STUDIES ON NITROPHENOLS. PART IV. THE STANDARD ENTHALPIES OF COMBUSTION AND FORMATION OF 1,2-DIHYDROXYBENZENE, 1,2- AND 1,4-NITROPHENOL *

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ABSTRACT

Using a static oxygen-bomb calorimeter, the standard enthalpies of combustion at 298.15 K of 1,2-nitrophenol, the yellow dimorph of 1,4-nitrophenol and of 1,2-dihydroxybenzene were determined as -2873.27 ± 0.62, -2863.21 ± 0.54 and -2865.49 ± 0.74 kJ mole^{-1}, respectively. The corresponding standard enthalpies of formation were -202.4 ± 1.0, -212.4 ± 1.0 and -353.1 ± 1.1 kJ mole^{-1}. The enthalpy of transition of the white to yellow dimorph of 1,4-nitrophenol was determined as 0.07 ± 0.08 kJ mole^{-1}. The lower limit for the strengths of the intramolecular H-bonds in 1,2-nitrophenol and 1,2-dihydroxybenzene was estimated as ~ 10 kJ mole^{-1}.

INTRODUCTION

Although the isomeric nitrophenols are standard textbook examples illustrating inter and intramolecular H-bonds, there are no reliable thermodynamic data for these isomers from which to estimate the strength of the intramolecular H-bond in 1,2-nitrophenol. For the isomeric dihydroxybenzenes, reliable thermodynamic data exist [1] for the 1,4 isomer but not for catechol. This paper describes the determination of the enthalpies of combustion of 1,2-dihydroxybenzene, 1,2- and 1,4-nitrophenol and the estimation of the strengths of the intramolecular H-bond in the 1,2 isomers.

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EXPERIMENTAL

Materials

1,2-Nitrophenol (sample from Propellants, Explosives and Rocket Motor Establishment, Woolwich East, London, 60 g) was dissolved in 60/80 petroleum ether (200 cm³) at 70°C, filtered hot, and the product from the filtrate recrystallised four times from 60/80 petroleum ether. The product was stored in vacuo (m.p. 44.7–45.7°C, m.p. [2] 44.9°C). The purity of two batches, A and B, using differential scanning calorimetry (DSC) was 99.9% and the water content by Karl Fischer titration was 0.11 ± 0.04%.

1,4-Nitrophenol is dimorphic [2,3] at room temperature, a stable yellow form coexisting with a metastable white form. The transition temperature is approximately 50°C and the stable yellow form is photosensitive at room temperature, turning red on exposure to light. The crystal structures of both forms are known [4,5] and the phase transformation white → yellow and the photochemical conversion yellow → red have been studied [6] by spectroscopic [7–9] and thermal methods [10]. 1,4-Nitrophenol (B.D.H.) was dissolved in chloroform at 60°C, and recrystallised four times to yield a mixture of the yellow and white forms. This mixture was dissolved in diethylether at room temperature and slow evaporation of the solvent in the dark yielded well-formed crystals of the yellow dimorph. The white form was obtained by dissolving the yellow/white mixture (34 g), obtained from chloroform as described earlier, in distilled water (600 cm³) at 60°C. The solution crystallised to yield a bed of ill-formed crystals over an amorphous mass, but a substantial yield of white needle-like crystals formed above this. These were separated and stored in vacuo. The purity of the yellow form, using DSC, was 99.9% and the water content by Karl Fischer titration was 0.10 ± 0.05%.

1,2-Dihydroxybenzene (B.D.H., 50 g) was dissolved in benzene (300 cm³), crystallised and then recrystallised (four times from benzene, twice from toluene). The purity by DSC was 99.88% (m.p. 104.4°C, m.p. [2] 105°C). The water content by Karl Fischer titration was 0.11%.

1,1,2,2-Tetrachloroethane (B.D.H.), used for solution calorimetry, was dried by passing it down a 3 × 80 cm column containing 4 Å activated molecular sieves.

