

Heat capacities of alkanols III. Some 1-alkanols from C₁₀ to C₂₀

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Abstract

Saturation molar heat capacities C_{sat} in the liquid phase of five 1-alkanols from C₁₀ to C₂₀ were measured by a commercial SETARAM heat conduction calorimeter. Results obtained cover the following temperature range: 1-decanol 304–405 K, 1-dodecanol 307–358 K, 1-hexadecanol 328–387 K, 1-octadecanol 338–397 K, and 1-eicosanol 347–517 K. The heat capacity data obtained in this work were merged with available experimental data from literature, critically assessed and sets of recommended data were developed by correlating selected data as a function of temperature.

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1. Introduction

Heat capacities belong among the basic thermophysical and thermodynamic properties that characterize a liquid. They are used in chemical engineering for establishing energy balances, in thermodynamics for obtaining entropy and enthalpy values, and in thermochemistry for calculating changes in reaction enthalpies with temperature. Even though experimental data are available for a fairly large group of compounds [1,2] more data are needed for compounds with no data available at all, for extending the temperature range the available data cover or for verifying the presently available literature data.

This work, which is the third paper in a series devoted to heat capacities of alkanols (for the first and second papers in the series see references [3] and [4], respectively), was concerned with measurement of heat capacities in the liquid phase for five 1-alkanols from C₁₀ to C₂₀. New experimental data were obtained for all even carbon number 1-alkanols but 1-tetradecanol where ample data were measured by Steele et al. [5]. Our objective was to provide more data in particular in the region where the dependence of heat capacity

of 1-alkanols on temperature is untypical [6] exhibiting an inflexion point as illustrated in Fig. 1. The upper temperature limit of the experimental data determined in this work depends upon the volatility of the compound, as its vapor pressure in the cell of the C80 calorimeter should be below about 10 kPa. Thus, the data for 1-eicosanol, the least volatile compound of this study, extend to highest temperature.

2. Experimental

2.1. Materials

All samples were purchased from Aldrich. Its purity was determined by GC (Hewlett-Packard 6890 equipped with column HP 5, cross-linked 5% PH ME Siloxan, length 30 m, film thickness 0.25 μm, ID 0.32 mm) with FID detector.

1-Decanol and 1-dodecanol were purified by fractional distillation in a packed column and dried over molecular sieves type 4A. Water content was lower than 0.004 wt.% (detection limit of the Schot Geräte apparatus for Fisher analysis). 1-Hexadecanol, 1-octadecanol and 1-eicosanol were purified by four times repeated zone refining at ambient temperature.

2.2. Apparatus and procedure

A commercial C80 calorimeter made by SETARAM (Caluire, France) was used for measurement of saturation

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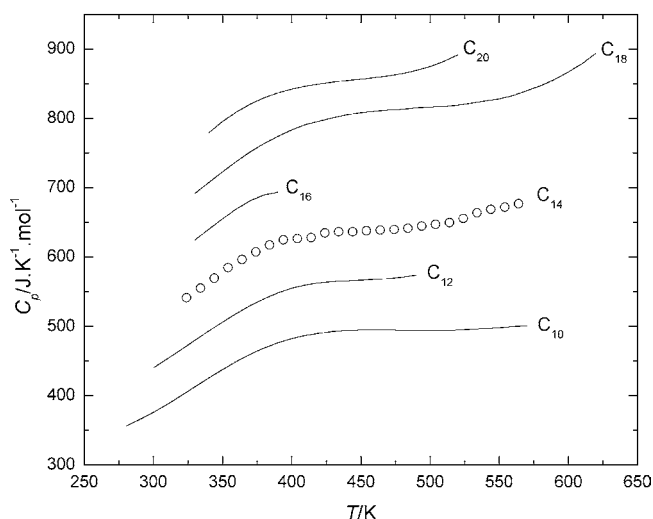


Fig. 1. Isobaric heat capacities of 1-alkanols. C_{10} , C_{12} , C_{16} to C_{20} smoothed data from this work, C_{14} experimental data from reference [5].

heat capacities. In this work the calorimeter was equipped with standard cells having the inner volume of 9.5 cm^3 reduced by a raised bottom. Heat capacity was measured by the well known three-step procedure [7]. The measurement vessel was empty in first run and it was filled with the measured sample and with the calibration compound in the second and the third runs, respectively. Reference vessel was empty during all three runs. In a typical heat capacity experiment with samples of several grams, the temperature was increased in steps of 5–10 K with a heating rate of 0.2 K/min. The average heat capacity for a given temperature step ΔT is obtained from the time integrals of the differential thermopile signal for the filled and empty sample cells and is related to the temperature $T = T_{\text{in}} + \Delta T/2$ (T_{in} is the initial temperature).

