Journal of Thermal Analysis, Vol. 37 (1991) 155-169

THERMAL PROPERTIES OF ORGANIC LATENT COLD STORAGE MATERIALS

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(Received May 25, 1990)

The thermal properties of organic compounds intended to be applied as latent cold storage substances were investigated by methods of analysis according to Tammann, and by DTA and DSC. Findings were made concerning the melting and solidification behaviour, subcooling, specific melting enthalpies and specific heat capacities of paraffin mixtures, low and high-molecular glycols, ethylenediamine hydrates and formamide. Ethylenediamines and ethylene glycol, with high storage densities of 200-300 MJ $\cdot$m^{-3}, appear to be promising cold storage substances if subcooling is reduced. Through the use of congruently melting low-molecular glycols and formamide, storage densities of about 170 MJ $\cdot$m^{-3} are obtained. Paraffin mixtures and high-molecular glycols melt in wide ranges of temperatures and give values of about 100 MJ $\cdot$m^{-3}, which are not relevant to cold storage units.

At present, energy storing systems based on melting and solidification or lattice transformation processes, also called latent storage units, have been developed for a temperature range of about 0-1500° [1]. The application of latent cold storage units in the fields of room air conditioning, cooling and frost protection has been achieved for conditions below room temperature. Especially mixtures of salt and water, paraffins, glycols, amines and clathrates with high specific melting enthalpies, inexpensive ness and ready availability are considered as cold storing substances. Among these substances, attention has so far been paid only to the salt-water systems. The relevant data on the substances which are required for the development of storage systems are essentially known in the literature [2-5]. In contrast, there is only incomplete information on organic latent cold storage substances. There is some knowledge of the thermoanalytical behaviour of n-alkanes and paraffin fractions [4, 6-8], caprylic, acrylic and acetic acids [6, 8-10], polyethylene glycols [9, 11, 12] and some substance combinations such as ethylenediamine with dimethyl sulphoxide [13].

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The present work mainly involves investigations of organic, technically available latent cold storage substances, as concerns the melting and solidification behaviour under dynamic heating and cooling conditions and the determination of subcooling, specific melting enthalpy and specific heat capacity with an accuracy sufficient for the development of latent cold storage units.

Experimental

A survey of the chemicals used and their characterization is given in Table 1. The water contents of the samples were determined with an accuracy of 0.5% according to Karl Fischer. Dried ethylenediamine with a water content as given in Table 1 was prepared from ethylenediamine monohydrate by repeated distillation from sodium. After predrying, formamide was twice distilled with Wolfen-Zeosorb 3 A (VEB Jenapharm Laborchemie Apolda). Investigations of the thermal behaviour of the organic compounds were performed by cooling and heating methods according to Tammann [15], differential thermal analysis and differential scanning calorimetry [14]. Thermal analysis according to Tammann allowed determination of melting and solidification temperatures (±1 deg) and of subcooling (±2 deg) in larger samples of 30-40 g. Such results can be better transferred to the behaviour of storage materials in static latent storage units than those of DTA and DSC. At the same time, the method has the advantage of permitting visual observation of the crystallization processes. The method of differential thermal analysis with small samples of 30-80 mg and large samples of 8-11 g was the main method of investigation of the thermal behaviour and determination of the specific melting enthalpy (error ±5%). The melting and solidification temperatures were determined from the extrapolated onset temperatures. The DTA thermoanalyzers (Setaram, France and self-made equipment [14]) were calibrated with water as a calorimetric standard. For samples of 6-15 mg, specific heat capacities (error ±3%) were determined with a DSC-2C differential scanning calorimeter (Perkin-Elmer, USA) and the specific melting enthalpies of selected samples were specified (error ±1%).

The applied methods are referred to as follows: analysis according to Tammann = TA; differential thermal analysis in small samples = DTA 1; DTA in large samples = DTA 2; and differential scanning calorimetry = DSC. *n* = number of investigations. All error indications given in this work refer to a confidence limit of 0.95.

