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Thermodynamic study of the condensed phases of $NaNO_3$, KNO_3 and $CsNO_3$ and their transitions ☆

T. Jriri *, J. Rogez, C. Bergman, J.C. Mathieu

Centre de Thermodynamique et de Microcalorimétrie du C.N.R.S., 26 Rue du 141ᵉᵐᵉ R.I.A., 13331 Cedex 3-Marseille, France

Abstract

The heat capacities of solid and liquid $NaNO_3$, KNO_3 and $CsNO_3$ and their enthalpies of transition have been measured by DSC. A review of the literature data and a comparison with the results of this investigation are presented.

Keywords: Binary system; Fusion; Heat capacity; Nitrate; Transition

1. Introduction

As part of a more complete thermodynamic study of the $NaNO_3$–KNO_3–$CsNO_3$ system, the pure compounds which represent the basic elements of the binary or ternary mixtures are first investigated. They will constitute the reference states in further thermochemical studies of the various solid and liquid solutions of this system. The final goal is to construct, using a computer program, a coherent representation containing all the available experimental values obtained, whether by thermodynamic measurements or by phase diagram evaluations.

For the pure nitrates, the heat capacity and the transition energies have already been measured by various methods. The drop method and adiabatic calorimetry have been used since the beginning of the twentieth century. In the 1970s, differential scanning calorimetry appeared and its increasing success with experimentalists is principally due to its relative easy manipulation.

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* Corresponding author.

☆ Dedicated to Hiroshi Suga on the Occasion of his 65th Birthday.

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In this paper, new measurements of the heats of transition and fusion, together with the heat capacities in both solid and liquid phases of $NaNO_3$, KNO_3 and $CsNO_3$, measured by a DSC method, are presented. A review of the literature is also given. The aim of this review is to determine the most reliable thermodynamic data to be introduced into future computer calculations of the ternary system.

2. Experimental

The calorimeter is a Perkin-Elmer DSC4. The well-known principle of the measurement is to record the differences between the enthalpy changes occurring in the sample and a reference heated at the same temperature rate ($\alpha = \mathrm{d}T/\mathrm{d}t$). The system involves two control loops: on the one hand, the sample and reference temperatures may increase at a chosen rate; on the other, when a temperature difference occurs between the sample and the reference concurrent with an exothermic or endothermic reaction in the sample, the second control loop adjusts the thermal power to the sample in order to reduce this difference to zero. A signal proportional to the difference between the energies provided per unit time to the sample and the reference ($\mathrm{d}\Delta H/\mathrm{d}t$) is then recorded versus the temperature (T).

The baseline characteristic of the calorimeter is first stored during a precalibration experiment without the sample and then subtracted from the calorimetric signal with the sample. Two distinct calibrations are performed for enthalpy or heat capacity measurements.

The samples are placed in platinum crucibles in the DSC apparatus. The sample masses used are between 1 and 3 mg. Thermograms are recorded between 350 and 750 K with heating rates of 10 and 40 K min^{-1} with pure argon as the purge gas. All the products were purchased from Aldrich ($CsNO_3$, 99.99%; KNO_3 and $NaNO_3$, 99.5% purity) and used without further purification.

In such experiments the main difficulty is to reproduce identical experimental conditions during the calibration and the measurement runs. The thermal resistance between the sample and the measurement cell must be as good as possible. In order to assure such a constraint, the nitrates are melted before each series inside the measurement cell and then slowly cooled to room temperature. In all cases the products are heated at most 50–70 K above the liquidus temperature to avoid thermal decomposition.

2.1. Transition enthalpy measurements

A transition enthalpy measurement consists of a computer integration of the corresponding peak observed in the thermogram $\mathrm{d}\Delta H/\mathrm{d}t = f(T)$, such as

$$\Delta H = \int_{\mathrm{peak}} \frac{1}{\alpha}\frac{\mathrm{d}\Delta H}{\mathrm{d}t}\,\mathrm{d}T$$

More currently, transition enthalpy measurements are performed by comparing, under the same thermal conditions, the areas under the recorded thermograms of the studied transition and of a phase transformation the energy of which is well known. In the present study, such a calibration of the apparatus is performed by melting 0.19 mmol of pure indium. This transition is used in the definition of a fixed point in the E.I.T.-90 at 429.7485 K [1]. The corresponding enthalpy is given as 3263.52 mol^{-1} [2].

2.2. Heat capacity measurements

The heat capacity measurement consists of a comparison between the baseline $(\mathrm{d}\Delta H/\mathrm{d}t)_0$ recorded with the empty crucible and the thermogram relative to the sample $(\mathrm{d}\Delta H/\mathrm{d}t)_\mathrm{S}$. The heat capacity is then calculated continuously versus the temperature as

$$C_p(T) = \frac{1}{\alpha}\left[\left(\frac{\mathrm{d}\Delta H}{\mathrm{d}t}\right)_\mathrm{S} - \left(\frac{\mathrm{d}\Delta H}{\mathrm{d}t}\right)_0\right]$$

In addition, a calibration on a pure α-alumina sample is performed. The present results are compared with the literature data [3] in Fig. 1. A good agreement is observed at ± 1.5% in the temperature range 400–800 K.

