermal expansion alkali halides and prevented the present measurements from being made below 140 K. Measurements have been made below 140 K on the alkali halides using natural gas or 4-methyl, 1-pentane but these materials must be applied at low temperatures and this was not possible for the two transducers needed for the sing-around system. A number of bond materials were tried on a specimen of sodium chloride with varying degrees of success as shown in table 1.

Table 1. Transducer bond materials and their useful range for NaCl

Material	Temperature range (K)
Dow Resin 276-V9	250-300
Glycerine	210-280
Edwards High Vacuum Silicone Grease	189-290
Midland Silicone Reasil 7 Silicone Compound	170-290
Apiezon N Vacuum Grease	170-290
Dow Corning 200 Fluid 12500 cS viscosity	134-210
Midland Silicone Fluid 1×10^6 cS and 5×10^6 cS	150-230
Fisher Nonaq Stopcock Grease	150-290

The measurements on sodium chloride and rubidium chloride were made using Dow Corning 200 silicone fluid from 134 K to 200 K above which it became too fluid to allow shear waves to be supported across it. From 200 K to 280 K Apiezon N grease was used as a bond material.

3. Results

3.1. Length, thermal expansion and density of specimens

The length at room temperature of the various specimens used is given in table 2.

The density of 2.1644 g cm^{-3} for sodium chloride given by Bartels and Schuele (1965) was used in the calculations of the elastic constants from the velocities. The density of rubidium chloride used was 2.8172 g cm^{-3} at 300 K. This was calculated from the lattice spacing of 6.4810 \AA given by the ASTM Powder Diffraction File and the molecular weight of 120.92.

To correct for the thermal expansion of sodium chloride the linear thermal expansion coefficients of Rubin *et al.* (1951) were used. For rubidium chloride the linear expansion coefficient has not been determined at low temperatures. However, Deshpande and Sirdeshmukh (1961) have measured it from 20°C to 190°C and give a mean value of $38.13 \times 10^{-6} \text{ degC}^{-1}$. Bailey and Yates (1967) have compared their low temperature results for calcium fluoride, barium fluoride and strontium fluoride with those of five other

Table 2. Length of specimens used

Specimen	Length (cm)	Temperature (K)
NaCl [001]	2.1495	296.85
NaCl [110]	1.9732	296.85
RbCl [001]	1.9765	294.95
RbCl [110]	1.0667	294.85

workers, finding excellent agreement between all the results except those of Deshpande and Sirdeshmukh, whose results differ by up to 13%. For this reason the room temperature thermal expansion of rubidium chloride was estimated from the Hanneman–Gatos relationship (Hanneman and Gatos 1965) modified by Sirdeshmukh (1967) to the form

$$\alpha V = K\chi_T$$

where α is the linear expansion coefficient, V is the molar volume, K is a constant and χ_T is the compressibility. Sirdeshmukh has shown that αV plotted against χ_T for the cubic metallic elements and the alkali halides result in straight lines. However, he used high temperature data for the linear expansion coefficients of the alkali halides and obtained a scatter of up to 10%. This graph for the alkali halides has been replotted for this investigation in figure 2 using more recent values of α at 280 K. The linear expansion coefficients

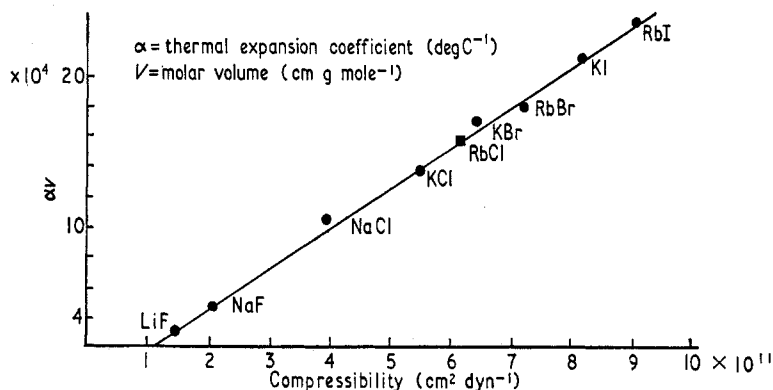


Figure 2. The Hanneman–Gatos relation.

used are due to James and Yates (1965 a) for NaF and RbBr, Yates and Panter (1962) for LiF, NaCl, KCl, KBr and KI and to White (1965) for RbI. The compressibilities were calculated from the elastic constant data of Haussuhl (1960) by substitution in the equation

$$\chi = \sum_{ij=0}^3 S_{ij} \quad (9)$$

where $S_{ij}C_{jk} = \delta_{ik}$.

