

Standard molar enthalpies of formation of three *N*-benzoylthiocarbamic-*O*-alkylesters

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Abstract

The standard ($p^\circ = 0.1$ MPa) molar enthalpies of combustion in oxygen of three crystalline *N*-benzoylthiocarbamic-*O*-alkylesters, PhCONHCSOR, R = Et (Hbtcee), *n*-Bu (Hbtcebe), *n*-Hex (Hbtche), were measured at $T = 298.15$ K by rotating bomb calorimetry. The standard molar enthalpies of sublimation of the three compounds were measured using Calvet microcalorimetry. These values were used to derive the standard molar enthalpies of formation of the compounds in their crystalline and gaseous phases, respectively.

	$-\Delta_c U_m^\circ(\text{cr})/(\text{kJ} \cdot \text{mol}^{-1})$	$\Delta_{\text{cr}}^\circ H_m^\circ/(\text{kJ} \cdot \text{mol}^{-1})$
<i>N</i> -benzoylthiocarbamic- <i>O</i> -ethylester	5777.9 ± 2.2	112.2 ± 1.3
<i>N</i> -benzoylthiocarbamic- <i>O</i> - <i>n</i> -butylester	7094.5 ± 2.5	120.7 ± 1.8
<i>N</i> -benzoylthiocarbamic- <i>O</i> - <i>n</i> -hexylester	8402.2 ± 3.1	139.7 ± 2.4

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1. Introduction

N-benzoylthiocarbamic-*O*-alkylesters had their first appearance as early as 1874 reported by Lössner [1] followed by the identification by Dixon [2] in 1895. Although mainly used in heterocyclic chemistry in the late 60s of the last century [3–6] and applied as collectors in ore flotation processing [7,8], they seem to have been overlooked as chelating ligands in spite of their easy synthetical access. Also, the extraction behaviour of the *N*-benzoylthiocarbamic-*O*-alkylesters

towards metal ions was the subject of investigations [9]. They were proposed as intermediates in regio- and chemoselective deoxygenation of primary and secondary aliphatic alcohols [10]. The first reference due to their coordinating properties was published in 1995 [11]; recently, more attention has been devoted to their thermodynamical behaviour in solution [12].

There are no reported values for thermochemical properties of the *N*-benzoylthiocarbamic-*O*-alkylesters so far, neither in the condensed nor in the gaseous phases. As part of a broad thermochemical study on compounds containing double bonded oxygen as well as sulphur (e.g. monothio- β -diketones and *N*-acylthioureas) this paper reports on the thermochemistry of the

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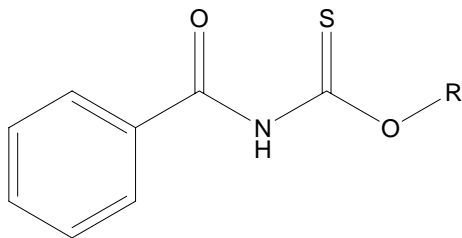


FIGURE 1. General formula of the *N*-benzoylthiocarbamic-*O*-alkylester. R = Et: *N*-benzoylthiocarbamic-*O*-ethylester (Hbtcee); R = *n*-Bu: *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtcbe); R = *n*-Hex: *N*-benzoylthiocarbamic-*O*-*n*-hexylester (Hbtche).

following compounds: *N*-benzoylthiocarbamic-*O*-ethylester (Hbtcee), *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtcbe), and *N*-benzoylthiocarbamic-*O*-*n*-hexylester (Hbtche), as shown in figure 1.

The presented results enabled the standard molar enthalpies of formation in both crystalline and gaseous phases to be derived.

The obtained data for the condensed and gaseous phases are necessary for a thermochemical study of the metal complexes, in order to derive thermochemical parameters for the metal–ligand binding.