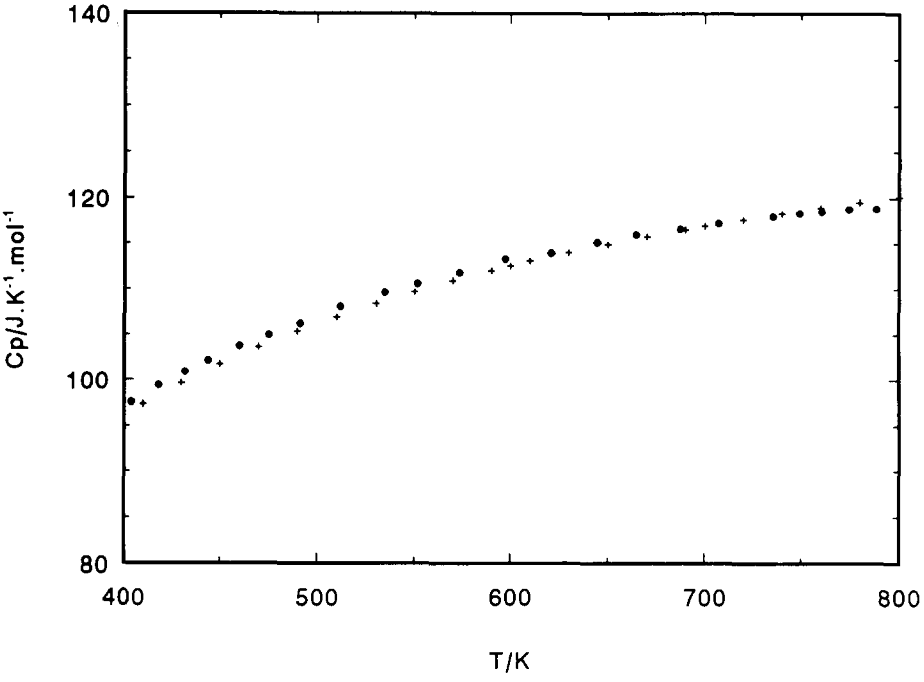

Fig. 1. Heat capacity of pure α-alumina: ●, this work; +, Ref. [3].

3. Results and discussion

Our results are reported in Tables 1 and 2. The fitted heat capacity measurements are shown in Table 3 and represented in Figs. 2–6. The following comparison concerns all the literature data found (Tables 2 and 3). Only the experimental results are mentioned; therefore, the compilations are not included.

In general, the scattering is much lower for the transition temperatures than for the enthalpy measurements. Overall, the determinations by adiabatic calorimetry and to a certain extent by differential scanning calorimetry when the heating rate is slow, are more reliable. The near-equilibrium conditions in these measurement methods are

Table 1
(a) Heat capacity measurements of $NaNO_3$

T/K	C_p/J mol^{-1} K^{-1}	T/K	C_p/J mol^{-1} K^{-1}	T/K	C_p/J mol^{-1} K^{-1}
364	103	536	209	394	1015
368	104	539	218	594	954
373	106	541	229	594	911
377	106	543	245	595	855
381	108	545	257	595	776
386	108	546	274	596	676
393	110	548	296	597	560
397	111	549	275	598	424
402	112	550	257	600	297
409	113	551	234	601	243
413	114	552	214	603	198
419	115	553	199	604	182
425	118	555	178	605	174
429	119	558	157	606	166
435	121	561	147	607	159
442	122	564	142	608	153
448	125	566	142	609	151
453	126	567	140	611	144
459	127	569	140	613	140
463	130	570	139	616	139
468	131	572	139	622	139
474	135	573	139	624	139
481	138	575	140	627	139
486	140	575	144	632	138
491	144	576	150	635	138
495	146	577	164	644	138
500	149	578	296	651	139
506	153	580	494	655	138
510	157	581	604	660	138
516	165	583	739	664	138
523	174	584	852	667	138
527	183	586	963	678	138
532	195	587	1020		

