Saturated Heat Capacities of Some Linear and Branched Alkyl-Benzenes between 288 and 348 K

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The saturated heat capacities of benzene and of some linear (toluene, ethylbenzene, propylbenzene, butylbenzene, hexylbenzene, 1-phenylheptane, and 1-phenylhexadecane) and branched (*m*-xylene, cumene, sec-butylbenzene, tert-butylbenzene, and isobutylbenzene) alkyl-benzenes have been measured. A Micro DSC II from Setaram (France) with batch cells designed in our laboratory, and the "scanning or continuous method," have been used to perform the measurements. The estimated uncertainty of the saturated heat capacities was better than 0.3%. In the literature there exists $c_{\rm sat}(T)$ data for some of the liquids studied. Agreement with our measurements is better than 0.5%. For linear alkyl-benzenes and for each temperature, the variation in the saturated heat capacity with the alkyl length is nearly linear. The selected branched alkyl-benzenes are isomers of the linear alkyl-benzenes studied; accordingly, the influence of molecular structure on the saturated heat capacities has been analyzed.

KEY WORDS: alkyl-benzenes; Calvet type DSC; saturated heat capacity.

1. INTRODUCTION

The most important reason for the long operational life of refrigeration systems is that of compressor lubrication. A lubricant must be stable, provide excellent lubricity between the machine's moving parts, and be soluble in the refrigerant. Mineral oils have been largely used for lubricant

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purposes but also alkyl-benzenes, which are among one of the first synthetic lubricants ever developed. With the elimination of chlorine in refrigerants, lubricity and solubility of existing lubricants were diminished. However, some small systems, such as packaged air conditioners, vending machines, and home refrigerators, where the lubricant will return to the compressor in spite of poor solubility, have continued to use alkyl-benzene lubricants. Mixtures of alkyl-benzenes are also used as secondary thermal fluids in indirect heat pump and refrigeration systems to transport heat from the object to be cooled to the evaporator [1].

There are several requirements that any of these liquids must meet. Firstly, it should possess good thermophysical properties [2]. One of the most important properties with respect to the applications indicated is the heat capacity needed for designing refrigeration and air-conditioning systems, selecting the right lubricant for compressors and making calculations of the processes in which they are involved.

In this paper, measurements of the saturated heat capacity as a function of temperature of benzene and of some linear and branched alkylbenzenes are presented, with a temperature range from 288 to 348 K. Linear alkyl-benzenes studied are toluene, ethylbenzene, propylbenzene, butylbenzene, hexylbenzene, 1-phenylheptane, and 1-phenylhexadecane, while branched alkyl-benzenes studied are m-xylene, cumene, sec-butylbenzene, tert-butylbenzene, and isobutylbenzene. These branched alkylbenzenes have been selected because they are isomers of some of the linear alkyl-benzenes. In this way, the influence of the molecular structures of the liquids on their heat capacities might be analyzed.

2. EXPERIMENTAL

2.1. Materials

Benzene (Fluka AG, puriss p.a.; \geq 99.5 mol%), toluene (Fluka AG, puriss p.a.; \geq 99.5 mol%), ethylbenzene (Fluka AG, puriss p.a.; \geq 99 mol%), propylbenzene (Fluka AG, puriss; \geq 99 mol%), butylbenzene (Fluka AG, purum; \geq 99 mol%), hexylbenzene (Fluka AG, purum; \geq 97 mol%), 1-phenylheptane (Fluka AG, puriss; \geq 99 mol%), 1-phenylheptane (Fluka AG, puriss; \geq 99 mol%), 1-phenylheptane (Fluka AG, purum; \geq 97 mol%), m-xylene (Fluka AG, puriss p.a.; \geq 99 mol%), cumene (Fluka AG, purum; \geq 98 mol%), sec-butylbenzene (Fluka AG, purum; \geq 97 mol%), and isobutylbenzene (Fluka AG, purum; \geq 99 mol%), were used without further purification. Prior to measurements the liquids were stored over a molecular sieve type 4 Å from Fluka to remove any traces of water.