2. Experimental

The *N*-benzoylthiocarbamic-*O*-alkylesters were prepared as described earlier [11]: 0.1 mol (16.3 g) of benzoyl isothiocyanate were dissolved in 60 cm³ of toluene; the corresponding alcohol was added dropwise in twofold molar excess at room temperature under stirring and slowly heated up to 333 K for 1 h. Overnight, the reaction mixture was evaporated to dryness. The remaining crude solid was dissolved in methanol, and water was added up to maintaining turbidness. Continually repeating of this procedure leads to pure, yellow crystals, with $T_{\text{fus}} = 347$ K (Hbtcee), 330–331 K (Hbtcbe) and 346 K (Hbtche).

The purity of the samples was checked by i.r. spectroscopy and by elemental analysis; the mass fraction w of C, H, N and S were as follows: for Hbtcee, C₁₀H₁₁NO₂S, found 10² $w(\text{C}) = 56.94$, 10² $w(\text{H}) = 5.22$, 10² $w(\text{N}) = 6.86$, 10² $w(\text{S}) = 16.37$, calculated 10² $w(\text{C}) = 57.39$, 10² $w(\text{H}) = 5.30$, 10² $w(\text{N}) = 6.69$, 10² $w(\text{S}) = 15.32$; for Hbtcbe, C₁₂H₁₅NO₂S, found 10² $w(\text{C}) = 60.56$, 10² $w(\text{H}) = 6.41$, 10² $w(\text{N}) = 5.89$, 10² $w(\text{S}) = 13.54$, calculated 10² $w(\text{C}) = 60.73$, 10² $w(\text{H}) = 6.37$, 10² $w(\text{N}) = 5.90$, 10² $w(\text{S}) = 13.51$; for Hbtche, C₁₄H₁₉NO₂S, found 10² $w(\text{C}) = 62.73$, 10² $w(\text{H}) = 7.19$, 10² $w(\text{N}) = 5.24$, 10² $w(\text{S}) = 12.76$, calculated 10² $w(\text{C}) = 63.40$, 10² $w(\text{H}) = 7.17$, 10² $w(\text{N}) = 5.28$, 10² $w(\text{S}) = 12.08$.

3. Combustion calorimetry

The enthalpies of combustion were measured with the rotating-bomb calorimeter formerly used at the National Physical Laboratory, Teddington, UK [13], with a platinum-lined bomb of internal volume 0.337 dm³. Water was added to the calorimeter from a weighed acrylic vessel, and for each experiment a correction to the energy equivalent was made for the deviation from 4059.0 g of the mass of water added. Calorimetric temperatures were measured to 1 · 10^{−4} K with a quartz thermometer (Hewlett Packard HP 2804) interfaced to a microcomputer programmed to compute the adiabatic temperature change. Ignition temperatures were chosen so that the final temperatures were very close to 298.15 K. Fore-period readings were taken for about 20 min, the main period was about 25 min and the after period about 20 min. The frictional work of bomb rotation was automatically included in the correction for heat exchange and work of stirring by using the procedure described by Good *et al.* [14].

The energy equivalent of the calorimeter was determined from the combustion of benzoic acid (Bureau of Analysed Samples; Thermochemical Standard BCS-CRM 190-r), as described previously [15]. The electrical energy for the ignition was determined from the change in potential across a capacitor when 40 V were discharged through a platinum ignition wire. For the cotton thread fuse (empirical formula CH_{1.686}O_{0.843}), the massic energy of combustion is assigned to $-\Delta_c u^\circ = 16250 \text{ J} \cdot \text{g}^{-1}$ [15], a value which has been confirmed in our laboratory. Corrections for nitric acid formation were based on $-59.7 \text{ kJ} \cdot \text{mol}^{-1}$ for the molar energy of formation of 0.1 mol · dm^{−3} HNO₃(aq) from O₂, N₂, and H₂O(l) [16].

From series of eight calibration experiments the energy equivalent of the calorimeter, $\epsilon(\text{calor})$, were found to be $(20684.5 \pm 4.0) \text{ J} \cdot \text{K}^{-1}$ (for combustion experiments with Hbtcee) and $(20690.2 \pm 3.3) \text{ J} \cdot \text{K}^{-1}$ (for combustion experiments with Hbtcbe and Hbtche series) for an average mass of water added to the calorimeter of 4059.0 g; the quoted uncertainty refers to the standard deviation of the mean.