Table 1
(b) Heat capacity measurements of KNO_3

T/K	C_p/J mol^{-1} K^{-1}	T/K	C_p/J mol^{-1} K^{-1}	T/K	C_p/J mol^{-1} K^{-1}
362	107	435	214	612	704
368	108	437	178	613	881
373	109	440	156	614	971
378	110	442	149	616	982
385	112	444	141	617	958
391	113	447	134	622	727
396	113	457	127	623	521
401	114	464	127	625	348
404	114	468	127	628	210
406	115	473	127	629	182
408	116	484	128	631	164
409	124	489	128	633	152
410	138	499	129	636	145
412	180	511	129	642	142
413	212	522	130	653	143
414	242	535	132	656	143
416	285	549	133	666	143
418	334	560	134	672	143
420	374	573	137	678	143
422	409	584	138	684	143
423	414	595	140	689	143
425	406	602	142	697	143
427	376	605	144	704	142
429	341	607	146	708	142
431	300	608	199	711	142
432	263	610	499		

better than those of other techniques, e.g. drop calorimetry. High heating rate apparatuses cannot provide good thermodynamic data on phases whose stability domain is narrow, e.g. the β solid phase in $NaNO_3$. For this reason the temperatures and enthalpies of transition are better defined for KNO_3 and $CsNO_3$ than for $NaNO_3$. From this point of view we think that $NaNO_3$ is a poor choice as calibration material for scanning calorimetry. For the heat capacities of the ptassium and sodium nitrates, good agreement is found between most experimentalists. The results on $CsNO_3$ seem to be less reliable. In all cases, greater discrepancies are observed in the liquid state. The interpolation equations mentioned by the authors are reported in Table 3. When only discrete values are available an interpolation equation has been calculated.

3.1. *Solid–solid transitions (Table 2)*

The solid-state transition temperature for sodium nitrate given in the literature has a remarkably constant value (548.5–550 K) except for Nguyen-Duy and Dancy [31] and Dancy [34] who give respectively 535 and 530 K. Our result is 549 K. For

Table 1
(c) Heat capacity measurements of $CsNO_3$

T/K	C_p/J mol^{-1} K^{-1}	T/K	C_p/J mol^{-1} K^{-1}	T/K	C_p/J mol^{-1} K^{-1}
346	106	444	139	676	1410
359	109	451	139	681	1260
368	110	459	139	681	1120
374	111	470	140	681	932
385	113	484	141	682	697
394	115	494	143	683	502
405	118	507	144	685	309
412	121	523	145	687	206
417	122	543	146	688	171
420	127	560	146	690	154
422	136	578	147	692	148
423	145	591	149	699	146
424	176	606	148	701	146
425	237	624	148	705	146
426	381	637	148	712	146
426	523	653	149	717	146
427	626	663	151	722	146
428	670	668	156	725	146
430	530	669	166	728	146
431	404	671	248	736	147
433	245	672	372	741	146
434	205	673	614	748	146
436	174	674	865	753	146
437	155	675	1150	756	146
439	146	676	1340	757	146

potassium nitrate, a good agreement is found for 11 of the 13 determinations obtained by various methods (400.5–403 K). The average value is 402.2 K. Our result lies only 1.8 K above this value. For the caesium nitrate, our result is 1 K higher than the mean value of the three available data.

Among the eight enthalpic data of the second-order phase transition of $NaNO_3$, great discrepancy is observed but two groups of values may be distinguished. The first group lying around 1 kJ mol^{-1} is mainly determined by the drop method. The second group whose mean value lies at 3.84 kJ mol^{-1} is for determinations by adiabatic or differential scanning calorimetry. Our result is 3.62 kJ mol^{-1}. For KNO_3, the eleven literature values lie between 4.91 and 5.64 kJ mol^{-1}. Our determination is 0.14 kJ mol^{-1} lower than the average value of 5.2 kJ mol^{-1}. It is very close to the results obtained by differential scanning calorimetry of Rogers and Janz [36] and Westphal et al. [43]. Carling [37] who used the same method, gives 5.64 kJ mol^{-1} which is the highest literature value. For $CsNO_3$, our determination is 0.35 kJ mol^{-1} lower than the mean value over the three available literature values.

Table 2
Thermodynamic data for the solid–solid transition and the fusion of $NaNO_3$, KNO_3 and $CsNO_3$

