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THREE N_2O_2 LIGANDS DERIVED FROM THE CONDENSATION OF 1,2-CYCLOHEXANEDIAMINE WITH SALICYLALDEHYDE, ACETYLACETONE AND BENZOYLACETONE
A new contribution to the energetical characterization of Schiff bases

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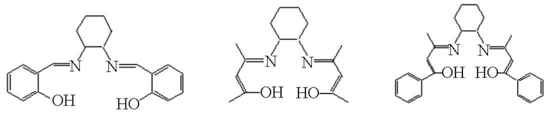
The standard (p^0=0.1 MPa) molar enthalpies of formation, at T=298.15 K, in the gaseous phase, for three tetradentate Schiff bases involving a N_2O_2 set, *N,N*'-bis(salicylaldehydo)cyclohexanediimine (H_2salch), *N,N*'-bis(acetylacetone)cyclohexanediimine (H_2acacch) and *N,N*'-bis(benzoylacetone)cyclohexanediimine (H_2bzacch), were determined from their enthalpies of combustion and sublimation, obtained by static bomb calorimetry in oxygen and by the Knudsen effusion technique, respectively. The results are compared with identical parameters for related compounds previously studied, resulting from the condensation of salicylaldehyde or β-diketone with aliphatic diamines.

Keywords: *enthalpy of formation, enthalpy of sublimation, N,N'-bis(acetylacetone)cyclohexanediimine, N,N'-bis(benzoylacetonecyclohexanediimine, N,N'-bis(salicylaldehydo)cyclohexanediimine, thermochemistry*

Introduction

The characterization of Schiff bases with a N_2O_2 donor atom set has been done extensively by the scientific community, due to their relevance in biological activity and, more recently, by their applications on catalysis [1, 2]. Schiff base ligands are considered 'privileged ligands' to stabilize several different transition metals in different oxidation states, and the resulting complexes have been successfully applied in catalytic reactions control, mainly due to their high activity, selectivity and enantioselectivity [3–5].

We have been also involved in the study of the thermochemical properties of several molecules of this class of tetradentate ligands derived from condensation of salicylaldehyde and β-diketones with aliphatic alkyldiamines [6, 7], as well as of some of their metallic complexes [8, 9]. In the present study, we report a new contribution for the energetical characterization of N_2O_2 tetradentate Schiff bases resulting from condensation of salicylaldehyde, acetylacetone or benzoylacetone with 1,2-cyclohexanediamine, yielding *N,N*'-bis(salicylaldehydo)cyclohexanediimine, *N,N*'-bis(acetylacetone)cyclohexanediimine or *N,N*'-bis(benzoylacetone)cyclohexanediimine, respectively, whose structural formula are represented in Fig. 1.



H_2salch H_2acacch H_2bzacch

Fig. 1 Structural formulae for *N,N*'-bis(salicylaldehydo)cyclohexanediimine (H_2salch), *N,N*'-bis(acetylacetone)cyclohexanediimine (H_2acacch) and *N,N*'-bis(benzoylacetone)cyclohexanediimine (H_2bzacch)

The enthalpies of formation of these three crystalline compounds and their enthalpies of sublimation, at T=298.15 K, have been derived from static bomb calorimetry and from Knudsen effusion technique, respectively. These results yield the standard molar enthalpies of formation of the gaseous species, which are interpreted in terms of the structural characteristics of the compounds.

Experimental

Synthesis and characterization of compounds

The compound *N,N*'-bis(salicylaldehydo)cyclohexanediimine, $C_{20}H_{22}N_2O_2$ (H_2salch), was prepared according a literature method [10], by refluxing 0.05 mol of salicylaldehyde (Merck-Schuchard, puri-

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fied by distillation) and 0.025 mol of 1,2-cyclohexanediamine (Aldrich Chemie, 99%, purified by distillation) in dried ethanol (30 cm^3), for 30 min, and cooling the reaction mixture. The compound was separated as a yellow powder and recrystallized twice from ethanol (yield ≈70%). The compound is stable at room temperature and has been characterized by IR spectroscopy and elemental analysis.