Table 1 Characterization of the examined compounds

Compound	Name of the product	Producer	Density liquid, kgm^{-3}	Content of impurities, max. %	Experimental water content, %
Ethylene glycol	Ethylenglycol R	VEB Komb. Chem. Werke Buna	1127_{0}	water 0.25 diethylene glycol 0.3	0.20
Diethylene glycol	Diethylenglycol	VEB Komb. Chem. Werke Buna	1132_{0}	water 0.50 ethylene glycol 1.0 triethylene glycol 1.0	0.15
Triethylene glycol	Triethylenglycol	VEB Laborchemie Apolda	1129_{15}	water 0.1	0.13
Tetraethylene glycol	Tetraethylenglycol 50	VEB Komb. Chem. Werke Buna	1128_{15}	ethylene glycol 0.1 diethylene glycol 0.2 triethylene glycol 23.5	0.03
Polyethylene glycol	Polyethylenglycol	VEB Komb. Chem. Werke Buna			
	A 300		1150_{15}	water 2.0	0.70
	A 400		1135_{15}	water 2.0	1.00
n-Alkane C_{10} to C_{13}	Parex Paraffin 1	VEB PCK Schwedt	745	aromatics 0.2 Vol.% C_9 0.8 C_{13} 11.3 C_{13} 5.5 $>C_{13}$ 0.2	–

Table 1 continued

Compound	Name of the product	Producer	Density liquid, kgm^{-3}	Content of impurities, max. %	Experimental water content, %
n-Alkanes C_{12} to C_{18}	Parex Paraffin II	VEB PCK Schwedt	F68	aromatics 1.4 Vol.% C_{12} 2.5 C_{13} 25.6 C_{17} 8.8 $>C_{17}$ 4.2	–
Chlorinated n-Alkanes C_{12} to C_{18}	Chlorparaffine	VEB Synthesewerk Schwarzheide		volatile 6	–
	WP 35		$1030\text{-}1115_{20}$	chlorine 35-40	
	WP 50		$1230\text{-}1330_{20}$	chlorine 49-54	
	WP 60		$1360\text{-}1400_{20}$	chlorine 59-62	
Ethylene diamine monohydrate	Ethylendiamine Monohydrat, techn.	VEB Laborchemie Apolda	970_{15}	2	23.30
Ethylene diamine dry			902_{15}		1.90
Formamide	Formamid	VEB Laborchemie Apolda	1149_{0}		0.09

Results

Glycols

Ethylene glycol HOC_2H_4OH

Figure 1 gives a representative DTA 1 curve of ethylene glycol. Ethylene glycol melts congruently at (–14±1)°, n =14. The melting process shows a good reproducibility. Crystallization in small amounts (DTA 1) begins at (-43±4) n =7; the subcooling is (29± 4) deg. In large samples, the subcooling is (18±2) deg, n = 7. The melt solidifies at (–32±2)° (DTA 2 and TA). In the used DSC equipment, no crystallization can be observed. The specific melting enthalpies were determined as (188±11) kJ/kg, n = 7, by DTA 1, and as (186±4) kJ/kg, n = 3, by DTA 2.