Equation (9) simplifies to

$$\chi = 3(S_{11} + 2S_{12}) \quad (10)$$

for cubic materials.

The scatter of these new points plotted on the graph is lower than that of the original data. The deviation is less than 3% of the midpoint curve whereas the previous data deviates by up to 10%.

Using the uncorrected lengths, the velocity and elastic constants at 280 K were calculated from the measured transit times. The compressibility calculated from these was used to estimate the linear expansion coefficient at 280 K using figure 2. This gave a value for α of $35.4 \times 10^{-6} \text{ deg C}^{-1}$ as compared with the average high temperature value of Deshpande and Sirdeshmukh (1961) of $38.13 \times 10^{-6} \text{ deg C}^{-1}$.

Table 3. Linear thermal expansion coefficient of RbCl

T (K)	$\alpha \times 10^6$ (K ⁻¹)	T (K)	$\alpha \times 10^6$ (K ⁻¹)
10	—	160	30.8
20	1.1	170	31.1
30	5.0	180	31.5
40	10.4	190	32.0
50	15.6	200	32.4
60	19.2	210	32.8
70	21.9	220	33.2
80	23.9	230	33.6
90	25.4	240	34.0
100	26.6	250	34.4
110	27.5	260	34.7
120	28.2	270	35.1
130	28.9	280	35.4
140	29.5	290	35.8
150	30.1	300	36.1

Plotting this value for α at 280 K on the graph of α against temperature together with the expansion curves for RbI and RbBr the expansion curve for RbCl was inferred by examining the shapes of the other two. The values of the linear expansion coefficient taken from this curve at 10 K intervals are given in table 3. It is estimated that the uncertainty in the linear expansion coefficient at 280 K was less than 3%. The values of α in table 2 were used in the calculation of the velocities at all temperatures for RbCl.

3.2. Elastic constants and their accuracy

The elastic constants of sodium chloride are plotted in figure 3. The measured transit times and the calculated elastic constants of rubidium chloride are given in table 4. The values of C_{12} at the temperatures given in the table were calculated from values of the appropriate transit times σ obtained by plotting graphs of transit time against temperature. The total absolute error in the measured velocities for all the specimens was estimated to be less than $\pm 0.2\%$, the largest contributions being due to the uncertainty in the length

Table 4. Transit time and elastic constants of RbCl

Temperature (K)	Transit time σ_1 (μ s)	$C_{11} \times 10^{-11}$ (dyn cm ⁻²)	Temperature (K)	Transit time σ_5 (μ s)	$C_{44} \times 10^{-11}$ (dyn cm ⁻²)
298.45	5.52660	3.6053	298.31	8.28616	0.46711
290.94	5.50910	3.6291	290.99	8.27960	0.46798
281.14	5.48756	3.6589	281.32	8.27070	0.46915
273.15	5.46500	3.6899	273.15	8.26300	0.47009
269.96	5.46293	3.6935	269.23	8.26033	0.47054
261.00	5.44310	3.7216	261.01	8.25426	0.47136
251.00	5.42366	3.7497	250.42	8.24736	0.47232
241.22	5.40003	3.7837	240.93	8.24116	0.47319
230.16	5.38003	3.8134	229.99	8.23080	0.47454
220.40	5.35476	3.8506	220.34	8.22326	0.47558
210.61	5.33310	3.8830	210.53	8.21596	0.47659
200.24	5.31330	3.9135	200.80	8.20820	0.47763
190.06	5.29416	3.9432	190.02	8.19990	0.47877
179.34	5.27260	3.9769	179.05	8.19120	0.47995
169.75	5.25210	4.0092	169.49	8.18430	0.48078
158.99	5.23293	4.0399	158.53	8.17650	0.48198
149.44	5.21380	4.0708	149.58	8.16943	0.48295
140.10	5.19626	4.0984	140.18	8.16246	0.48392

Table 4—continued

Temperature (K)	Transit time σ_6 (μ s)	$C_{12} \times 10^{-11}$ (dyn cm ⁻²)
298.78	4.63923	0.62621
291.31	4.62140	0.62500
281.69	4.59596	0.61991
273.15	4.57200	0.61940
269.68	4.56086	0.60788
260.99	4.53866	0.60269
250.97	4.51500	0.59948
241.01	4.49213	0.59653
229.96	4.46610	0.59222
220.13	4.44260	0.58749
209.15	4.41420	0.57849
200.54	4.39480	0.57509
189.75	4.36800	0.56634
179.09	4.34253	0.55913
168.80	4.32163	0.55689
158.72	4.29900	0.55141
149.28	4.27866	0.54716
139.94	4.25580	0.53653