Samples in pellet form were ignited in oxygen at a pressure of 3.04 MPa with a volume of 10 cm³ of water added to the bomb. The amount of nitric acid was determined using the Devarda alloy method [17]. The densities of the three crystalline *N*-benzoylthiocarbamic-*O*-alkylesters were assumed to be 1.3 g · cm^{−3} and for benzoic acid $\rho = 1.32 \text{ g} \cdot \text{cm}^{-3}$. For each compound, $(\partial u / \partial p)_T$ at $T = 298.15 \text{ K}$ was assumed to be $-0.2 \text{ J} \cdot \text{g}^{-1} \cdot \text{MPa}^{-1}$, a typical value for solid organic compounds [18].

Standard state corrections were calculated by the procedures given by Hubbard *et al.* [19] and by Good and Scott [20]. The relative atomic masses used were

those recommended by the IUPAC Commission in 2001 [21].

4. Microcalorimetry

The standard molar enthalpies of sublimation were measured using the “vacuum sublimation” drop microcalorimetric method [22,23]. Samples of about 5 mg of each compound, contained in thin glass capillary tubes and sealed at one end, were dropped from room temperature into the hot reaction vessel in the Calvet High Temperature Microcalorimeter (SETARAM HT 1000D), held at the temperature $T = 375$ K and then removed from the hot zone by vacuum sublimation. The observed standard molar enthalpies of sublimation $\{H_m^\circ(g, T) - H_m^\circ(\text{cr}, 298.15 \text{ K})\}$ were corrected to $T = 298.15$ K using $\{H_m^\circ(g, T) - H_m^\circ(g, 298.15 \text{ K})\}$ estimated by a group method based on the values of Stull *et al.* [24]. This is, $\text{PhCONHCSOEt} = \text{PhCHO} + \text{HNCS} + \text{EtOH} - \text{H}_2$, which gives $\Delta_{298.15 \text{ K}}^T H_m^\circ(g) = (17.0 \pm 1.0) \text{ kJ} \cdot \text{mol}^{-1}$ for Hbtcee, $\text{PhCONHCSO-}n\text{-Bu} = \text{PhCHO} + \text{HNCS} + n\text{-BuOH} - \text{H}_2$, with $\Delta_{298.15 \text{ K}}^T H_m^\circ(g) = (20.8 \pm 1.0) \text{ kJ} \cdot \text{mol}^{-1}$ for Hbtcebe, $\text{PhCONHCSO-}n\text{-Hex} = \text{PhCHO} + \text{HNCS} + n\text{-HexOH} - \text{H}_2$, which gives $\Delta_{298.15 \text{ K}}^T H_m^\circ(g) = (24.7 \pm 1.0) \text{ kJ} \cdot \text{mol}^{-1}$ for Hbtche, all at $T = 375$ K.

The calorimeter was calibrated *in situ*, making use of the reported standard molar enthalpy of sublimation of naphthalene, C_{10}H_8 , $\Delta_{\text{cr}}^g H_m^\circ = (72.513 \pm 0.071) \text{ kJ} \cdot \text{mol}^{-1}$ [25]. The calibration constant of the calorimeter was obtained as the average of six independent

experiments at $T = 375$ K as $K = (0.9861 \pm 0.0050)$. The temperature was measured *in situ* using a small size Pt100 probe previously calibrated against a SPRT (25 Ω) Tinsley 5187SA standard probe using an ASL bridge F26. The dependency of the blank tube sign from the mass difference of the blank tubes in the reference and measuring cell was taken into account [23].

5. Results

Table 1 lists typical combustion results for each compound in which $\Delta m(\text{H}_2\text{O})$ is the deviation of the mass added to the calorimeter from 4059.0 g, the mass assigned to $\varepsilon(\text{calor})$, and ΔU_Σ is the correction to the standard state. The remaining quantities are as previously described [19].