The calorimeter was calibrated by measuring heat capacity of synthetic sapphire ($\alpha\text{-Al}_2\text{O}_3$), the NIST Standard Reference Material 720 [8]. Temperature calibration was provided by the calorimeter manufacturer. Temperature reading was checked by measuring melting temperature of benzoic acid (Lachema, purity 99.97%), which gave value of 395.49 K, in agreement with literature value [9,10]. In our previous experiments we estimated the uncertainty of heat capacity data measured by C80 calorimeter to be $\pm 1\%$ by making test experiments with several compounds of well-known heat capacity values. In the case of 1-eicosanol, uncertainty is slightly higher ($\pm 2\%$) due to lower reproducibility above 390 K.

The temperature dependence of heat capacity for all studied substances was supposed to be close to linear over the measuring temperature step. Thus, the average heat capacity over the individual temperature step could be considered as true heat capacity relating to the mean temperature of the interval.

For the compounds where experimental values of reasonable accuracy are available above the normal boiling

temperature (1-decanol and 1-dodecanol), distinct recommended data sets are presented for C_p and C_{sat} . Recommended C_{sat} data relate by definition to the saturation line. Recommended C_p data relate above the normal boiling temperature T_b to the saturation pressure. Below T_b , they represent both the heat capacity at standard pressure 101.325 kPa and the saturation pressure as their difference is smaller than the accuracy of the best literature data. When necessary, conversion between C_p and C_{sat} was performed as described in [2].

3. Results

The direct experimental $T - C_{\text{sat}}$ values are presented in Table 1.

The data determined in this work were combined with all other calorimetrically determined heat capacities available in literature. All available sources were critically assessed. The important part of the selection process was the simultaneous correlation of all experimental data, which served to test the consistency, and helped to reveal systematic errors. The following criteria were observed in the selection process: (a) accuracy of the experimental technique claimed by the author, (b) laboratory performance history, (c) consistency of the data with values from other laboratories (if available), (d) substance purity, (e) calorimeter type, (f) time of data origin, (g) scatter of the data. It is not possible to apply the above selection criteria in a rigorous manner following strictly the outlined policy. In some cases, the error of measurement claimed by the author is too optimistic and it may even be unrealistic. In addition, evaluating the reputation of various laboratories is a relatively subjective process.

The selected data were fitted with the polynomial equation using the weighted least-squares method:

$$\frac{C}{R} = \sum_{i=0}^n A_{i+1} \left(\frac{T}{100} \right)^i \quad (R = 8.314472 \text{ J K}^{-1} \text{ mol}^{-1}) \quad (1)$$

Considering the width of the temperature range of experimental values for 1-decanol, 1-dodecanol and 1-octadecanol, the data were correlated by cubic splines in two to three subintervals. In this case the correlation is constrained to yield the identical C , dC/dT and d^2C/dT^2 values at the knot (temperature splitting the overall temperature range of experimental values into subintervals). Results of the correlation by cubic splines are presented in terms of parameters of Eq. (1) with $n = 3$. More detailed information regarding temperature correlation of C with cubic splines can be found in the monograph by Záborský et al. [11].