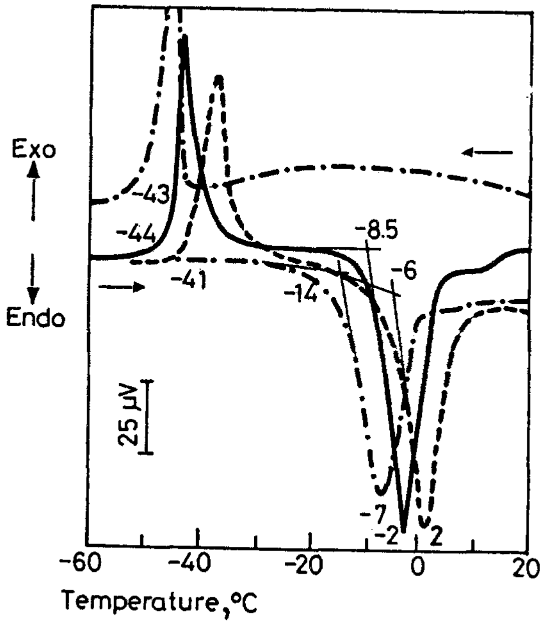

Fig. 1 Melting and solidification behaviour of low-molecular glycols; DTA 1; q =±2 deg·min^{-1}; – · – ethylene glycol, m =0.036 g; – diethylene glycol, m =0.041 g; – – – teraethylene glycol, m =0.049 g

The mean specific heat capacity of the melt was determined in the temperature range 12-42°. In two series of measurements, a value of (2.31±0.06) kJ/kg deg was found (DSC).

Diethylene glycol $HOC_2H_4OC_2H_4OH$

As shown in Fig. 1, the thermal behaviour of diethylene glycol is analogous to that of ethylene glycol. In contrast with ethylene glycol, however, crystallization is initiated in the heating phase. With TA and DTA 1, the melting point of diethylene glycol was found to be $(-8.5\pm1)^{\circ}$, $n = 12$. TA gave a mean solidification temperature of -26° and a mean subcooling of 18 deg. With DTA 1, the solidification temperature was determined as $(-44\pm4)^{\circ}$, $n = 7$; the subcooling was found to be (35.5 ± 4) deg. Diethylene glycol melts with a specific melting enthalpy of (148 ± 8) kJ/kg, $n = 9$ (DTA 1).

Triethylene glycol $HOC_2H_4OC_2H_4OC_2H_4OH$

Triethylene glycol displays a distinct tendency to subcooling. In the range from -50° to $+25^{\circ}$, no crystallization was observed.

Tetraethylene glycol $HOC_2H_4OC_2H_4OC_2H_4OC_2H_4OH$

As can be seen from Fig. 1, the thermal behaviour of tetraethylene glycol is similar to that of diethylene glycol. The melting temperature is $(-6\pm2)^{\circ}$, $n = 3$ (DTA 1). The specific melting enthalpy was found to be (152 ± 10) kJ/kg, $n = 3$ (DTA 1). The solidification temperature was -41° and there was a mean subcooling of 33 deg in small samples (DTA 1). Under the conditions of TA and DSC, no crystallization of the compound could be initiated.

Polyethylene glycols A 300 and A400 $(-CH_2-CH_2-O)_n$

The melting of polyethylene glycols A 300 and A 400 in a temperature range is shown by the results of TA. Polyethylene glycol A 300 melts in the range from -22° to -17°, and polyethylene glycol A 400 in the range between 4° and 8°. The mean solidification temperatures of -28° for A 300 and $+1^{\circ}$ for A 400 show a subcooling of 5-9 deg for A 300 and of 2-3 deg for A 400. The thermal behaviour of the two substances in the DTA 1 thermoanalyzer is shown in Fig. 2. Polyethylene glycol A 300 melts congruently on average at -40°, with a melting enthalpy of (70 ± 4) kJ/kg, $n = 3$. On average it solidifies at -32°. Polyethylene glycol A 400 melts with (104 ± 9) kJ/kg. Two melting points are indicated by the occurrence of two DTA peaks on heating. A small part of the compound melts at -17°, and the main part at -3°. Polyethylene glycol A 400 solidified uniformly in DTA 1 samples at a mean temperature of 5°.