The internal energy for the isothermal bomb process, $\Delta U(\text{IBP})$, was calculated according to:

$$\Delta U(\text{IBP}) = -\{\varepsilon(\text{calor}) + c_p(\text{H}_2\text{O}, 1)\Delta m(\text{H}_2\text{O})\}\Delta T_{\text{ad}} + (T_i - 298.15)\varepsilon_i + (298.15 - T_i - \Delta T_{\text{ad}})\varepsilon_f + \Delta U(\text{ign}), \quad (1)$$

where ΔT_{ad} is the calorimeter adiabatic temperature change corrected for the heat exchange and the work of stirring.

For each compound, the products of combustion in the experiments consist of a gaseous phase and an aqueous mixture of sulphuric acid for which the thermodynamic properties are known.

The values of $-\Delta u_c^\circ$ refer to the reaction

TABLE 1

Typical combustion experimental results for *N*-benzoylthiocarbamic-*O*-ethylester (Hbtcee), *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtcebe), *N*-benzoylthiocarbamic-*O*-*n*-hexylester (Hbtche), at $T = 298.15$ K

	Hbtcee	Hbtcebe	Hbtche
$m(\text{cpd})/\text{g}$	0.89814	0.79091	0.86827
$m'(\text{fuse})/\text{g}$	0.00554	0.00527	0.00561
T_i/K	297.1467	297.1150	296.9896
T_f/K	298.3667	298.2926	298.3525
$\varepsilon_i/(\text{J} \cdot \text{K}^{-1})$	53.93	53.95	4.03
$\varepsilon_f/(\text{J} \cdot \text{K}^{-1})$	53.46	53.65	53.96
$\Delta m(\text{H}_2\text{O})/\text{g}$	-18.4	16.9	1.3
$\Delta T_{\text{ad}}/\text{K}$	1.20759	1.15147	1.33318
$-\Delta U(\text{IBP})/\text{J}$	24949.25	23803.60	27661.94
$\Delta U(\text{HNO}_3)/\text{J}$	39.57	45.48	49.11
$\Delta U(\text{ign})/\text{J}$	1.20	1.19	1.06
$\Delta U_\Sigma/\text{J}$	26.58	26.11	28.29
$m' \Delta_c u^\circ(\text{fuse})/\text{J}$	89.97	85.58	91.11
$-\Delta_c u^\circ(\text{cpd})/(\text{J} \cdot \text{g}^{-1})$	27604.97	29897.75	31664.61

$\Delta U(\text{IBP})$ includes $\Delta U(\text{ign})$; $m(\text{cpd.})$ is the mass of compound burnt in each experiment; $m(\text{fuse})$ is the mass of fuse (cotton) used in each experiment; T_i is the initial temperature; T_f is the final temperature rise; ε_i is the energy equivalent of contents in the initial state; ε_f is the energy equivalent of contents in the final state; $\Delta m(\text{H}_2\text{O})$ is the deviation of mass of water added to the calorimeter from 4059.0 g; ΔT_{ad} is the corrected temperature rise; $\Delta U(\text{IBP})$ is the energy change for the isothermal combustion reaction under actual bomb conditions; $\Delta U(\text{HNO}_3)$ is the energy correction for the nitric acid formation; $\Delta U(\text{ign})$ is the electrical energy for ignition; ΔU_Σ is the standard state correction; $\Delta_c u^\circ(\text{fuse})$ is the massic energy of combustion of the fuse (cotton); $\Delta_c u^\circ$ is the standard massic energy of combustion.