The compounds *N,N*'-bis(acetylacetone)cyclohexanediimine, $C_{16}H_{26}N_2O_2$ (H_2acacch) and *N,N*'-bis(benzoylacetone)cyclohexanediimine, $C_{26}H_{30}N_2O_2$ (H_2bzacch) were also prepared according to literature methods [11]: 0.05 mol of recently distilled 1,2-cyclohexanediamine (Aldrich Chemie, 99%), was added, slowly, to 0.1 mol of freshly distilled acetylacetone (Merck, p.a.) or 0.025 mol of sublimed benzoylacetone (Merck, for synthesis), in hot dried refluxing ethanol. The solutions were boiled for half an hour, after which the mixtures were cooled to room temperature and the colourless precipitates were filtered off, washed with cold ethanol and recrystallized twice from ethanol (yields ≈50% and ≈70%, respectively). The compounds have been characterized by IR spectroscopy and elemental analysis.

The three compounds were stored in desiccators over phosphorus(V) oxide. The analytical results (Microanalytical Services of the University of Manchester) are as follows for the mass fractions w: for H_2salch ($C_{20}H_{22}N_2O_2$), found 10^2 w(C)=74.6, 10^2 w(H)=7.0, 10^2 w(N)=8.8, calculated 10^2 w(C)=74.5, 10^2 w(H)=6.9, 10^2 w(N)=8.7; for H_2acacch ($C_{16}H_{26}N_2O_2$), found 10^2 w(C)=69.1, 10^2 w(H)=9.5, 10^2 w(N)=10.0, calculated 10^2 w(C)=69.0, 10^2 w(H)=9.4, 10^2 w(N)=10.1; for H_2bzacch ($C_{26}H_{30}N_2O_2$), found 10^2 w(C)=77.2, 10^2 w(H)=7.6, 10^2 w(N)=7.1, calculated 10^2 w(C)=77.6, 10^2 w(H)=7.5, 10^2 w(N)=7.0.

The purity of the compounds has also been confirmed by the CO_2 recoveries in the combustion experiments. The average ratios of the mass of carbon dioxide recovered to that calculated from the mass of sample, together with the standard deviation of the mean, were: H_2salch, (1.0006±0.0003); H_2acacch, (0.9999±0.0004); H_2bzacch, (1.0000±0.0002). The density at T=298.15 K for each crystalline compound was estimated to be ρ=1.00 g cm^{-3}.

The compounds H_2salch, H_2acacch and H_2bzacch were studied by DSC over the temperature ranges 323–380, 323–400 and 323–430 K, respectively, and no phase transitions were found before the melting temperatures.

Combustion calorimetry

The combustion experiments were performed with a static bomb calorimeter [12, 13], with a twin valve bomb, Parr Instrument Company (PIC), type 1108 or 1105. For the study of H_2salch and H_2bzacch, the calorimeter (bomb PIC 1108) was calibrated with benzoic acid (Bureau of Analysed Samples, Thermochemical Standard, BCS-CRM-190 p), whose massic energy of combustion is –26431.8±3.7 J g^{-1}, under certificate conditions; from eight calibration experiments, ε(calor)=16009.5±1.1 J K^{-1}. For the study for H_2acacch the calorimeter (bomb PIC 1105) was also calibrated using the same sample of benzoic acid; from nine calibration experiments, ε(calor)= 15911.2±1.5 J K^{-1}. In both cases the values of ε(calor) was corrected for an average mass of water of 3119.6 g added to the calorimeter and the uncertainty quoted is the standard deviation of the mean.