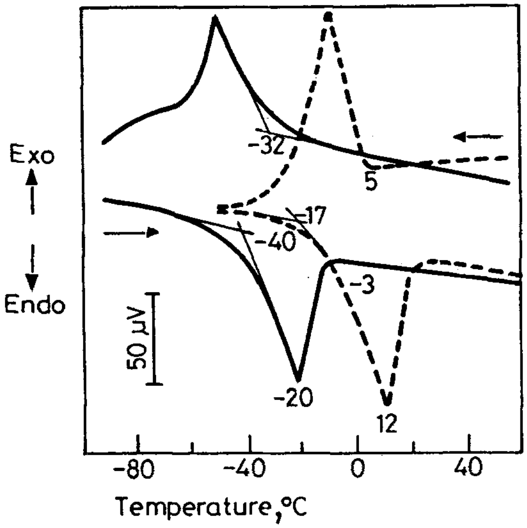

Fig. 2 Melting and solidification behaviour of polyethylene glycols; DTA 1; q = ±2 deg·min^{-1}; — A 300, m = 0.053 g; - - - A 400, m = 0.049 g

Paraffins

Chlorinated paraffins WP 35, 50 and 60

In spite of the noticeable solid-liquid phase change, no significant isothermal parts can be found in the TA curves. Setting points of –20° for WP 35, –6° to –8° for WP 50, and 0° for WP 60 (determined by visual observation) can be given from the TA measurements. The DTA 1 diagrams do not show any melting and solidification peaks.

Parex paraffins I and II

In contrast with Parex paraffin I, which melts in the interval –26° to –25°, Parex paraffin II melts in the wider range of –3° to +3° in the TA curve. On heating in the DTA 1 thermoanalyzer, thermal effects were observed at –32° for the main component of Parex paraffin I, with (139±12) kJ/kg deg, n = 3, and at –8° for the main component of Parex paraffin II, with (125±8) kJ/kg deg, n = 3 (Fig. 3). Small prepeaks occurred for both compounds in the DTA 1 diagrams. The solidification temperatures from TA and DTA 1 measurements are in good agreement at –24° for Parex paraffin I and +3° for Parex paraffin II.

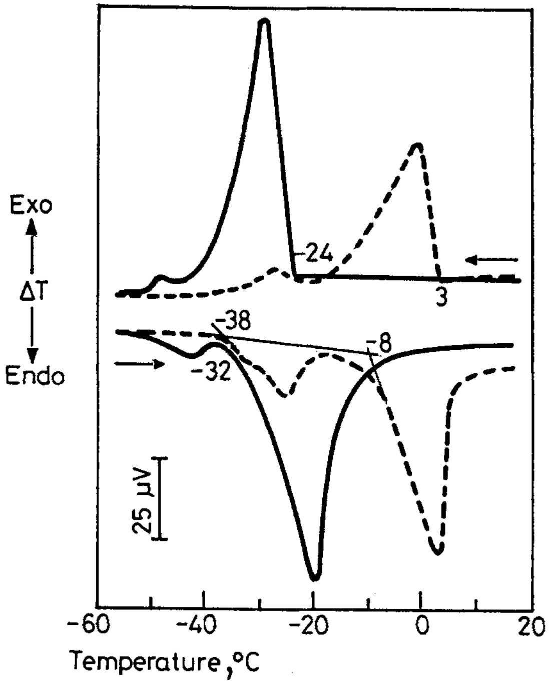

Fig. 3 Melting and solidification behaviour of paraffins; DTA 1; q = ±2 deg·min^{-1}; — Parex paraffin I, m = 0.034 g; – – – Parex paraffin II, m = 0.034 g

Ethylenediamine $H_2NC_2H_4NH_2 \cdot xH_2O$

The investigation results on ethylenediamines with a low water content and on the monohydrate, $H_2NC_2H_4NH_2 \cdot H_2O$ are summarized in Table 2. The thermal behaviour of $H_2NC_2H_4NH_2 \cdot xH_2O$ is illustrated in Fig. 4. Ethylenediamine monohydrate melts congruently at a reproducible temperature in the range from 9° (DTA) to 12° (DSC). The mean subcooling is 20 deg for small samples (DTA 1), but an average of 16 deg for large samples, and 10 deg for the largest samples. The specific melting enthalpy was determined as (280±4) kJ/kg, n = 7. It can be seen from the DSC curves in Fig. 4 that the occurrence of two melting peaks on heating is typical of the thermal behaviour of the ethylenediamines with a low water content. For the first peak, onset temperatures were found in the range –7° up to +1°, most temperatures being between –0.5° and +1°. An extrapolated melting temperature of (9±2)°, n = 8, was found for the second melting peak, except for samples 4 and 7. The uncertainty in the specific melting enthalpies of the