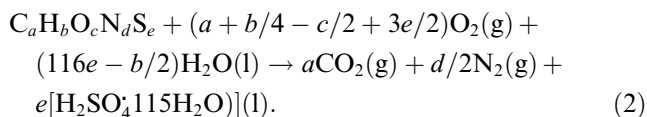


Table 2 lists the individual values of $-\Delta_c u$ together with the mean and its standard deviation. Table 3 lists for each compound the derived standard molar values for the energies and enthalpies of the combustion reactions, $\Delta_c U_m^\circ$ and $\Delta_c H_m^\circ$, the standard molar enthalpy of formation, $\Delta_f H_m^\circ$, of the crystalline solids as well as the standard molar enthalpies of sublimation and the derived standard molar enthalpies of formation in the gaseous state. In accordance with the normal thermochemical practice [26], the uncertainties assigned to the standard molar enthalpies of combustion and formation are twice the overall standard deviation of the mean and include the uncertainties in calibration and in the auxiliary quantities used. To derive $\Delta_f H_m^\circ(\text{cr})$ from $\Delta_c H_m^\circ(\text{cr})$, the standard molar enthalpies of formation of $\text{H}_2\text{O}(\text{l})$, of $\text{CO}_2(\text{g})$ and H_2SO_4 in $115 \text{ H}_2\text{O}(\text{l})$, at $T = 298.15 \text{ K}$, respectively, $-(285.830 \pm 0.040) \text{ kJ} \cdot \text{mol}^{-1}$ [27], $-(393.51 \pm 0.13) \text{ kJ} \cdot \text{mol}^{-1}$ [27], and $-(887.81 \pm 0.01) \text{ kJ} \cdot \text{mol}^{-1}$ were used [16].

The individual results of the measurements of the enthalpies of sublimation for *N*-benzoylthiocarbamic-*O*-ethylester, *N*-benzoylthiocarbamic-*O*-*n*-butylester and

TABLE 2

Individual values of the massic energy of combustion, $-\Delta_c u^\circ$ of *N*-benzoylthiocarbamic-*O*-ethylester (Hbtcee), *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtcbe), *N*-benzoylthiocarbamic-*O*-*n*-hexylester (Hbtche), at $T = 298.15 \text{ K}$ ($p^\circ = 0.1 \text{ MPa}$)

Hbtcee	Hbtcbe	Hbtche
$-\Delta_c u^\circ/(\text{J} \cdot \text{g}^{-1})$		
27605.0	29897.8	31664.6
27615.4	29895.2	31688.1
27616.1	29894.2	31655.6
27611.6	29896.3	31663.2
27602.1	29892.5	31667.4
27613.0	29896.3	31666.8
	29889.9	31665.8
$-\langle \Delta_c u^\circ \rangle/(\text{J} \cdot \text{g}^{-1})^a$		
27610.5 ± 4.7	29894.6 ± 2.0	31667.4 ± 7.5

The mean values are represented by $\langle \Delta_c u^\circ \rangle$.

^a Mean value; uncertainty given as twice the standard deviation of the mean.

TABLE 3

Derived standard ($p^\circ = 0.1 \text{ MPa}$) molar energies of combustion, $-\Delta_c U_m^\circ$, standard molar enthalpies of combustion, $\Delta_c H_m^\circ$, standard molar enthalpies of formation, $\Delta_f H_m^\circ$, and standard molar enthalpies of sublimation, $\Delta_{\text{cr}}^g H_m^\circ$, for *N*-benzoylthiocarbamic-*O*-ethylester (Hbtcee), *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtcbe), *N*-benzoylthiocarbamic-*O*-*n*-hexylester (Hbtche), at $T = 298.15 \text{ K}$

	$-\Delta_c U_m^\circ(\text{cr})/(\text{kJ} \cdot \text{mol}^{-1})$	$-\Delta_c H_m^\circ(\text{cr})/(\text{kJ} \cdot \text{mol}^{-1})$	$-\Delta_f H_m^\circ(\text{cr})/(\text{kJ} \cdot \text{mol}^{-1})$	$\Delta_{\text{cr}}^g H_m^\circ/(\text{kJ} \cdot \text{mol}^{-1})$	$-\Delta_f H_m^\circ(\text{g})/(\text{kJ} \cdot \text{mol}^{-1})$
Hbtcee	5777.9 ± 2.2	5784.7 ± 2.2	324.5 ± 2.9	112.2 ± 1.3	212.3 ± 3.2
Hbtcbe	7094.5 ± 2.5	7103.8 ± 2.5	364.1 ± 3.0	120.7 ± 1.8	243.4 ± 3.5
Hbtche	8402.2 ± 3.1	8414.0 ± 3.1	411.2 ± 4.0	139.7 ± 2.4	271.5 ± 4.7