For all experiments, samples in pellet form were ignited at T=298.150±0.001 K in oxygen, at a pressure p=3.04 MPa, with a volume of 1.00 cm^3 of water added to the bomb. Hexadecane (Aldrich Gold Label, mass fraction >0.99) was used as an auxiliary combustion on measurements of H_2acacch and H_2bzacch. The standard massic energies of combustion of two different hexadecane samples were measured: $-\Delta_c u^0$ (l)/J g^{-1}=47156.9±3.3 used with H_2acacch and $-\Delta_c u^0$ (l)/J g^{-1}=47151.4±3.1 used with H_2bzacch; both values are in agreement with the value of Fraser and Prosen [14], for a sample of mass fraction 0.9996, $-\Delta_c u^0$ (l)/J g^{-1}=47155.0±3.8. The electrical energy for ignition was determined from the change in potential difference across a capacitor when discharged through the platinum ignition wire. For the cotton thread fuse of empirical formula $CH_{1.686}O_{0.843}$, $-\Delta_c u^0$=16250 J g^{-1} [15], a value which has been confirmed in our laboratory. The corrections for nitric acid formation were based on –59.7 kJ mol^{-1}[16], for the molar energy of formation of 0.1 mol dm^{-3} HNO_3(aq) from N_2(g), O_2(g), and H_2O(l). The amount of substance used in each experiment was determined from the total mass of carbon dioxide produced, after allowance was made for that formed from the cotton thread fuse and hexadecane. An estimated pressure coefficient of massic energy: $(\partial u/\partial p)_T$= –0.2 J g^{-1} MPa^{-1}, at T=298.15 K, a typical value for most organic compounds, was assumed [17]. For each compound, the massic energy of combustion, $\Delta_c u^0$, was calculated by the procedure given by Hubbard *et al.* [18]. The relative atomic masses used throughout this paper were those recommended by the IUPAC Commission in 2001 [19].

Knudsen effusion technique

The standard molar enthalpies of sublimation of the three Schiff bases were determined by the Knudsen effusion method using the apparatus described by Burkinshaw and Mortimer [20], with the detailed modifications previously reported [21]. The performance of the equipment was confirmed measuring the standard molar enthalpies of sublimation of benzantrone, squaric acid and 4-hydroxy-2-methylquinoline, whose results [21] are in good agreement with the literature values.

The vapour effusing from the Knudsen cell was allowed to condense on a quartz crystal positioned above the effusion hole; changes in the frequency Δf of oscillation of the quartz crystal were proportional to the mass condensed in its surface [22], $\Delta f=C_f\Delta m$, where C_f is a proportionally constant. From the Knudsen Eq. (1), where $(\Delta m/\Delta t)$ is the rate of mass loss, a is the effective area of the effusion hole, and M is the molar mass of the effusing vapour:

$$p = (\Delta m/\Delta t)a^{-1}(2\pi RT/M)^{1/2} \qquad (1)$$

as the measured rate of change of frequency of oscillation with time, $\nu=\Delta f/\Delta t$, is directly proportional to the rate of sublimed mass of the crystalline sample [22], $\nu=C_f\Delta m/\Delta t$, the Eq. (1) leads to Eq. (2).

$$p = \nu T^{1/2}(2\pi R/M)^{1/2}/(aC_f) \qquad (2)$$

By applying the integrated form of the Clausius-Clapeyron equation, the enthalpy of sublimation, $\Delta_{cr}^{g}H_m^0$, may be derived from the slope of $\ln(\nu T^{1/2})$ *vs.* T^{-1}. From at least five independent sets of experimental measurements of the frequency of the quartz oscillator for each Schiff base, within convenient temperature intervals, the enthalpy of sublimation of each compound, referred to the mean temperature of the experimental range, was calculated.

Results

Combustion measurements

Results for a typical combustion experiment on each compound are given in Table 1; $\Delta m(H_2O)$ is the deviation of the mass of water added to the calorimeter from 3119.6 g, the mass assigned for ε(calor); ΔU_Σ is the correction to the standard state; the remaining terms are as previously described [18].

As samples were ignited at T=298.15 K, the energy associated to the isothermal bomb process is calculated by Eq. (3), where ΔT_{ad} is the calorimeter temperature change corrected for the heat exchange and the work of stirring.

$$\Delta U(\text{IBP}) = -\{\varepsilon_{cal} + \Delta m(H_2O)c_p(H_2O,\ l) + \varepsilon_f\}\Delta T_{ad} + \\ + \Delta U(\text{ign.}) \qquad (3)$$

The individual values of $\Delta_c u^0$, together with the mean and its standard deviation, are given in Table 2. Table 3 lists the derived standard molar enthalpies of combustion and of formation, in the condensed state. In accordance with normal thermochemical practice [23], 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 $\Delta_f H_m^0$(cr) from