For 1-decanol, 1-dodecanol and 1-octadecanol, where the selected experimental data cover a wide temperature range and where a reliable estimate of critical temperature could be obtained the isobaric heat capacity data were fitted to

Table 1
Experimental liquid heat capacities

	<i>T</i> (K)	<i>C</i> _{sat} / <i>R</i>	Δ <i>C</i> _{sat} / <i>R</i>		<i>T</i> (K)	<i>C</i> _{sat} / <i>R</i>	Δ <i>C</i> _{sat} / <i>R</i>		<i>T</i> (K)	<i>C</i> _{sat} / <i>R</i>	Δ <i>C</i> _{sat} / <i>R</i>
1-Decanol	304.4	45.5	−0.3	1-Hexadecanol	327.7	74.7	0.1	1-Eicosanol	347.4	94.5	−0.7
	304.4	45.5	−0.3		327.7	74.7	0.1		347.4	94.4	−0.9
	304.4	45.5	−0.3		327.7	74.6	−0.1		347.6	94.1	−1.1
	307.4	45.8	−0.4		327.7	74.7	0.0		357.4	96.3	−0.7
	307.4	45.8	−0.5		327.7	74.6	−0.1		357.4	96.1	−0.8
	307.4	45.8	−0.4		337.6	76.6	0.1		357.5	95.9	−1.1
	310.9	46.4	−0.4		337.6	76.4	−0.1		367.3	97.9	−0.5
	310.9	46.4	−0.4		337.6	76.5	0.0		367.3	97.7	−0.6
	310.9	46.4	−0.4		337.6	76.6	0.1		367.4	97.3	−1.0
	315.3	47.1	−0.3		337.6	76.4	−0.1		367.6	98.0	−0.4
	315.4	47.1	−0.3		347.5	78.5	0.2		377.2	99.5	0.0
	315.4	47.0	−0.4		347.5	78.4	0.1		377.3	99.0	−0.5
	322.8	48.2	−0.4		347.5	78.3	0.0		377.3	98.6	−0.9
	322.8	48.2	−0.3		347.5	78.3	−0.1		377.6	99.2	−0.3
	322.8	48.2	−0.3		347.5	78.2	−0.1		387.1	100.0	−0.4
	332.7	49.7	−0.4		357.4	80.0	0.0		387.2	100.1	−0.3
	332.8	49.8	−0.3		357.4	80.0	−0.1		387.2	99.9	−0.5
	332.8	49.7	−0.4		357.4	80.1	0.1		387.5	100.2	−0.2
	342.7	51.3	−0.3		357.5	80.0	−0.1		397.1	100.8	−0.4
	342.7	51.3	−0.3		357.5	79.9	−0.2		397.1	100.9	−0.3
	345.2	51.6	−0.3		367.4	81.7	0.1		397.5	100.1	−1.0
	345.2	51.8	−0.2		367.4	81.6	0.0		407.0	101.5	−0.2
	350.1	52.3	−0.4		367.4	81.6	0.1		407.1	100.6	−1.1
	350.1	52.4	−0.3		367.4	81.6	0.0		407.4	100.5	−1.1
	350.1	52.4	−0.3		367.4	81.6	0.0		417.0	102.2	0.1
	350.1	52.3	−0.3		377.3	82.5	−0.2		417.0	100.9	−1.2
	357.6	53.4	−0.3		377.3	82.6	−0.1		417.3	101.2	−0.9
	357.6	53.5	−0.2		377.3	82.7	0.0		426.9	102.5	0.1
	367.5	54.8	−0.2		377.3	82.1	−0.7		427.0	101.5	−0.9
	367.5	54.8	−0.2		377.3	83.4	0.8		427.2	102.0	−0.4
	377.4	55.9	−0.2		387.2	83.7	0.5		436.8	102.7	0.0
	377.4	55.9	−0.2		387.2	83.6	0.3		436.9	102.1	−0.6
	387.3	56.9	−0.2		387.2	83.5	0.2		437.2	102.3	−0.4
387.3	56.8	−0.3	387.2	83.8	0.6	446.8	102.8	−0.1			
397.2	57.6	−0.3	387.2	82.1	−1.5	446.9	102.7	−0.3			
397.2	57.4	−0.4	1-Octadecanol	337.6	85.1	0.6	447.1	102.8	−0.1		
404.7	58.1	−0.2		337.6	85.3	0.8	456.7	103.2	0.0		
404.7	57.9	−0.4		337.7	85.1	0.6	456.8	102.9	−0.3		
1-Dodecanol	307.2	54.1	0.1	337.7	85.1	0.6	457.0	103.1	−0.1		
	307.4	54.0	−0.1	347.5	87.0	0.6	466.7	103.5	0.0		
	307.5	53.9	−0.4	347.5	87.1	0.7	466.8	103.2	−0.3		
	310.7	54.7	0.1	347.6	87.0	0.5	467.0	103.4	0.0		
	310.9	54.6	−0.1	347.6	87.0	0.6	476.6	104.0	0.2		
	310.9	54.6	−0.1	357.4	88.6	0.4	476.7	103.8	0.0		
	315.2	55.4	0.1	357.5	88.7	0.5	476.9	104.1	0.2		
	315.4	55.3	−0.2	357.6	88.9	0.7	486.6	104.5	0.2		
	315.4	55.2	−0.4	357.6	88.9	0.7	486.6	104.5	0.1		
	320.1	56.2	−0.1	367.3	90.4	0.5	486.8	104.5	0.1		
	320.4	56.1	−0.2	367.4	90.4	0.5	496.6	105.3	0.3		
	320.4	56.1	−0.2	367.5	90.4	0.5	496.8	105.1	0.1		
	327.6	57.4	0.0	367.5	90.3	0.4	506.7	105.9	0.1		
	327.9	57.2	−0.3	377.2	91.7	0.3	516.6	106.8	−0.1		
	327.9	57.4	0.0	377.3	91.8	0.3					
	337.5	59.0	0.1	377.4	91.8	0.3					
	337.8	59.1	0.1	377.4	91.8	0.4					
	337.8	58.9	−0.2	387.2	92.6	−0.2					
	347.5	60.6	0.1	387.2	92.8	0.0					
	347.7	60.6	0.1	387.3	92.9	0.1					
	347.7	60.5	0.0	387.3	92.9	0.1					
	357.4	62.1	0.2	397.1	93.6	−0.3					
	357.7	62.1	0.1	397.1	93.6	−0.3					
	357.7	62.1	0.1	397.2	93.7	−0.3					
				397.2	93.7	−0.2					