T_{tr}/K	T_f/K	ΔH_{tr}/(J mol^{-1})	ΔH_f/(J mol^{-1})	Method[a]	Ref.
$NaNO_3$					
–	584	–	22396	MM	[5]
–	581	–	15439	DC	[6]
548.6	–	3770	–	AC	[9]
547	579	–	15049	AC	[10]
549.2	579.2	3950	14604	AC	[13]
–	581	–	15315	FEM	[18]
–	583	–	15465	DisC	[19]
–	579	–	14850	DTA	[20]
–	580	–	14938	DisC	[21]
549.2	580.2	728	14727	DC	[22]
–	–	–	15125	DSC	[28]
535	580	1590	14644	DC	[31]
549	580	3410	15004	DSC	[32]
530	578	3683	14815	DSC	[34]
549	579	941	15439	DSC	[36]
550	583	4420	15100	DSC	[37]
548.5	577.5	3825	14705	DSC	[39]
–	584	–	15810	AC	[40]
549	579	3620	14980	DSC	This work
KNO_3					
–	612	–	20018	MM	[5]
–	606	–	10753	DC	[6]
401	–	4905	–	VD	[7]
402.5	–	5413	–	AC	[9]
401	607	5096	9623	AC	[11]
400.5	–	4984	–	AC*	[16]
402	–	5415	–	AC	[17]
–	608	–	11193	FEM	[18]
–	610	–	10097	DisC	[19]
–	610	–	10000	DC	[25]
401	–	5402	–	DSC	[27]
402.1	607.7	5038	9805	AC	[29]
–	610	–	9205	DC	[31]
403	608	5075	10020	DSC	[36]
406	612	5640	10500	DSC	[37]
402.9	607.4	5111	9757	DSC	[39]
–	619	–	10504	AC	[40]
402.4	–	5065	–	DSC	[43]
404	606	5050	10015	DSC	This work
$CsNO_3$					
427	–	3482	–	VD	[7]
424.5	678.5	3736	14100	AC	[14]
–	679	–	13352	FEM	[18]
–	690	–	13440	DisC	[19]
426.5	678.2	3800	15500	DSC	[42]
427	679	3325	12100	DSC	This work

[a] MM, mixing method; VD, volume discontinuity method; DC, drop calorimetry; AC, adiabatic calorimetry; AC*, near-adiabatic conditions (for more detail, see the original publication); FEM, electrochemical measurement; DSC, differential scanning calorimetry; DTA; differential thermal analysis; DisC, dissolution calorimetry.

Table 3

Heat capacities of the solid and liquid phases of $NaNO_3$, KNO_3 and $CsNO_3$

α Solid	β Solid	Liquid	Method[a]	Ref.
$NaNO_3$				
98.9 (273–373 K)	–	–	MM	[4]
–	–	147 (593–703 K)	MM	[5]
138.1 (508–576 K)	–	152.9 (596–640 K)	DC	[6]
$28.49 + 0.218T$ (275–285 K)	–	–	AC	[8]
$13.41 + 0.251T$ (320–460 K)	140 (565–575 K)	–	AC	[9]
$26.75 + 0.216T$ (190–450 K)	137 (565–570 K)	140.5 (580–660 K)	AC	[10]
–	–	155.7	DC	[12]
$22.71 + 0.229T$ (330–450 K)	149 (570–575 K)	157.5 (620–625 K)	AC	[13]
–	–	149.4	AC	[15]
165.8 (483–548 K)	186.6 (554–573 K)	130.5 (597–747 K)	DC	[22]
–	136.2 (565–575 K)	–	AC	[23]
–	–	$141.8 - 0.0653(T - 578)$	DC	[26]
–	–	$157 + 0.0016(T - T_f)$	AC	[30]
168.2 (450–535 K)	202 (535–580 K)	212.1 (580–650 K)	DC	[31]
$39.3 + 0.187T$ (330–450 K)	143.9 (560–572 K)	142.7 (580–700 K)	DSC	[36]
$18.26 + 0.235T$ (350–450 K)	153 (560–565 K)	$150.5 - 0.0153T$ (590–790 K)	DSC	[37]
$26.54 + 0.215T$ (330–450 K)	140 (570–575 K)	139.1 (604–675 K)	AC	[38]
$268 - 1.0T + 0.0015T^2$ (334–493 K)	139 (560–565 K)	142.0 (586–653 K)	DSC	[39]
$22.62 + 0.222T$ (363–424 K)	139 (568–575 K)	138.2 (620–678 K)	DSC	This work
KNO_3				
100.9 (273–373 K)	–	–	MM	[4]
–	–	140.2 (623–708 K)	MM	[5]
–	123.4 (513–596 K)	140.7 (611–684 K)	DC	[6]