The results of the measurements of molar saturated heat capacities at 298.15 K and atmospheric pressure are listed in Table I and compared with published values, except for 1-phenylhexadecane, for which no data have been found in the literature. Agreement was good, although we observed some discrepancies which may be justified by the poor quality of some existing data. In fact, literature values for hexylbenzene and 1-phenylheptane given in Ref. 17 are taken from an old paper of 1948, in which only two points for each liquid were measured, and with an uncertainty above 3%. This is also the case for tert-butylbenzene, for which, according to the

	Molar saturated heat capacity, $c_{sat} (J \cdot K^{-1} \cdot mol^{-1})$		
Liquid	This work	Literature	
Benzene	135.58	135.76 [7]	
		135.64 [10]	
		135.707 [11]	
Toluene	157.15	157.29 [7]	
		157.15 [12]	
		157.057 [13]	
Ethylbenzene	185.74	185.57 [7]	
		185.76 [14]	
		185.572 [13]	
		185.83 [19]	
Propylbenzene	214.89	214.73 [7]	
		214.30 [15]	
Butylbenzene	243.48	244.17 [7]	
		242.76 [16]	
Hexylbenzene	281.86	290 ^a [17]	
1-Phenylheptane	332.56 ^b	322^{b} [17]	
1-Phenylhexadecane	617.37 ^c	-	
<i>m</i> -Xylene	182.03	183.44 [7]	
		181.637 [18]	
		181.550 [13]	
		181.66 [20]	
Cumene	215.02	215.2 [17]	
sec-Butylbenzene	243.25	243.6 [17]	
tert-Butylbenzene	241.43	239.6 ^a [17]	
Isobutylbenzene	242.93	240.6 [7]	

 Table I.
 Molar Saturated Heat Capacities of Alkyl-benzenes at 298.15 K and Atmospheric Pressure

^a Extrapolated value.

^b At 295 K.

^c At 308.15 K.

same reference, there is only one publication from 1930 on its heat capacity, and no specification relating to the purity of the liquid is given.

2.2. Equipment

In order to measure the molar saturated heat capacities, we have used a Micro DSC II micro-calorimeter from Setaram (France). The basic design of this apparatus is similar to that of a standard Calvet calorimeter [3], but with the further exception that the temperature is set by Peltier elements with an internal liquid (*n*-undecane) loop which flows around the calorimeter block. An additional external water circulation circuit is needed to provide or remove heat from the thermo-electric elements. This arrangement produces accurate temperature control, excellent thermal homogeneity, and low inertia, factors which account for its very good baseline stability. By applying two symmetrical semi-conducting heat–flow meters, sensitivity is increased to about 106 μ V ·mW⁻¹, which is double that of the thermopile detectors in the standard Calvet calorimeter. The assembly is set in an inert atmosphere of dry nitrogen.

The experimental calorimetric "batch" vessels used have been designed in our laboratory in such a way to avoid any leak so that the mass of the sample (and reference) can be considered constant throughout the measurement. The internal vessels volume is approximately 1 cm³. The features of these vessels have been described elsewhere [4].

The temperature is measured in the calorimetric block which contains the two vessels, but it does not correspond to the true temperature of the measured sample. For this reason the micro-calorimeter requires a temperature calibration procedure which has been undertaken by means of a calibrated Pt-1000 probe embedded in a stainless steel block of the same geometry as the vessels. This process has been carried out at different scanning rates, from 0° C·min⁻¹ (isothermal mode) to 0.2° C·min⁻¹, and the corrections smoothed with a polynomial equation, the coefficients of which have been introduced in the software of the apparatus. We estimate the final uncertainty of the temperature to be $\pm 0.01^{\circ}$ C for the isothermal mode and $\pm 0.03^{\circ}$ C for the scanning mode.