$R = 8.314472 \text{ JK}^{-1} \text{ mol}^{-1}$, $\Delta C_{\text{sat}}/R = (C_{\text{sat}} - C_{\text{sat}}^{\text{calc}})/R$ where $C_{\text{sat}}^{\text{calc}}$ was calculated from Eq. (1) using parameters from Table 3.

Table 2
Survey of all available calorimetrically measured heat capacity data for 1-alkanols

Compound	Selected data	Temperature range (K)	Error (%)	Purity (%)	Rejected data	Temperature range (K)
1-Decanol	[21]	303	0.5 ^a	n.s. ^b	[20]	308–338
	[12]	306–463	0.9	n.s.	[16]	323–503
	[13]	301–461	1.5 ^a	99.80	[15]	304–523
	[22]	283–313	0.5 ^a	99	[24]	298–313
	[23]	298	0.5 ^a	98		
	[25]	298	0.5 ^a	99.0		
	[3]	326–571	2.0	99.87		
	[14]	281–388	0.2	99.70		
1-Dodecanol	This work	304–405	1.0	99.70		
	[13]	316–486	1.5 ^a	99.98	[17]	303–533
	[23]	298	0.5 ^a	98		
1-Hexadecanol	This work	307–358	1.0	99.00		
	This work	328–387	1.0	99.60	[18]	323–346
1-Octadecanol	[16]	353–623	2.0	99.8		
	[19]	333–359	0.2	99.8		
1-Eicosanol	This work	338–397	1.0	99.60		
	[19]	339–371	0.2	98.0		
	This work	347–517	2.0	98.5		

^a Assigned by evaluator.