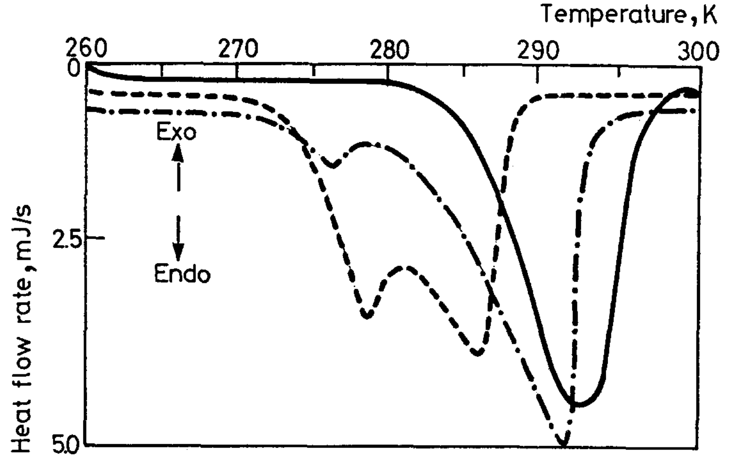

Fig. 4 Melting and solidification behaviour of ethylenediamine·x water; DSC; q = ±5 deg·min^{-1}; — x = 1.011 mol, *m* = 0.011 g; - - - x = 0.039 mol, *m* = 0.008 g; — · — x = 0.039, *m* = 0.009 g

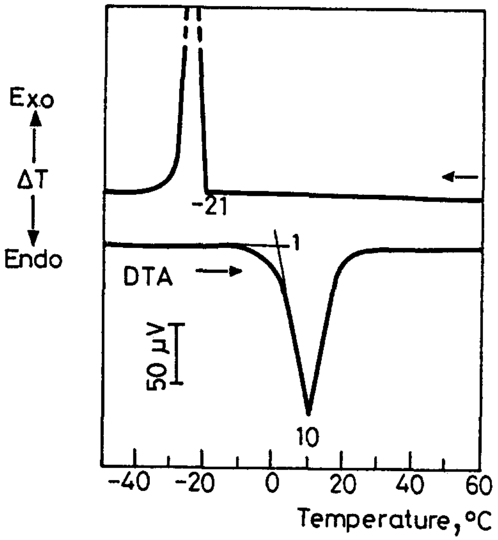

Fig. 5 Melting and solidification behaviour of formamide; DTA 1; q = ±2 deg·min^{-1}, *m* = 0.065 g

measured ethylenediamine samples is 17% and 9%, respectively, although the reproducibility for each sample is better (e.g. sample 8, 0.5%; Table 2). In the case of ethylenediamine with a water content of ≤2%, the mean specific heat capacity for the crystalline crop is (1.64±0.10) kJ/kg deg in the temperature range from –28° to –14° and that for the melt is (2.85±0.04) kJ/kg deg in the range between 27° and 45°.

Formamide $HCONH_2$

Formamide melts homogeneously at 1° (DTA 1) and 2° (TA), with a specific melting enthalpy of (152±0) kJ/kg, $n = 3$ (Fig. 5). The mean subcooling in large samples (TA) is 3-4 deg, and in small samples (DTA 1) is 22 deg.