In this type of calorimeter the output signal measured by the instrument is proportional to the total heat flow rate. Therefore, we need to perform a caloric calibration to determine the proportionality coefficient between those two magnitudes at each temperature. To carry out this calibration, we have used the Setaram vessels specially designed to produce the electrical heating of an already known power in the socket of the measuring heat-flow meter with the EJ2 constant power source (Setaram). Taking into account the incertitude of this calibration method, those of the experimental procedure (scanning mode), and the purity of the materials used we consider that the uncertainty of our molar saturated heat capacity measurements is of the order of 0.3% [4, 5].

2.3. Experimental Procedure

The "scanning or continuous method" has been used to measure the saturated heat capacity of the alkyl-benzenes liquids with the differential scanning calorimeter. This method is based on the relation between the heat flow rate \dot{Q} ,—which is proportional to the measured calorimetric signal—, the overall heat capacity of the sample (liquid and vapor) inside the sample cell, c_i ,—which corresponds to saturation conditions in "batch" cells—, and the scanning rate β :

$$\dot{Q} = c_t(T) \beta \tag{1}$$

The experimental procedure consists of three steps in which the reference cell is always filled with a liquid (*n*-dodecane in this work):

- (a) Determination of the heat flow rate of the baseline with an empty sample cell, which we term \dot{Q} [Empty, C₁₂].
- (b) A calibration liquid (water) of a recognized heat capacity [6] is put into the sample cell. The corresponding heat flow rate is termed \dot{Q} [Water, C₁₂].
- (c) The calibration liquid is replaced by the sample liquid and the heat flow rate obtained is termed \dot{Q} [Sample, C₁₂].

By successively applying Eq. (1) to each of the above steps, we obtain the following ratio for the overall heat capacity of the sample:

$$(c_t)_{\text{Sample}} = \frac{m_{\text{Water}}}{m_{\text{Sample}}} (c_t)_{\text{Water}} \frac{\dot{Q}[\text{Sample, C}_{12}] - \dot{Q}[\text{Empty, C}_{12}]}{\dot{Q}[\text{Water, C}_{12}] - \dot{Q}[\text{Empty, C}_{12}]}$$
(2)

where m_{Water} and m_{Sample} denote the water and sample mass, respectively, and $(c_t)_{\text{Water}}$ the specific heat capacity of water inside the cell (liquid and vapor).

The scanning rate does not appear in Eq. (2), but in practice the one chosen must be fast enough to avoid very long experiments and slow enough to prevent excessive thermal delay between the programmed and the real heating or cooling rates, with the subsequent disturbance of the quasi steady-state condition in the cells. A scanning rate of $0.15 \text{ K} \cdot \text{min}^{-1}$ has been selected in our experiments. The scanning method allows a large

temperature range to be covered in one run. This gives a continuous heat capacity-temperature curve.

2.4. Corrections

The saturation heat capacity, c_{sat} , can be obtained from the calorimetric determinations of the overall heat capacity of a sample inside a "batch" cell, c_t , given by the Eq. (2). Only a few small corrections have to be applied in order to obtain the value of c_{sat} . Furthermore, for liquids it is not necessary to make any distinction below $\sim 0.9T_{nb}$ (which is the case in our measurements) between the isobaric heat capacity at the saturation curve, c_p , and c_{sat} as the differences are less than the experimental uncertainty.

In our "batch" cells the liquid fills most of the inner space and is in equilibrium with its vapor. The overall heat capacity of such a two-phase system, c_t , consists of the contribution from the saturation heat capacities of the liquid and vapor and further reflects the heat absorbed or evolved during vaporization or condensation inside the sample cell. The relationship between the measured c_t and c_{sat} can be obtained as follows.

The entropy of the two-phase equilibrium system, S_t , can be written as:

$$S_t = n^l s^l + n^v s^v \tag{3}$$

where s^l and s^v are the molar entropies of the coexisting phases and n^l and n^v are the amounts of substance in the liquid and vapor phases, respectively. As the cells are hermetically sealed, we consider the total amount of substance, $(n^l + n^v)$, to be constant throughout the experiment.