^b Not specified.

another empirical, the so-called quasi-polynomial, equation that allows a meaningful extrapolation to the critical point

$$\frac{C_p}{R} = A_1 \ln(1 - T_r) + \frac{A_2}{1 - T_r} + A_3 + A_4 T_r \quad (2)$$

where $T_r = T/T_c$, and T_c is the critical temperature. The derivation of Eq. (2) (see [1,2,11]) is based on the assumption that the heat capacity is always an increasing function of temperature and that it is unbounded at the critical point. An objective function for the least-squares minimization was used of the form:

$$S = \sum_{i=1}^n \left(\frac{\Delta C_i}{R} \right)^2 (\sigma_{C/R,i})^{-2} \quad (3)$$

where the variance $\sigma_{C/R,i}$ was estimated for each value on the basis of the assumed experimental error of the set of data used in the correlation. The input information was the percentage error of the experimental data $\sigma_r C$ given by the author or estimated by the evaluator for the whole data set. The variance of the i th data point was expressed as

$$\sigma_{C/R,i} = 10^{-2} \frac{C_i \sigma_r C}{R} \quad (4)$$

A survey of all selected data used to determine parameters of Eqs. (1) and (2) as well as a list of rejected data is given in Table 2.

Parameters of Eq. (1) derived from the fit are given in Table 3 along with the relative standard deviation of the

Table 3
Parameters of Eq. (1) for liquid heat capacity

Compound	Type of heat capacity	Parameters				Temperature range		Uncertainty	
		A_1	A_2	A_3	A_4	T_{\min} (K)	T_{\max} (K)	s_r^a (%)	%
1-Decanol	C_p	246.733	210.063	68.3785	6.91542	281.5	380.0	0.5	0.5
	C_p	324.266	240.726	50.2501	3.49060	380.0	500.0		1
	C_p	180.569	62.1746	10.3301	0.548078	500.0	570.7		5
	C_{sat}	246.560	209.903	68.3296	6.91045	281.5	380.0	0.5	0.5
	C_{sat}	322.345	239.233	49.8639	3.45740	380.0	500.0		1
	C_{sat}	196.608	72.1390	12.4104	0.694226	500.0	570.7		5
1-Dodecanol	C_p	185.891	−152.201	52.0271	−5.35551	298.1	400.0	0.2	1
	C_p	−673.239	492.147	−109.060	8.06840	400.0	486.0		3
	C_{sat}	185.618	−151.964	51.9589	−5.34900	298.1	400.0	0.2	1
	C_{sat}	−671.366	490.774	−108.726	8.04138	400.0	486.0		3
1-Hexadecanol	C_p	890.027	−755.550	227.949	−22.3720	327.6	387.2	0.4	1
1-Octadecanol	C_p	301.548	−235.565	76.8013	−7.71879	332.6	380.0	0.5	0.5
	C_p	−309.133	246.551	−50.0713	3.41038	380.0	622.9		3
1-Eicosanol	C_p	−423.184	350.373	−78.3 128	5.87483	339.0	516.6	0.5	1

^a $s_r = 10^2 \left(\sum_{i=1}^n \left[\frac{(C - C^{\text{calc}})/C}{(n - m)} \right]^2 \right)^{1/2}$ where C is the molar heat capacity, C_p or C_{sat} , n is the number of fitted data points, and m the number of independent adjustable parameters.

Table 4
Parameters of Eq. (2) for liquid isobaric heat capacity

Compound	Parameters						Temperature range		Uncertainty	
	A_1	A_2	A_3	A_4	A_5	A_6	T_{\min} (K)	T_c (K)	s_r^a (%)	%
1-Decanol	-8.75010	2.31171	-120.908	668.370	-840.387	-840.387	281.5	689	1.4	5
1-Dodecanol	564.092	94.5197	-156.372	841.622	0	0	298.1	720	0.4	3
1-Octadecanol	941.086	94.3427	-239.952	2074.51	-1868.36	1814.06	332.6	790	1.6	3

^a $s_r = 10^2 \left(\sum_{i=1}^n [(C_p - C_p^{\text{calc}})/C_p] / (n - m) \right)^{1/2}$, where n is the number of fitted data points, and m the number of independent adjustable parameters.

fit. Parameters of Eq. (2) are given in Table 4. It should be emphasized that extrapolation above the upper temperature limit of the experimental data by the quasi-polynomial equation is correct only in a qualitative manner. No guarantee can be given that the extrapolation will describe quantitatively the real heat capacity in this region except for the value at the critical point. The flexibility of the quasi-polynomial equation is in general worse than that of the polynomial Eq. (1), even though for the sets of data fitted in this work there is either no or a small difference between the two equations as indicated by the relative standard deviation of the fit s_r that is given in Tables 3 and 4.