$36.67+0.20T$ (275–295 K)				
	–	–	AC	[8]
$30.01+0.208T$ (320–400 K)	$155.1-0.22T+0.00033T^2$ (450–570 K)	–	AC	[9]
$40.73+0.179T$ (327–400 K)	$236.2-0.518T+0.0006T^2$ (400–600 K)	139.3 (620–660 K)	AC	[11]
–	–	123.4	DC	[12]
–	–	149.4	AC	[15]
–	131.8 (513–593 K)	135.6 (633–663 K)	DC	[25]
–	–	$141.8-0.0653(T-611)$	DC	[26]
$44.69+0.167T$ (293–402 K)	$258.98-0.616T+0.0007T^2$ (402–608 K)	141 (608–641 K)	AC	[29]
–	–	$177+0.0029(T-T_f)$	AC	[30]
–	134.3 (500–610 K)	232.6 (610–675 K)	DC	[31]
–	–	141.0 (630–700 K)	DSC	[35]
$23.44+0.222T$ (333–390 K)	$77.15+0.108T$ (410–590 K)	142.5 (600–720 K)	DSC	[36]
$22.22+0.221T$ (330–400 K)	$250.3-0.572T+0.00065T^2$ (410–605 K)	$168.9-0.046T$ (616–790 K)	DSC	[37]
$51.3+0.149T$ (320–400 K)	$227.2-0.457T+0.00053T^2$ (430–600 K)	140.3 (610–726 K)	AC	[38]
$32.93+0.193T$ (334–400 K)	$246.6-0.574T+0.00067T^2$ (423–600 K)	140.4 (620–733 K)	DSC	[39]
$46.31+0.166T$ (300–400 K)	$148.74-0.055T$ (415–450 K)	–	DSC	[43]
$37.49+0.191T$ (362–404 K)	$255-0.569T+0.00063T^2$ (456–560 K)	142.7 (640–710 K)	DSC	This work
$CsNO_3$				
$27.55+0.231T$ (323–424 K)	$84.12+0.124T$ (425–678 K)	$40.13+0.188T$ (679–724 K)	AC	[4067]
–	–	$172+0.0062(T-T_f)$	AC	[5240]
$44.99+0.170T$ (300–347 K)	–	–	AC	[4210]
$36.95+0.206T$ (338–425 K)	146.8 (451–677 K)	142.3	AC	[4194]
113 (350–426 K)	140 (427–678 K)	197 (678–700 K)	DSC	[4971]
$45.88+0.174T$ (347–400 K)	$116.5+0.0515T$ (450–650 K)	146.0 (698–755 K)	DSC	This work

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[a] MM, mixing method; VD, volume discontinuity method; DC, drop calorimetry; AC, adiabatic calorimetry; AC*, near-adiabatic conditions (for more detail, see the original publication); FEM, electrochemical measurement; DSC, differential scanning calorimetry; DTA, differential thermal analysis; DisC, dissolution calorimetry.

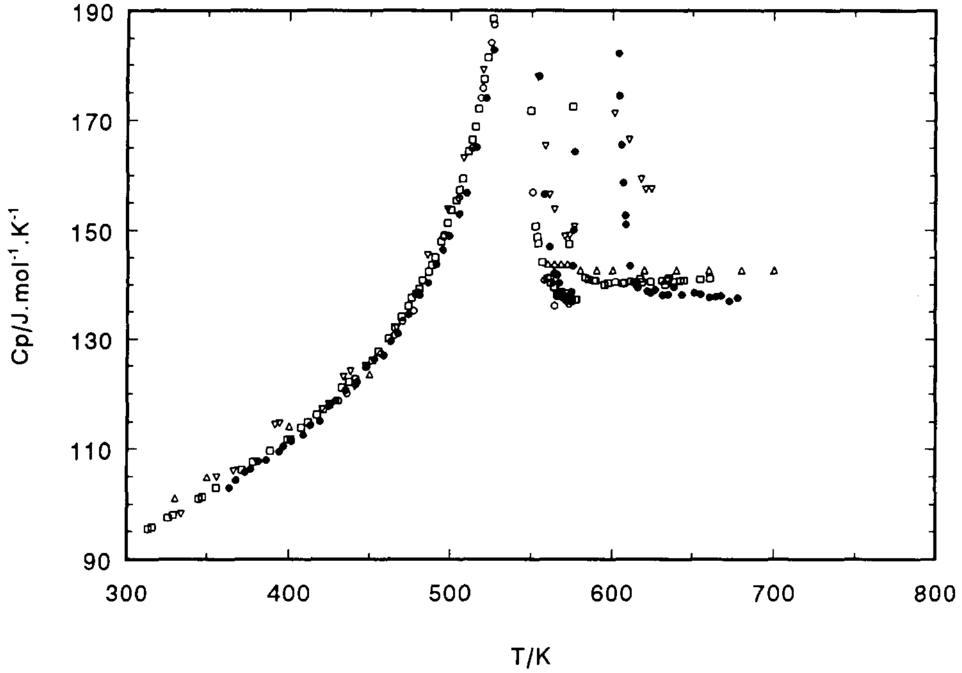

Fig. 2. Heat capacity of $NaNO_3$: ●, this work; □, Sokolov and Shmidt [10]; △, Rogers and Janz [36]; ○, Reinsborough and Wetmore [23]; ∇, Mustajoki [13].

3.2. Solid–liquid transitions (Table 2)

For the melting temperature of sodium nitrate, the mean value of the sixteen determinations is 580.5 K. All the values are inside a ± 4 K range and eight of them are included in the narrow interval 579–580.2 K. Our result is 579 K. For KNO_3, good agreement is obtained with all the various methods. The twelve determinations are included in the interval 606–619 K with a mean value of 609.8 K. Our result lies 3.8 K lower than this value at the lower limit of the discrepancy interval. The melting temperature of $CsNO_3$ has been given at 648 ± 6 K. Our result is 679 K.