Apparatus

The combustion calorimeter was a commercial instrument (Gallenkamp Automatic Bomb calorimeter, Model CB-110) and modified as described elsewhere [11]. The system was calibrated using benzoic acid (B.C.S.: No. 190j Thermochemical standard) for which $-\Delta u_c = 26435 \pm 5$ J g^{-1} (standard bomb conditions). The mean energy equivalent for nine calibration runs was $\epsilon_{sf} = 8.6164 \pm 0.0013$ kJ K^{-1}, in satisfactory agreement with a

previous figure [11] $(8.6186 \pm 0.0026$ kJ $K^{-1})$ for this apparatus. The notation of Coops et al. [12] is used throughout and uncertainty intervals are quoted as $\pm$ two standard deviations of the mean. For the nitrophenol combustion experiments the bomb was initially charged with 10.8 cm^3 of water as recommended by Johnson [13]. This gives a final concentration of nitric acid (determined by titration to a methyl orange end point) in the bomb cylinder of approximately 0.1 M, for which a correction calculated from $\Delta U_f^0[HNO_3, 500 \ H_2O] = -59.72$ kJ mole^{-1} was applied. Some samples (see Results and Discussion) were encapsulated in polythene prior to combustion. The polythene was from the same batch as was used in previous studies [14] and its energy of combustion determined as -46.379 kJ g^{-1} (two experiments), a value in good agreement with a literature value [15] for this material $(-46394 \pm 14$ J g$^{-1})$. The Washburn reduction (q_w) and the corrected temperature rise $(\Delta\theta)$ for the near-adiabatic system were computed as described before [11].

The solution calorimeter was an LKB Precision Calorimetry System 8700-1 with the out-of-balance bridge signal displayed on a chart recorder. The calorimeter was tested by measuring the enthalpy of neutralization of tris[(hydroxymethyl)amino]methane (THAM) in an excess of 0.1 M HCl for which $\Delta H(298.15$ K, $5030 < n < 5700) = 29.74 \pm 0.05$ kJ mole^{-1} where n is the mole ratio of water to THAM; Prosen and Kilday [16] obtained $\Delta H(298.15$ K, $n = 1345) = 29.77 \pm 0.03$ kJ mole^{-1}. The enthalpy of dilution of THAM is negligible [17].

The scanning calorimeter was a Perkin Elmer DSC2 and purities were determined using the method of Plato and Glasgow [18].

RESULTS AND DISCUSSION

Repeated attempts with the differential scanning calorimeter operating between 283 and 373 K at high sensitivity and varying scan speeds failed to detect the yellow $\rightarrow$ white transition for 1,4-nitrophenol reported [6] at about 323 K. A similar conclusion was noted in an earlier DTA study [10].

The enthalpies of solution in 1,1,2,2-tetrachloroethane of both forms were determined at 298.15 K giving approximately the same final concentration for each run. Assuming that the final thermodynamic state (the solution phase) is identical for each dimorph, then the enthalpy of transition will be given by the difference in the enthalpies of solution; the results are collected in Table 1.

The volatility of the title compounds under combustion conditions was investigated before combustion work was undertaken. Pelleted samples of known mass were equilibrated overnight in the bomb containing 30 atm O_2 and at room temperature. Only for 1,2-nitrophenol was there a detectable mass change, the change in mass for the other materials being < 10 μg. The

TABLE 1

Enthalpies of solution at 298.15 K of 1,4-nitrophenol (yellow) and 1,4-nitrophenol (white) in 1,1,2,2-tetrachloroethane
N is the mole ratio of solvent to nitrophenol.

1,4-$NO_2C_6H_4OH$(c, white)		1,4-$NO_2C_6H_4OH$(c, yellow)	
N	ΔH(solution) (kJ mole^{-1})	N	ΔH(solution) (kJ mole^{-1})
1505.5	21.76	1573.6	21.82
1541.3	21.81	1529.7	21.98
1539.7	21.93	1546.6	21.96
1495.8	21.93	1568.7	21.86
1585.3	21.88	1497.7	21.97
1552.2	21.74	1551.9	21.88
1618.7	21.83	1592.8	21.94
	$\langle \Delta H \rangle = 21.84 \pm 0.06$		$\langle \Delta H \rangle = 21.91 \pm 0.05$

ΔH(transition, white $\rightarrow$ yellow) $= 0.07 \pm 0.08$ kJ mole^{-1}

mass loss for 1,2-nitrophenol over 60 min (a typical equilibration time) was ~ 0.34 mg (about 0.03%). Examination of the bomb water by UV spectroscopy revealed 0.033 mg of dissolved 1,2-nitrophenol (2.2×10^{-5} M). The bomb washings after combustion also indicated the presence of an aromatic