Table 1 Typical combustion experiments for *N,N*'-bis(salicylaldehydo)cyclo-hexanediimine (H_2salch), *N,N*'-bis(acetylacetone)cyclohexanediimine (H_2acacch) and *N,N*'-bis(benzoylacetone)cyclohexanediimine (H_2bzacch), at T=298.15 K

	H_2salch	H_2acacch	H_2bzacch
$m(CO_2$, total)/g	1.94940	2.26908	1.86431
m(cpd)/g	0.71217	0.59206	0.48587
m(hexadecane)/g	–	0.24592	0.15358
m'(fuse)/g	0.00315	0.00421	0.00356
ΔT_{ad}/K	1.48475	1.99600	1.52448
$\varepsilon_f/(J\ K^{-1})$	15.95	17.69	16.30
$\Delta m(H_2O)$/g	0.0	0.1	0.1
$-\Delta U$(IBP)/J[a]	23792.60	31793.71	24430.46
$\Delta U(HNO_3)$/J	34.87	42.55	23.9
ΔU(ign.)/J	1.19	1.19	1.19
ΔU_Σ/J	14.28	13.24	11.44
ΔU(hexadecane)/J	–	11597.04	7241.51
ΔU(fuse)/J	51.16	68.37	57.81
$-\Delta_c u^0/J\ g^{-1}$	33267.74	33902.83	35185.95

[a] ΔU(IBP) already includes the ΔU(ign.)

Table 2 Individual values of the massic energy of combustion, $\Delta_c u^0$, of (H_2salch), (H_2acacch) and (H_2bzacch), at T=298.15 K

$-\Delta_c u^0$/J g^{-1}		
H_2salch	H_2acacch	H_2bzacch
33279.97	33914.53	35184.08
33254.82	33890.13	35176.56
33262.68	33902.83	35185.10
33267.74	33895.55	35175.94
33257.20	33907.72	35185.95
33275.53	33913.71	35184.00
33264.11		
$-<\Delta_c u^0>$/J g^{-1a}		
33266.0±3.5	33904.1±4.0	35181.9±1.8

[a] Mean value and standard deviation of the mean

$\Delta_c H^0_m$ the standard molar enthalpies of formation of H_2O(l): –285.83±0.04 kJ mol^{-1} and of CO_2(g): –393.51±0.13 kJ mol^{-1}, were used [24].

Sublimation measurements

For H_2acacch, eleven measurements of the change of frequency of the quartz oscillator at different temperatures were made, over the temperature range 341.7 to 356.7 K. Figure 2 shows that the plot of ln($\nu T^{1/2}$) *vs.* T^{-1}, for these measurements, yields a straight line, from the slope of which the standard molar enthalpy of sublimation, at the weighted mean temperature of the experimental range, T=349.2 K, is derived as $\Delta^g_{cr}H^0_m$ (T=349.2)=141.9±2.6 kJ mol^{-1}. Several series of measurements over the temperature intervals of 10–12 K in the temperature range 352.3 to 372.6 K and 379.4 to 415.4 K for H_2salch and H_2bzacch, respectively, yield the standard molar enthalpies of sublimation at the mean temperature, $<T>$, of each interval.

For the three compounds, the values of $\Delta^g_{cr}H^0_m$ ($<T>$) were corrected to T=298.15 K, by means of the Eq. (4), assuming $\Delta^g_{cr}C^0_{p,m}$ = –50 J K^{-1} mol^{-1}, a value previously estimated [20], yielding for $\Delta^g_{cr}H^0_m$, at T=298.15 K, 178.7±3.4 kJ mol^{-1} for H_2salch, 144.5±2.6 kJ mol^{-1} for H_2acacch and 157.8±3.3 kJ mol^{-1} for H_2bzacch, where the uncertainties assigned are twice the overall standard deviations of the mean.