TABLE 4

Observed standard molar enthalpies of sublimation $\Delta_{\text{cr}}^{g,T} H_m^\circ$ of *N*-benzoylthiocarbamic-*O*-ethylester (Hbtcee), *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtcbe), *N*-benzoylthiocarbamic-*O*-*n*-hexylester (Hbtche), at $T = 375 \text{ K}$

Hbtcee	Hbtcbe	Hbtche
$\Delta_{\text{cr}}^{g,T} H_m^\circ/(\text{kJ} \cdot \text{mol}^{-1})$		
128.7	141.8	164.5
128.6	139.8	163.9
129.0	140.4	168.3
130.3	143.9	166.4
129.4	141.5	162.0
		161.4
$\langle \Delta_{\text{cr}}^{g,T} H_m^\circ \rangle/(\text{kJ} \cdot \text{mol}^{-1})^a$		
129.2 ± 0.6	141.5 ± 1.4	164.4 ± 2.1

The mean values are represented by $\langle \Delta_{\text{cr}}^{g,T} H_m^\circ \rangle$.

^a Mean value; uncertainty given as twice the standard deviation of the mean.

N-benzoylthiocarbamic-*O*-*n*-hexylester are given in Table 4 with uncertainties equal to twice the standard deviation of the mean.

6. Discussion

There are no previously determined values for the standard molar enthalpies of formation of these compounds and, at present, only a limited number of thermochemical studies on compounds containing similar structures are available.

The obtained results are shown in figure 2 and they are consistent in themselves. Linear regression gives $\Delta_f H_m^\circ(\text{g})/\text{kJ} \cdot \text{mol}^{-1} = -14.8 \cdot n_C - 183.3$ ($r^2 = 0.9991$) with n_C as the number of carbon atoms in the alkyl chain ($n_C > 1$). The slope of $-14.8 \text{ kJ} \cdot \text{mol}^{-1}$, is the increment in the standard molar enthalpy of formation, in the gaseous state, due to the CH_2 -group introduction in the alkyl chain, which is lower than that normally observed in linear alkyl chains, of $-20.5 \text{ kJ} \cdot \text{mol}^{-1}$, expected from the Group Scheme. Figure 2 also shows the application of this group contribution, $[\text{C}-(\text{C})(\text{H})_2] = -20.5 \text{ kJ} \cdot \text{mol}^{-1}$ [28], on the obtained experimental value of *N*-benzoylthiocarbamic-*O*-*n*-butylester; this procedure leads to values distinctively lying out of the range of the assigned experimental errors.

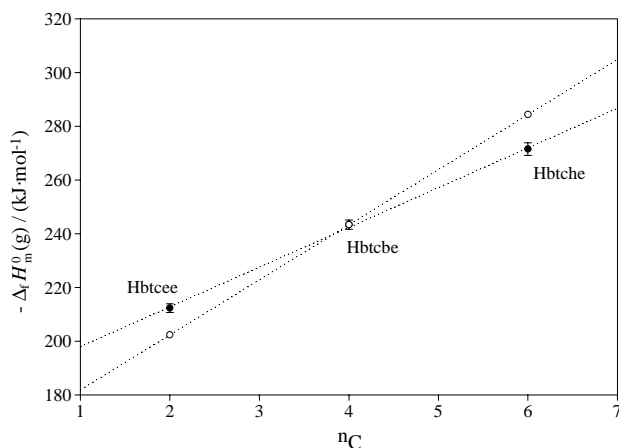


FIGURE 2. Plot of experimental values of $\Delta_f H_m^\circ(\text{g})$ (•) with the respective values derived by the Group scheme (○) [starting point: *N*-benzoylthiocarbamic-*O*-*n*-butylester (Hbtche)], against the number of carbons in the alkyl chain $n_C > 1$.

This difference is possibly an expression for the increasing instability of the molecule caused by a prolongation in the alkyl chain, which may give rise to steric hindrance in the surroundings of the ester–oxygen–atom.

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