4. Discussion

Table 2 presents a survey of literature sources of calorimetrically measured heat capacity data of studied 1-alkanols together with temperature range, measurement error and purity of the sample. The rejected sets of data, that is those not used in the calculation of parameters of Eqs. (1) and (2), are represented by mostly old and inaccurate measurements carried out with compounds of low purity. The rejected data show large deviations from the recommended data as demonstrated in deviation plots (Figs. 2–6).

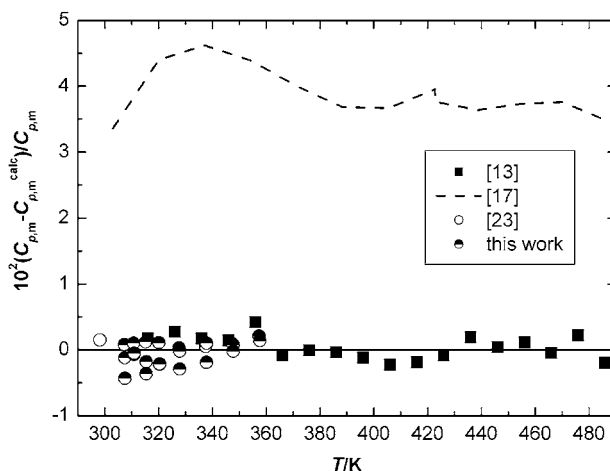


Fig. 3. Deviation plot for heat capacity of 1-dodecanol.

Most abundant data are available for 1-decanol. Several sets of reliable data extend up to high temperatures [3,12–14]; we thus rejected two sets of data by Russian authors covering a wide range [15,16] as they deviate from the recommended data by more than the stated measurement error. For 1-dodecanol we rejected another set of Russian data by Vasil'ev et al. [17], again due to their deviations higher

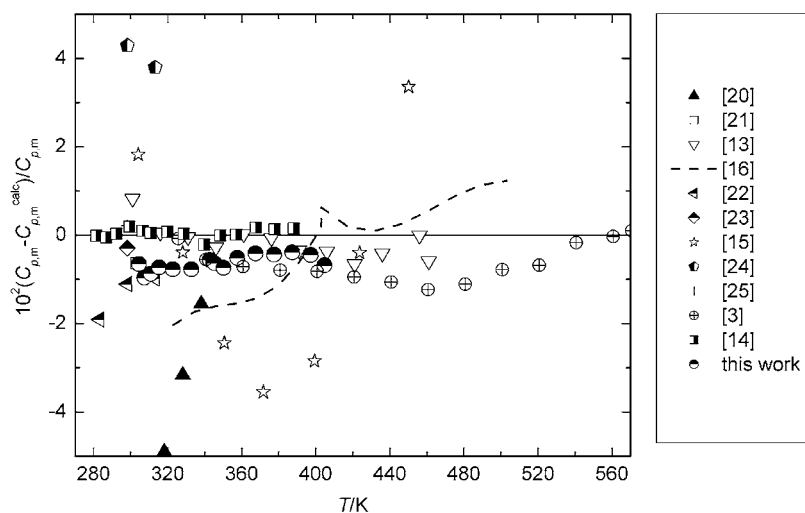


Fig. 2. Deviation plot for heat capacity of 1-decanol.

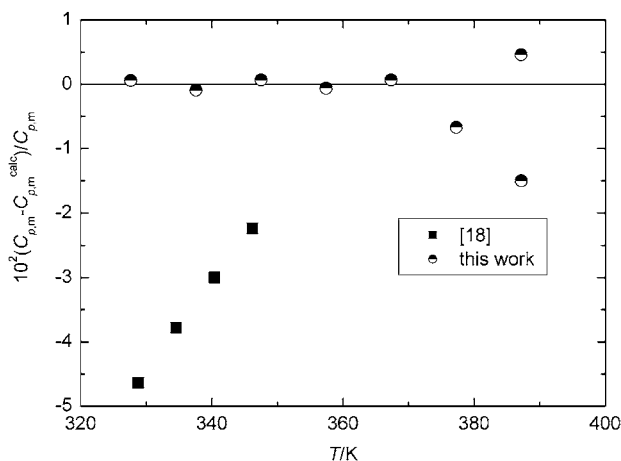


Fig. 4. Deviation plot for heat capacity of 1-hexadecanol.