Table 5. The errors in the calculated elastic constants

Material	Elastic constant	Relative error (%)	Absolute error (%)
NaCl	C_{11}	± 0.1	± 0.5
	C_{44}	± 0.1	± 0.5
	C_{12}	± 3.5	± 7.0
RbCl	C_{11}	± 0.1	± 0.5
	C_{44}	± 0.1	± 0.5
	C_{12}	± 3.5	± 7.0

measurements and the transit time measurements using the pulse superposition technique. Superimposed on this was the $\pm 0.05\%$ relative error for all velocities other than at 273 K due to uncertainties in the change of transit time, as measured by the sing-around system and the temperature. The scatter of the measured points is less than the estimated relative error. From these errors in the velocity, the errors in the elastic constants were estimated and are given in table 5.

3.3. Elastic constants of sodium chloride

The elastic constants of sodium chloride plotted against temperature are shown in figure 3, together with the results of the measurements of Lewis *et al.* (1967), Overton and Swim (1951), Bartels and Schuele (1965), Haussuhl (1960), Gluyas (1967), Fugate and Schuele (1966), Slagle and McKinstry (1967) and of Hart (1968). The room temperature values of all three independent elastic constants have a spread of approximately 3%, with the present results lying towards the centre of the range. The present results agree within the estimated errors with the four previous investigations of the temperature dependence of the elastic constants below 300 K excepting C_{12} due to Overton and Swim; their value of C_{12} at 190 K is 10% less than the present results. However, the results of Fugate and Schuele and of Lewis *et al.* also indicate a value of C_{12} some 12% higher than those due to Overton and Swim at the lowest temperature. No results were obtained below 190 K for sodium chloride owing to specimen damage whilst cooling. However the agreement amongst the present results and the previously published data between 190 K and 300 K confirm that the apparatus is functioning correctly.

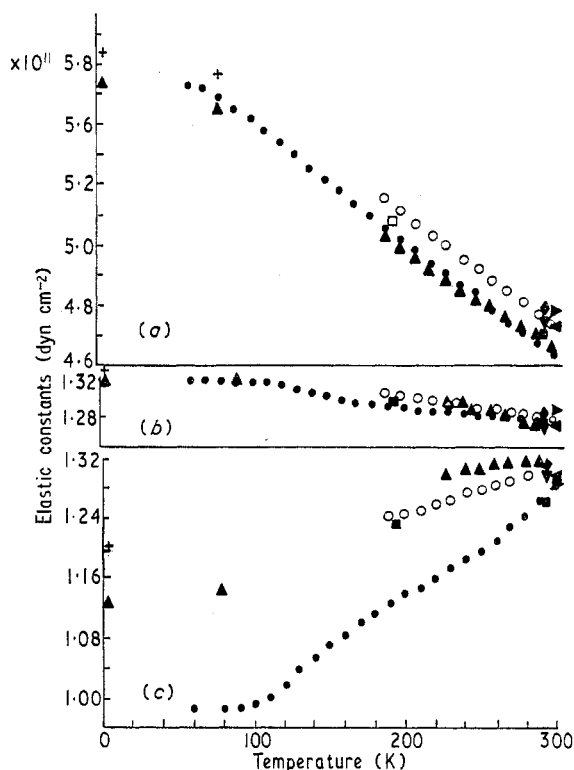


Figure 3. The elastic constants of sodium chloride (a) C_{11} , (b) C_{44} , (c) C_{12} . ○ Present work; ▲ Lewis *et al.* (1967); ● Overton and Swim (1951); ■ Bartels and Schuele (1965); ▼ Haussuhl (1960); ♦ Gluyas (1967); + Fugate and Schuele (1966); ► Slagle and McKinstry (1967); ◄ Hart (1968).