Considering the expression of heat capacity according to entropy, $C_x = T(\frac{\partial S}{\partial T})_x$, and by taking the temperature derivative of Eq. (3), we obtain the overall heat capacity of the substance inside the sample cell:

$$C_{t} = n^{l} c_{\text{sat}}^{l} + n^{v} c_{\text{sat}}^{v} + \Delta_{\text{vap}} H\left(\frac{\partial n^{v}}{\partial T}\right)_{\text{sat}}$$
(4)

where c_{sat}^l and c_{sat}^v are the saturation heat capacities in the liquid and vapor phases, respectively, and $\Delta_{\text{vap}}H$ is the heat of vaporization (or condensation) of the sample.

From Eq. (4) we can easily infer the correction terms which must be applied to the experimental measurement C_t to obtain the saturated heat capacity in the liquid phase, c_{sat}^l . First, it must be subtracted from C_t the second term in the right-hand part of Eq. (4), $n^v c_{sat}^v$, which corresponds to

	Ta	ible II. Ex	tperimental Mola	r Saturated Heat C	Capacities, c_{sat} , of	Alkyl-benzenes at	t Different Tempera	tures
					$c_{\mathrm{sat}} (\mathbf{J} \cdot \mathbf{K}^{-1} \cdot \mathrm{mol}^2)$	-1)		
$T\left(\mathbf{K} ight)$	Benzene	Toluene	Ethylbenzene	Propylbenzene	Butylbenzene	Hexylbenzene	1-Phenylheptane	1-Phenylhexadecane
288.15	133.31	154.31	182.07	210.72	236.66	297.43	327.22	
293.15	134.31	155.82	183.65	212.72	238.76	300.14	330.28	
298.15	135.59	157.30	185.71	214.84	241.42	303.08	333.24	
303.15	136.65	158.66	187.36	216.78	243.75	305.59	335.99	613.2
308.15	137.78	160.19	189.22	218.83	246.26	308.42	339.05	617.3
313.15	138.93	161.66	190.94	220.82	248.63	311.08	341.98	621.6
318.15	140.22	163.17	192.88	223.01	251.12	313.60	344.87	626.0
323.15	141.27	164.74	194.61	224.99	253.64	316.02	347.92	630.7
328.15	142.40	166.36	196.47	227.06	256.14		351.07	635.6
333.15	143.59	167.89	198.42	229.27	258.74		354.00	640.5
338.15	144.76	169.47	200.32	231.40	261.18		357.07	645.3
343.15	146.07	171.12	202.17	233.45	263.66		360.31	650.5
348.15	147.30	172.74	204.05	235.61	266.24		363.45	655.6

	$c_{\rm sat} \left(\mathbf{J} \cdot \mathbf{K}^{-1} \cdot \mathbf{mol}^{-1} \right)$						
T (K)	m-Xylene	Cumene	sec-Butylbenzene	tert-Butylbenzene	Isobutylbenzene		
288.15	178.36	210.96	238.97	236.66	237.97		
293.15	180.17	212.68	240.67	238.76	240.32		
298.15	182.01	215.00	243.21	241.42	242.93		
303.15	183.63	216.99	245.24	243.75	245.18		
308.15	185.49	219.13	247.55	246.26	247.66		
313.15	187.16	221.09	249.66	248.63	249.97		
318.15	188.94	223.23	251.89	251.12	252.55		
323.15	190.81	225.36	254.18	253.64	254.87		
328.15	192.72	227.34	256.69	256.14	257.31		
333.15	194.51		259.82	258.74	259.86		
338.15	196.39		262.10	261.18	262.35		
343.15	198.32		264.80	263.66	264.70		
348.15	200.25		267.29	266.24	267.19		

Table III. Experimental Molar Saturated Heat Capacities, c_{sat} , of Isomeric Alkyl-benzenesat Different Temperatures

the heat capacity of the substance in the vapor phase. This correction term can be calculated quite well, if we assume ideal behavior in the vapor phase. The ideal heat capacity is taken from the literature and the value of n^v at each temperature is calculated with the experimental measurements of the total amount of substance inside the sample cell, the vapor pressure, the density of the liquid, and the total volume of the cell.