than the stated ones. The data by Vasil'ev et al. [16,17] are presented as parameters of a quadratic equation in two temperature intervals. As seen from Figs. 2 and 3 the data in adjacent intervals do not have identical first and second derivative. For 1-hexadecanol we rejected the data by Moselman et al. [18] published in a study of polymorphism in the form of a linear equation covering the range from the melting temperature up to 346 K. For 1-octadecanol we selected also the data by Vasil'ev et al. [16] published in the form of a quadratic equation in three distinct temperature intervals, as this is the only dataset extending to high temperatures. Very accurate data for 1-octadecanol and 1-eicosanol measured by Van Miltenburg et al. [19] cover a narrow interval only.

It is worth noting that calorimetric records from our measurements indicate the investigated compounds may undergo some changes in the liquid phase. The obtained heat capacity depends on the speed of heating, on the previous history of the compound, in particular on the fact that the compound was cooled down below the melting temperature. However, the sensitivity of the C80 calorimeter does not permit a more detailed investigation and more comprehensive conclusion.

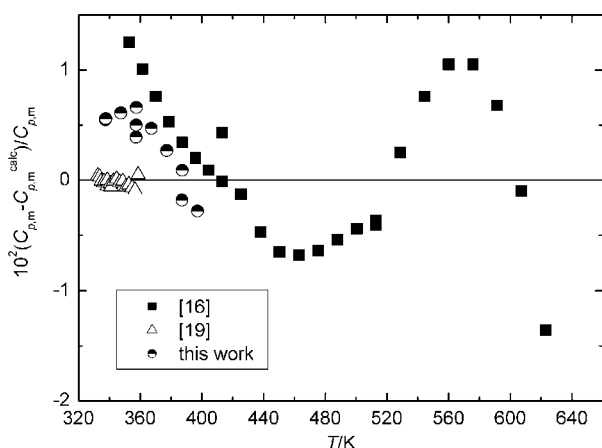


Fig. 5. Deviation plot for heat capacity of 1-octadecanol.

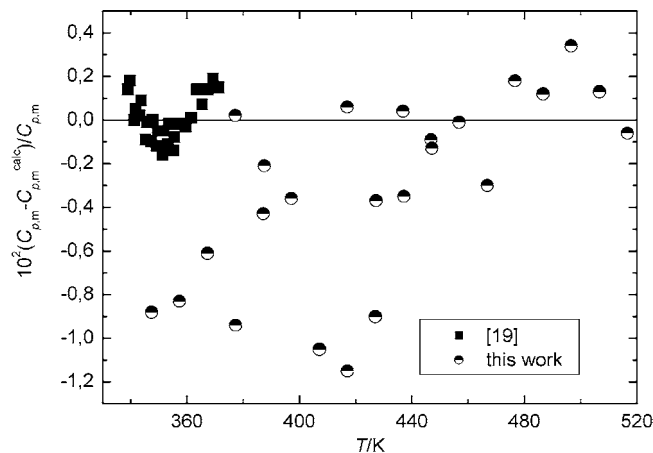


Fig. 6. Deviation plot for heat capacity of 1-eicosanol.

Similar observations for 1-tetradecanol were reported by Steele et al. [5]; they did not, however, bring up any explanation. Steele et al. [5] published only raw data over a wide temperature range from 324 to 564 K, but smoothed the data with a linear equation over a narrow temperature interval only, with parameters valid from 311 to 379 K. Similarly to our results for 1-eicosanol, reproducibility above 390 K is lower.

5. Conclusions

Recommended data on heat capacity of five liquid 1-alkanols from C₁₀ to C₂₀ were developed by critical assessment of newly determined and available literature data. Some sets of data published in the literature were rejected due to their large systematic deviations. The recommended data are presented in terms of parameters of empirical correlating equations expressing their dependence on temperature.

Acknowledgements

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