Discussion

Table 3 summarizes all the experimental data on these substances, with known values from the literature. The thermal behaviour of most compounds in characterized by congruent reversible melting (Figs 1, 4 and 5). The melting points of ethylene glycol R, diethylene and tetraethylene glycol, ethylenediamine monohydrate and formamide are in good agreement with the literature data (Table 3). The polyethylene glycols, paraffin mixtures and ethylenediamines with low water contents exhibit melting ranges in approximate agreement with the literature data (Table 3). The differences in the melting behaviour of the polymers A 300 and 400 in DTA and TA samples are connected with the thermal history and heating regime. They have an important effect on the crystallization process, i.e. crystallite size and crystal defect. Shorter-chain alkanes and aromatics account for the existence of prepeaks in the DTA curves for mixtures of Parex paraffin I and II (Fig. 3) The melting of ethylenediamines with low water contents begins at the eutectic temperature of ethylenediamine monohydrate and ethylenediamine, in the range –0.5° to +1.0°. After the monohydrate has been completely consumed, the pure ethylenediamine deliquesces in the hydrate water according to its saturation curve (Fig. 4). In accordance with [4], paraffin mixtures do not display any subcooling, unlike all the other investigated compounds (Table 3). A high subcooling and no crystallization can be observed in the case of low-molecular glycols. This fact is in agreement with earlier experience acquired with these viscous compounds, with a tendency to glass formation [15]. An increase in the sample weight from the milligram to the gram range results in a decrease in subcooling as a consequence of the greater probability of nucleus formation.

Table 2 Investigation results of ethylenediamine with varied water content

Water content, mol	Sample No. (number of measurements)	Invest. method	Heating θ_{on}, °C	θ_{on}, °C	Cooling θ_{o} °C	Spec. melt. enthalpy $\Delta_F H$, $kJ \cdot kg^{-1}$
0.039	1 (2)	DSC	0.5	7.5		398
	2 (1)		–5.5	10		357
	3 (1)		–4.0	9.5		358
	4 (1)		–0.5	–0.3		353
0.046	5 (1)	DSC	–7.0	10.0		362
	6 (1)		–4.0	9.0		354
	7 (1)		1.0	1.5		342
0.064	8 (5)	DSC	1.0	10.5		353±2
	9 (2)	DTA 1	0	8	–11.5	
	10 (2)	DTA 1	0	8	–13.5	
1.01	11 (2)	DSC	12.0			
	12 (2)	DTA 1	9.0		–11.5	280
	13 (2)	TA	10.0		– 0.5	
1.11	14 (2)	DTA 2	9.0		– 6.5	
1.11	15 (2)	DTA 2	9.5		– 5.5	280±4

The DSC sample preparation conditions did not permit determination of the specific heat capacities in the solid and liquid states of the latent cold storage materials of most samples. The mean specific heat capacity of the melt of ethylene glycol R between 12° and 42° (Table 3) fits well into the spread of the data given in the literature: 2.391_{15} $kJ \cdot kg^{-1} deg^{-1}$ [27], 2.407_{20} $kJ \cdot kg^{-1} deg^{-1}$ [27], 2.3497_{20} $kJ \cdot kg^{-1} deg^{-1}$ [17], 2.428_{20} $kJ \cdot kg^{-1} deg^{-1}$ [26] and $2.18_{20\text{-}27}$ $kJ \cdot kg^{-1} deg^{-1}$ [17]. The mean specific heat capacities of the melt and the crystalline crop of ethylenediamines with a water content of $\leq 2\%$ are approximate values.

Except for the ethylenediamines with a low water content, the specific melting enthalpies agree well with the literature data (Table 3). The great variations in the values for ethylenediamines with a low water content result from the change in the water content during sample preparation, irrespective of the inert gas atmosphere (Fig. 4). Reference [23] gives no data on the water content of the sample. For this reason, a comparison of the data does not appear advisable.