Table 3 Derived standard (p^0=0.1 MPa) molar energies of combustion, $\Delta_c U^0_m$, standard molar enthalpies of combustion, $\Delta_c H^0_m$, and standard molar enthalpies of formation, $\Delta_f H^0_m$, for crystalline (H_2salch), (H_2acacch) and (H_2bzacch), at T=298.15 K

	$-\Delta_c U^0_m$(cr)/ kJ mol^{-1}	$-\Delta_c H^0_m$(cr)/ kJ mol^{-1}	$\Delta_f H^0_m$(cr)/ kJ mol^{-1}
H_2salch	10725.2±3.1	10733.9±3.1	–280.4±4.1
H_2acacch	9438.7±3.2	9449.9±3.2	–562.0±3.8
H_2bzacch	14162.0±3.1	14175.6±3.1	–343.1±4.6

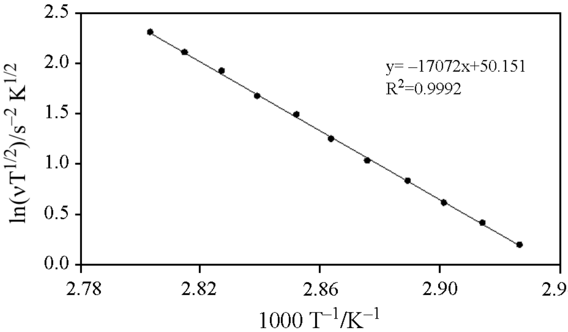

Fig. 2 Plot of ln($\nu T^{1/2}$) *vs.* T^{-1} for H_2acacch

$$\Delta^g_{cr}H^0_m(T = 298.15\text{ K}) = \Delta^g_{cr}H^0_m(<T>) + \\ + \Delta^g_{cr}C^0_{p,m}\ (298.15\text{ K} - <T>) \qquad (4)$$

For the three Schiff bases studied, the standard molar enthalpies of formation, in both crystalline and gaseous states, as well as the standard molar enthalpies of sublimation, at T=298 K, are summarised in Table 4.

Table 4 Derived standard (p^0=0.1 MPa) molar enthalpies of formation, $\Delta_f H^0_m$, and standard molar enthalipes of sublimation, $\Delta^g_{cr}H^0_m$, for (H_2salch), (H_2acacch) and (H_2bzacch), at T=298.15 K

	$\Delta_f H^0_m$(cr)/ kJ mol^{-1}	$\Delta^g_{cr}H^0_m$/ kJ mol^{-1}	$\Delta_f H^0_m$(g)/ kJ mol^{-1}
H_2salch	–280.4±4.1	178.7±3.4	–101.7±5.3
H_2acacch	–562.0±3.8	144.5±2.6	–417.5±4.6
H_2bzacch	–343.1±4.6	157.8±3.3	–185.3±5.7

Discussion

The present experimental work, reporting the thermochemical study of tetradentate N_2O_2 Schiff bases derived from condensation of β-diketones or salicylaldehyde with 1,2-cyclohexanediamine, represents another contribution to understand the energetical behaviour of that class of compounds. This work is a continuation of our previous studies, reporting thermochemical investigation of other compounds obtained from condensation of β-diketones (acetylacetone/benzoylacetone) with 1,2-ethanediamine (Schiff bases *N,N*'-bis(acetylacetone) ethylenediimine, H_2acacen, and *N,N*'-bis(benzoylacetone)ethylenediimine, H_2bzacen, [6]), or from condensation of salicylaldehyde with 1,2-ethanediamine/1,4-butanediamine (Schiff bases *N,N*'-bis-(salicylalde- hydo)ethylenediimine, H_2salen, and

2 salicylaldehyde (g) + en/buten/ch (g) → H_2salen/H_2salbuten H_2salch/(g)+2H_2O(g)

if diamine = 1,2-ethanediamine, $\Delta_1 H^0_m$(g) = –45.1±6.7 kJ mol^{-1}

if diamine = 1,4-butanediamine, $\Delta_2 H^0_m$(g) = –17.3±7.1 kJ mol^{-1}

if diamine = 1,2-cyclohexanediamine, $\Delta_3 H^0_m$(g) = –7.6±7.4 kJ mol^{-1}

Scheme 1 Hypothetical reaction of formation of Schiff bases derived from salicylaldehyde