Seventeen determinations of the melting enthalpy of $NaNO_3$ are available against nine determinations of the solid–solid transition enthalpy. Except for the very high value of Person [5], the values lie in the 14.6–15.8 kJ mol^{-1} range with a mean value of 15.1 kJ mol^{-1}. No systematic difference can be observed among the experimental methods. Our result is 14.98 kJ mol^{-1}. For KNO_3, the mean value is 10.1 kJ mol^{-1} and all the determinations lie between 9.2 and 11.2 kJ mol^{-1}, except for Person [5]. The discrepancy between the results obtained by drop calorimetry is twice that of the results obtained by differential scanning calorimetry. Our result is 10.02 kJ mol^{-1}. The melting enthalpy of $CsNO_3$ is given between 13.4 and 15.5 kJ mol^{-1}. Our result is much lower than all the other results, 12.1 kJ mol^{-1}.

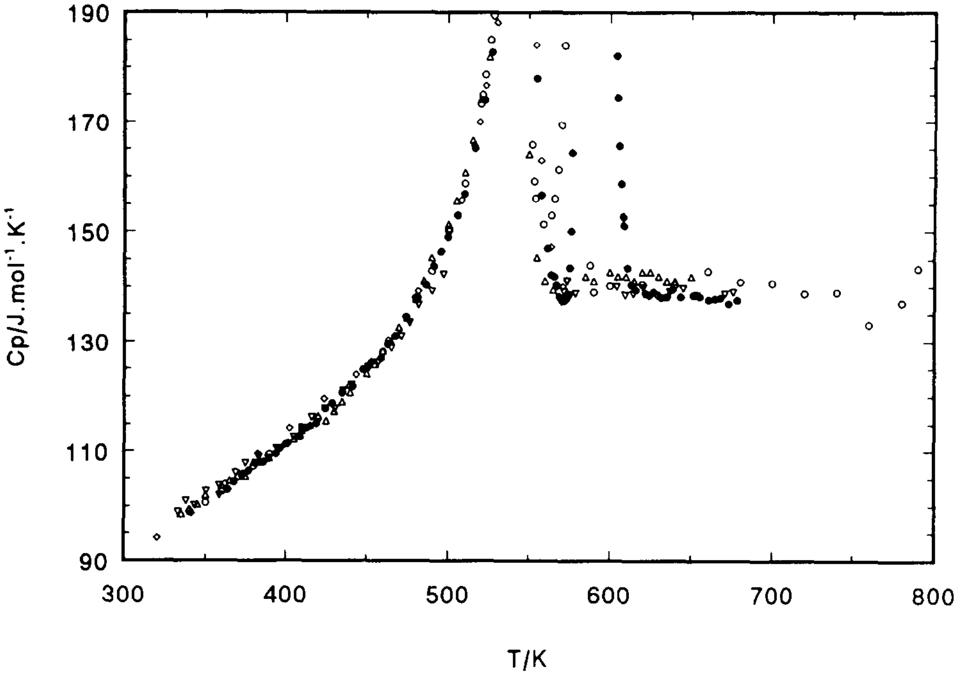

Fig. 3. Heat capacity of $NaNO_3$: ●, this work; ◇, Miekk-Oja [9]; Δ, Takahashi et al. [39]; ○, Carling [37]; ∇, Ichikawa and Matsumoto [38].

3.3. *Heat capacities (Table 3)*

$NaNO_3$ (Figs. 2 and 3)

In the α-phase, the constant values given in the literature are not realistic [6, 22]. They are generally too high. In the temperature range 300–450 K, the evolution of the heat capacity seems to be linear in continuity with the low-temperature sigmoid evolution (Southard and Nelson [8]). All the results lie in a ± 3% margin, except for Mustajoki [13] and Rogers and Janz [36] whose results are 5% higher than the mean value. Our results agree very well with the data obtained by adiabatic calorimetry of Ichikawa and Matsumoto [38] and those obtained by differential scanning calorimetry of Takahashi et al. [39]. All the results corresponding to the second-order transition agree well.

It is difficult to measure correctly the heat capacity of the β solid phase and its evolution with temperature cannot be precisely determined. Eight determinations lie in the 136–153 J K^{-1} mol^{-1} range (mean value 142.3 J K^{-1} mol^{-1}). The data obtained by drop calorimetry of Janz et al. [22] and Nguyen-Duy and Dancy [31] seem too high (200 J K^{-1} mol^{-1}). Our result is 139 J K^{-1} mol^{-1}.

In the liquid phase, no evolution with temperature can be accurately presented. A constant value in the 130–157 J K^{-1} mol^{-1} range seems to be realistic up to 650 K.