Table 3 Summary of the caloric data of the examined compounds

Compound	Melting temperature θ_F, °C		Density ρ_L, kg·m^{-3}	Specific melting enthalpy $\Delta_F H$, kJ·kg^{-1}		Storage density $\Delta_F H_y$, MJ·m^{-3}	Specific thermal capacity	Subcooling of great samples
	exp.	Ref.	(liquid)	exp.	Ref.	(theoretical)	C_P, kJ·kg^{-1}K^{-1}	U/K
Ethylene glycol	–14±1	–13 [16, 17]	1127_0	188±11	187.7 [16] 187.3 [18] 187.4 [19]	210±12	2.31±0.06 (12°C–42°C)	18±2
Diethylene glycol	– 8.5±1	– 8 [18] –10.1 [20] –10.5 [21]	1132_0	148±8	–	167±9	–	18
Tetraethylene glycol	– 6±2	– 5.5 [18] – 9.4 [22]	1128_{15}	152±10	–	171±11	–	
Polyethylene glycol								
A 300	–22 to –17	–	1150_{15}	70±4	–	80±5	–	5 to9
A 400	+4 to +8	8 [9]	1135_{15}	104±9	99.6 [9]	117±10	–	2 to 3
Parex paraffin I	–26 to –25	–	745	139±12	–	103±9	–	–
Parex paraffin II	– 3 to +3	4 to 10 [4] 2 to 7 [4]	768	125±8	147 [4] 152 [4]	96±6	–	–

Table 3 continued

Compound	Melting temperature θ_F, °C		Density ρ_l, $kg \cdot m^{-3}$	Specific melting enthalpy $\Delta_F H$, $kJ \cdot kg^{-1}$		Storage density $\Delta_F H_y$, $MJ \cdot m^{-3}$	Specific thermal capacity	Subcooling of great samples
	exp.	Ref.	(liquid)	exp.	Ref.	(theoretical)	C_P, $kJ \cdot kg^{-1} K^{-1}$	U/K
Ethylene diamine <2% water	–0.5 to +9	–0.8 to +8 [24]	902_{15}	360±40	321.1 [23]	325±36	1.64±0.10 (–28 to –14°C) 2.85±0.04 (27 to 45°C)	12
Ethylene diamine monohydrate	10±2	10 [24]	970_{15}	280± 4	–	272±4	–	10
Foramide	1 to 2	2.1 [25]	1149_{0}	152± 0	148.7 [26]	176±0	–	3 to 4

From the aspect of the specific melting enthalpies (Table 3), dried ethylenediamine, ethylenediamine monohydrate and ethylene glycol R have the best prerequisites for achievement of a high storage density in the range 200-300 MJ m^{-3}. However, dried ethylenediamine must be excluded as a potential cold storage material, because its drying is expensive. Other substances in Table 3 have storage densities of about 170 $MJ\cdot m^{-3}$. These substances are also promising storage materials. The paraffin mixtures frequently discussed in the literature, and polyethylene glycols as well, have low storage densities of about 100 $MJ\cdot m^{-3}$. Latent cold storage materials such as ethylenediamine monohydrate and ethylene glycol R, which have the highest storage densities, exhibit considerable subcooling. Stable storage behaviour can be ensured by the known methods of crystal stockpiling [28].

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Zusammenfassung — Mittels DTA, DSC und dem Analysenverfahren von Tammann wurden die thermischen Eigenschaften von organischen Substanzen untersucht, die als Kältespeichersubstanzen verwendet werden sollen. Dabei wurden Aussagen bezüglich des Schmelz- und Erstarrungsverhaltens, der Unterkühlung, der spezifischen Schmelzenthalpien und spezifischen Wärmekapazitäten von Paraffingemischen, nieder- und hochmolekularen Glykolen, Ethylendiaminhydraten und von Formamid getroffen. Ethylendiamine und Ethylenglykole mit einer hohen Speicherkapazität von 200-300 MJ$\cdot$m^{-3} scheinen gute Kältespeicherstoffe zu sein, wenn die Unterkühlung unterbunden wird. Bei der Verwendung von kongruent schmelzenden niedermolekularen Glykolen und von Formamid wurden Speicherkapazitäten von 170 MJ$\cdot$m^{-3} erreicht. Paraffingemische und hochmolekulare Glykole schmelzen in einem weiten Temperaturbereich und sind mit einer Speicherkapazität von 100 MJ$\cdot$m^{-3} für Kühlbatterien ungeeignet.