2 acetylacetone (g) + en/ch(g) → H_2acacen/H_2acacch(g) + 2H_2O(g)

if diamine = 1,2-ethanediamine, $\Delta_4 H^0_m$(g) = –56.9±4.2 kJ mol^{-1}

if diamine = 1,2-cyclohexanediamine, $\Delta_5 H^0_m$(g) = –45.6±5.9 kJ mol^{-1}

2 benzoylacetone(g) + en/ch(g) → H_2bzacen/H_2bzacch(g) + 2H_2O(g)

if diamine = 1,2-ethanediamine, $\Delta_6 H^0_m$(g) = –82.3±9.3 kJ mol^{-1}

if diamine = 1,2-cyclohexanediamine, $\Delta_7 H^0_m$(g) = –94.0±8.7 kJ mol^{-1}

Scheme 2 Hypothetical reaction of formation of Schiff bases derived from β-diketones

N,N'-bis(salicylaldehydo)tetramethylenediamine, H_2salbuten [7]).

Considering the enthalpies of the hypothetical reactions of formation of H_2salch, H_2acacch and H_2bzacch, in the gaseous state, $\Delta_r H^0_m$(g), from salicylaldehyde, acetylacetone or benzoylacetone and 1,2-cyclohexanediamine (ch), it is possible to compare them with those of the formation of the Schiff bases from the correspondent aldehyde/β-diketones with aliphatic diamines (1,2-ethanediamine/1,4-butanediamine, en/buten). Schemes 1 and 2 present the equations for those hypothetical gaseous reactions as well as the respective values for the enthalpies of reaction.

For the calculation of the $\Delta_r H^0_m$(g) values in Scheme 1, the following enthalpies of formation have been used:

$\Delta_f H^0_m$(salicylaldehyde, g)= –245.5±2.2 [25]

$\Delta_f H^0_m$[$NH_2(CH_2)_2NH_2$, g]= –18.0±0.7 [26]

$\Delta_f H^0_m$[$NH_2(CH_2)_4NH_2$, g]= –57.9±2.3 [27]

$\Delta_f H^0_m$[$NH_2c(C_6H_{10})NH_2$, g]= –86.7±2.7 [27]

and $\Delta_f H^0_m$(H_2O, g)= –241.82±0.04 kJ mol^{-1} [24].

Although the enthalpies of formation of gaseous 1,4-butanediamine and 1,2-cyclohexanediamine are estimated values, the recent experimental determination [25] of the enthalpy of formation of gaseous salicylaldehyde, implies the recalculation of $\Delta_1 H^0_m$(g) and $\Delta_2 H^0_m$(g) previously reported [7].

Previously, it has been observed an increase of the enthalpy of reaction with the number of methylene groups in the diimine bridge of the Schiff base [7]. However, the results reported in the present work for the formation of H_2salch, $\Delta_3 H^0_m$(g), points to a relative increase, although there are only two carbon atoms in the bridge. The comparison of the values of the enthalpies of reaction, obtained when the aliphatic diamine is substituted by a cyclic diamine, evidences a higher destabilization for H_2salch, and consequently the less favourable formation of the compound, what can be explained by the strain of the bridge as a part of a ring.

For the calculation of the $\Delta_r H^0_m$(g) values in Scheme 2, the following values of enthalpies of formation have been used: $\Delta_f H^0_m$(acetylacetone, g)= –384.4±1.3 kJ mol^{-1} [28], $\Delta_f H^0_m$(benzoylacetone, g)= –244.1±2.9 kJ mol^{-1} [29]. The greater delocalization and internal hydrogen bonding in the benzoylacetone derivatives, H_2bzacen and H_2bzacch, comparatively with the acetylacetone derivatives, H_2acacen and H_2acacch, justifies the relative magnitude between $\Delta_4 H^0_m$(g) or $\Delta_5 H^0_m$(g) and $\Delta_6 H^0_m$(g) or $\Delta_7 H^0_m$(g). The substitution of the aliphatic diamine by the cyclic diamine does evidence a very small destabilization for the acetylacetone derivative, although that effect is not observed for H_2bzacch comparatively with H_2bzacen, probably because it will hindered due to the stronger delocalization on the benzoylacetone derivative comparatively with the acetylacetone one.

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