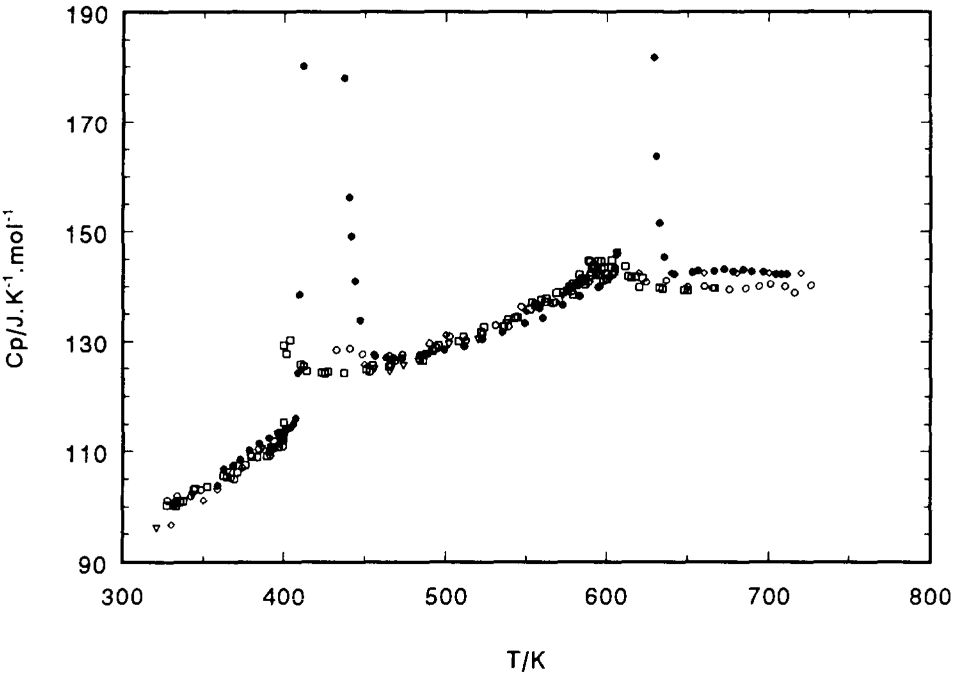

Fig. 4. Heat capacity of KNO_3: ●, this work; □, Sokolov and Shmidt [11]; ◇, Rogers and Janz [36]; ○, Ichikawa and Matsumoto [38]; ∇, Miekk-Oja [9].

Our result is 6% lower than the mean value calculated with the literature data. The result of Nguyen-Duy and Dancy [31] is undoubtedly too high.

The heat capacity measurements of Kubicar [24] (323–573 K) by a pulse method must also be mentioned but only a plot is available in the original paper and is not reported here.

KNO_3 (Figs. 4 and 5)

As for sodium nitrate, a linear evolution with temperature is observed in the α-phase. A good agreement is found for the slope among the various literature data. The results of Southard and Nelson [8] are 10% higher than all the other values. Our results are 3–5% higher than the mean literature values.

The β-phase is characterized by a non-linear evolution of the heat capacity with temperature. A linear evolution or a constant value for the heat capacity is probably erroneous. The data lie in a ± 5–10% discrepancy range. Our results agree well with the data of Sokolov and Shmidt [11] and Ichikawa and Matsumoto [38] in the low temperature domain and with the data of Carling [37] and Shmidt and Maksimov [29] in the high temperature domain.

For $NaNO_3$, the literature data suggest that the evolution of the heat capacity of the liquid phase with temperature has not been accurately determined. Most of the results