Liquid	a_0	a_1	<i>a</i> ₂	<i>a</i> ₃	$\sigma\left(\mathbf{J}\cdot\mathbf{K}^{-1}\cdot\mathrm{mol}^{-1}\right)$
Benzene	66.133	0.2327			0.09
Toluene	104.75	0.0610	0.000386		0.04
Ethylbenzene	101.39	0.2075	0.000251		0.07
Propylbenzene	117.19	0.2503	0.000258		0.05
Butylbenzene	121.53	0.3195	0.000276		0.08
Hexylbenzene	44.590	1.184	-0.00106		0.09
1-Phenylheptane	202.44	0.2949	0.000481		0.09
1-Phenylhexadecane	2000.8	-13.69	0.0424	-0.0000408	0.09
<i>m</i> -Xylene	110.29	0.1319	0.000363		0.06
Cumene	91.560	0.4138			0.11
sec-Butylbenzene	231.52	-0.3466	0.00129		0.21
tert-Butylbenzene	121.53	0.3195	0.000276		0.08
Isobutylbenzene	97.655	0.4868			0.08

Table IV. Parameters a_i of Eq. (5) and Standard Deviations, σ

The second correction concerns the third term, $\Delta_{vap}H(\frac{\partial n^v}{\partial T})_{sat}$ which provides the contribution of vaporization (or condensation) heat during measurement. The heat of vaporization (or condensation) is obtained from the literature [7–9], and the derivative factor is evaluated by calculating the change of n^g in a short temperature interval.

The cell is always filled almost completely with the liquid in order to have a very small vapor phase, and although corrections are very small and in many cases negligible, we have applied them to all the heat capacity measurements presented in this paper.



Fig. 1. Saturated heat capacities of normal alkyl-benzenes as a function of temperature: \bigcirc benzene, \spadesuit toluene (taken from Ref. 10), \square ethylbenzene, \blacksquare propylbenzene, \triangle butylbenzene, \blacktriangle hexylbenzene, \diamondsuit 1-phenylheptane, \blacklozenge 1-phenylhexadecane. Full curves represent the smoothing Eq. (5) with coefficients of Table IV.

3. RESULTS AND DISCUSSION

Experimental values of the saturated heat capacities as a function of temperature of benzene and liquids *n*-alkyl-benzenes studied (toluene, ethylbenzene, propylbenzene, butylbenzene, hexylbenzene, 1-phenylheptane, and 1-phenylhexadecane) are presented in Table II, and of the corresponding alkyl-benzenes isomers (*m*-xylene, cumene, sec-butylbenzene, tertbutylbenzene, and isobutylbenzene) in Table III. The reported values correspond to the average of two consecutive experimental determinations, one heating and the other cooling. For each pure liquid, a polynomial function of the type



$$c_{\text{sat}} / (\mathbf{J} \cdot \mathbf{K}^{-1} \cdot \text{mol}^{-1}) = a_0 + a_1 T + a_2 T^2 + a_3 T^3$$
(5)

Fig. 2. Saturated heat capacities of normal alkyl-benzenes as a function of the number of carbon atoms in the alkyl chain at different temperatures: \bigcirc 348.15 K, \square 328.15 K, \triangle 308.15 K, \bigtriangledown 288.15 K.

was used to correlate the experimental values of c_{sat} according to temperature with the method of least squares with all points weighted equally. The number of coefficients used in Eq. (5) for each fit was determined by applying an F-test. The coefficients a_i , together with the standard deviations σ obtained in the regression, are shown in Table IV.