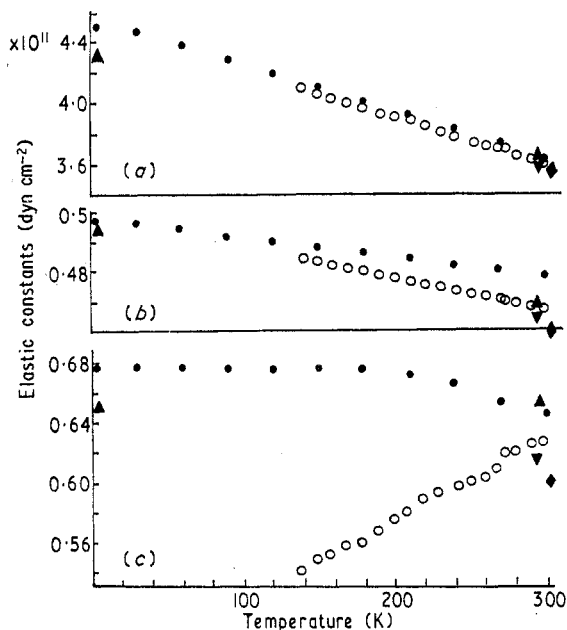


Figure 4. The elastic constants of rubidium chloride. (a) C_{11} , (b) C_{44} , (c) C_{12} . ○ Present work; ▲ Lewis *et al.* (1967); ● Marshall *et al.* (1967); ▲ Haussuhl (1960); ♦ Bergmann (1957).

3.4. Elastic constants of rubidium chloride

The elastic constants of rubidium chloride plotted against temperature are shown in figure 4 with the results of Lewis *et al.* (1967), Marshall *et al.* (1967), Haussuhl (1960) and Bergmann (1957). The values of all three independent elastic constants are in agreement within experimental accuracy at room temperature and there is agreement about the temperature variation of C_{11} and C_{44} . However, the present work gives a value for the elastic constant C_{12} lower than that of Marshall *et al.* by 21% at 150 K. It is felt that their quoted error of $\pm 3\%$ in C_{12} is an underestimate, since, using their estimated errors in $\rho VT^2 = \frac{1}{2}(C_{11} - C_{12})$ and C_{11} of $\pm 1\%$ and $\pm 0.8\%$ respectively it may be shown that the resultant error in C_{12} varies from $\pm 10\%$ to $\pm 15\%$. To correct for thermal expansion Marshall *et al.* used the values of Deshpande and Sirdeshmukh (1961) for the room temperature value of the coefficient of linear expansion. They estimated its temperature dependence from the Gruneisen relation using an estimated specific heat curve. For this reason it is not possible to assess the effect that their thermal expansion correction has on the value of C_{12} . However, it may be stated that a small error in the velocity for the transverse wave in the [110] direction produces a large error in the value of C_{12} calculated from it, as may be seen from the data in table 5.

The results of Marshall *et al.* indicate a rise in C_{12} with decreasing temperature, those of Lewis *et al.* at 4.2 K and 295 K indicate that C_{12} is constant, whilst the present results show a decrease with decreasing temperature. Since the relative error of the present results is $\pm 3.5\%$ compared with up to $\pm 15\%$ for the previous measurements which were made absolutely at each temperature, it is felt that the indicated decrease of C_{12} with decreasing temperature is real.

Similar decreases in C_{12} have been found for sodium chloride, potassium chloride and lithium chloride. Figure 3 shows the decrease in C_{12} with decreasing temperature in NaCl. The results of Norwood and Briscoe (1958) for potassium chloride also indicate a decrease in C_{12} with decreasing temperature. The measurements of C_{12} for lithium chloride by Marshall *et al.* (1967) also show a decrease in C_{12} down to about 160 K. It would seem therefore that a decrease in C_{12} for rubidium chloride with decreasing temperature is highly probable.

4. Conclusion

The specific heat of rubidium chloride has been measured recently by Kirkham and Yates (1968). However, it is not possible to calculate C_v , the specific heat at constant volume, accurately from C_p , the specific heat at constant pressure, without the experimentally determined expansion coefficient α , though approximate values of C_v from the estimated thermal expansion could be obtained. Since the thermodynamic analysis in terms of the Gruneisen parameter depends on α and C_v this was not pursued as the base data was not adequate. Kirkham and Yates have pointed out that rubidium chloride is a particularly interesting alkali halide and further accurate measurements of the elastic constants to lower temperatures and of the thermal expansion below 300 K would enable a full thermodynamic analysis of this material to be made.

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Note added in proof. The thermal expansion of RbCl has recently been measured by R. F. Cooper and B. Yates (private communication). The interpolated values used in this paper agree with their values within less than $\pm 3\%$ over the temperature range 40–260 K.

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