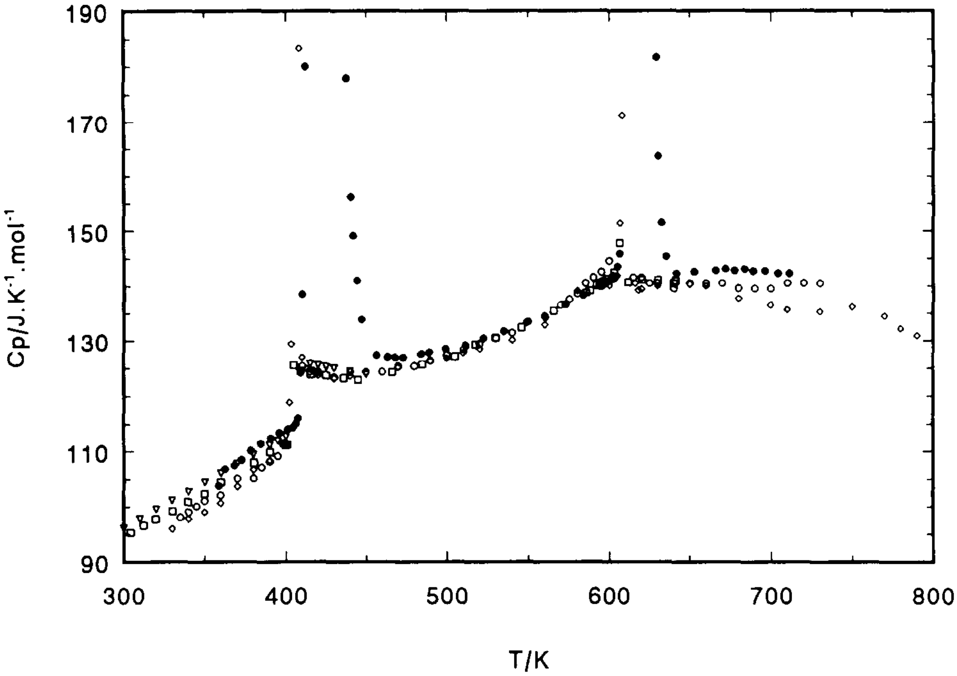

Fig. 5. Heat capacity of KNO_3: ●, this work; □, Sokolov and Shmidt [10]; ◇, Carling [37]; ○, Takahashi et al. [39]; ▽, Westphal et al. [43].

give a constant value of 141 ± 3 J K^{-1} mol^{-1} up to 700 K. The result of Douglas [12] is 14% lower than this value; those of Asahina et al. [30], Nguyen-Duy and Dancy [31] and Carling [37] lie 20% higher. Our results are very close to those of Rogers and Janz [36].

The heat capacity measurements of Taha et al. [41] using a pulse method must be mentioned, but only a plot is available in the original paper and is not reported here.

$CsNO_3$ (Fig. 6)

Over the whole temperature range, the results of Maeso and Largo [42] are 10% lower than all the other data.

All the determinations lie in a ± 5% discrepancy range for the α-phase. A good agreement is observed for the slope of the linear evolution with temperature.

The evolution with temperature of the heat capacity of the β-phase is characteristic. Except for Mustajoki [14], who determined an increasing heat capacity in the high temperature domain, all the authors have observed an increasing heat capacity in the 450–550 K range and a near constant value (148 J K^{-1} mol^{-1}) in the 550–675 K range. Our results seem to be too low in the low temperature domain and in good agreement with a mean value in the high temperature domain.

For $NaNO_3$ and KNO_3, only a constant value can be realistically presented. Some values higher than 170 J K^{-1} mol^{-1} have been published [14, 30, 42]. Our

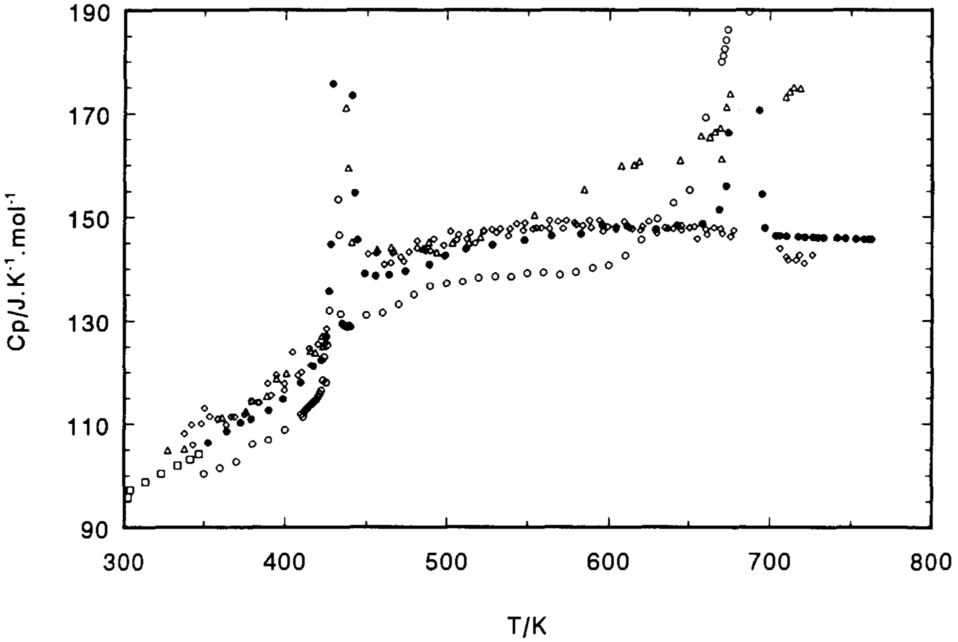

Fig. 6. Heat capacity of $CsNO_3$: ●, this work; □, Flotow et al. [33]; ◇, Ichikawa and Matsumoto [38]; ○, Maeso and Largo [42]; Δ, Mustajoki [14].

results, like the data of Ichikawa and Matsumoto [38] lie in the 140–145 J K^{-1} mol^{-1} range.

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