Graphical representations of our measurements of saturated heat capacities, c_{sat} , of pure liquids as a function of temperature are given in Fig. 1 for alkyl-benzenes, in Fig. 3 for ethylbenzene and isomer *m*-xylene, in Fig. 4 for propylbenzene with isomer cumene, and in Fig. 5 for butyl-benzene and its isomers sec-butylbenzene, tert-butylbenzene and isobutyl-benzene. We have also represented the values (full line) given by Eq. (5) with the coefficients of Table IV.

In the literature $c_{sat}(T)$ data exist for some of the liquids studied in this paper, and we can compare our measurements with these. All the data relating to the existing heat capacities of organic and inorganic liquid compounds have been collected and critically assessed in the recent monograph of Ref. 17. All our experimental data for benzene, ethylbenzene, propylbenzene, butylbenzene, *m*-xylene and cumene agree better than



Fig. 3. Saturated heat capacities of \bigcirc ethylbenzene and the isomer \triangle *m*-xylene. Full curves represent the smoothing Eq. (5) with coefficients of Table IV.

0.5% with the correlated heat capacities calculated in the monograph. Nevertheless, in the case of the isomers sec-butylbenzene and tert-butylbenzene, our data have greater discrepancies. This may be attributed to the poor level of uncertainty (1 per cent) assigned by the evaluators to the data generated from the correlation equations, and to the fact that there is only one reference in the given temperature sub-interval for each liquid, which in the case of tert-butylbenzene is very old.

For the liquids isobutylbenzene, hexylbenzene, 1-phenylheptane, and 1-phenylhexadecane, there are no experimental data in the literature for comparison.

From Fig. 1, it can be inferred that the temperature-dependent saturated heat capacities of linear alkyl-benzenes, $c_{sat}(T)$, increase as the alkyl chain length increases. The increment generated in $c_{sat}(T)$ by the addition of a new methylene group is apparently constant. In this regard, the heat capacities as a function of the number of carbon atoms in the alkyl chain at different temperatures have been represented in Fig. 2, and it can be concluded that the dependence is linear at least up to an alkyl chain of



Fig. 4. Saturated heat capacities of \bigcirc propylbenzene and the isomer \triangle cumene. Full curves represent the smoothing Eq. (5) with coefficients of Table IV.

sixteen carbon atoms. This behavior is similar to that obtained by Finke et al. [21] for the saturated heat capacities of a series of alkylcyclohexanes.

It is worth comparing the saturated heat capacities of ethylbenzene, propylbenzene and butylbenzene with those of their isomers. It can be observed in Fig. 3 that, in the case of ethylbenzene, the saturated heat capacity of this compound is higher than that of its isomer *m*-xylene. According to Fig. 4, propylbenzene has a saturated heat capacity which is practically equal to that of the isomer cumene at each temperature. From Fig. 5 we conclude that the saturated heat capacities of butylbenzene and those of its three isomers studied (sec, tert and iso) are also very similar, with the exception of tert-butylbenzene, which is slightly smaller. This behavior can be related to the structures of the molecules. In Table V, the



Fig. 5. Saturated heat capacities of butylbenzene and its isomers in function of temperature: \bigcirc butylbenzene, \triangle sec-butylbenzene, \bigtriangledown isobutylbenzene, \square tert-butylbenzene. Full curves represent the smoothing Eq. (5) with coefficients of Table IV.



Table V. Molecular Structures of Linear Alkyl-Benzenes and Isomers

different alkyl-benzenes have been graphed and the alkyl chains connected to the benzene ring are clearly shown. When the alkyl chain is similar in size and shape in the linear alkyl-benzene and in the isomer molecules, the corresponding heat capacities are nearly identical. This is the case with cumene, sec-butylbenzene, and isobutylbenzene. However, the alkyl chains in the other isomers differ in shape and size from those in the linear alkylbenzene, especially as regards *m*-xylene, where two alkyl groups are connected separately to the benzene ring. When this occurs, the heat capacity is clearly sensitive to these differences in shape and size.

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