TABLE 2

Results for the combustion calorimetry of 1,2-nitrophenol (cryst. B) at 298.15 K

	10	11	12	13	14	15
m, sample [a] (g)	1.389778	1.386469	1.403594	1.381168	1.35533	1.077482
m, fuse (g)	0.00314	0.00312	0.00302	0.00307	0.00284	0.00280
m, Pt wire (g)	0.00758	0.00654	0.00820	0.00680	0.00738	0.00710
m, crucible [b] (g)	5.65787	5.65778	3.33842	3.33808	3.33823	3.33806
m, soot (g)	0.00031	0.00028	0	0	0	0.00106
m, H_2O (g)	10.8	10.8	10.8	10.8	10.8	10.8
q_i(kJ)	0.05675	0.05640	0.05465	0.05552	0.05150	0.05080
q_n(kJ)	0.075844	0.077636	0.075247	0.075247	0.063303	0.061512
q_c(kJ)	0.01023	0.00924	0	0	0	0.03498
q_w(kJ)	0.045448	0.045308	0.045948	0.045149	0.036653	0.034661
$\Delta\theta$(K)	3.3328	3.3216	3.3662	3.3109	2.7235	2.5824
ϵ_{cf}(kJ K^{-1})	0.061373	0.061367	0.059635	0.059594	0.059151	0.059046
$-\Delta u_c^0$(298.15 K)(kJ g^{-1})	20.6892	20.6669	20.6822	20.6705	20.6747	20.6885

$\langle -\Delta u_c^0, 298.15 \rangle = 20.6787 \pm 0.0082$ kJ g^{-1} [c]

[a] Corrected for buoyancy ($\rho = 1.315$ g cm^{-3}) and for water content (0.11 mass%), see Experimental section.

[b] Fused silica.

[c] Includes error in ϵ_{sf}(benzoic acid).

TABLE 3

Results for the combustion calorimetry of 1,2-nitrophenol (cryst. C) at 298.15 K

	16	17	18	19	20	21	22	23
m, sample [a] (g)	0.932501	0.891260	0.938692	1.099484	1.109106	1.187497	1.136654	1.170232
m, $-CH_2-$ [b] (g)	0.101136	0.095480	0.116724	0.121690	0.114752	0.112569	0.126686	0.129459
m, fuse (g)	0.00271	0.00253	0.00322	0.00285	0.00285	0.00235	0.00279	0.00230
m, Pt wire (g)	0.00700	0.00726	0.00708	0.00764	0.00706	0.00614	0.00681	0.00698
m, crucible [c] (g)	3.33678	4.07460	3.33667	4.07466	3.33664	3.33667	4.07444	3.33678
m, soot (g)	0	0	0	0	0	0	0	0
m, H_2O (g)	10.8	10.8	10.8	10.8	10.8	10.8	10.8	10.8
q_i(kJ)	0.049228	0.046080	0.058148	0.051676	0.051676	0.042932	0.050627	0.042057
q_n(kJ)	0.056630	0.054942	0.060317	0.065871	0.067484	0.071067	0.069275	0.068678
q_c(kJ)	0	0	0	0	0	0	0	0
q_w(kJ)	0.019658	0.033805	0.036210	0.041870	0.041896	0.044484	0.043326	0.044620
$\Delta\theta$(K)	2.7894	2.6491	2.8782	3.2900	3.2746	3.4499	3.4032	3.4985
ϵ_{cf}(kJ K^{-1})	0.018882	0.059959	0.059604	0.060516	0.059939	0.060086	0.060617	0.060139
$-\Delta u_c^0$(298.15 K) (kJ g^{-1})	20.6645	20.6674	20.6684	20.6842	20.6713	20.6754	20.6650	20.6740

$\langle(-\Delta u_c^0(298.15 \text{ K}))\rangle = 20.6713 \pm 0.0062 \text{ kJ g}^{-1}$ [d]

[a] Corrected for buoyancy ($\rho = 1.315$ g cm^{-3}) and for water content (0.065%).

[b] Corrected for buoyancy ($\rho = 0.92$ g cm^{-3}).

[c] Fused silica.

[d] Includes error in ϵ_{sf} and $\Delta u_c^0(-CH_2-)$.

TABLE 4

Results for the combustion calorimetry of 1,4-nitrophenol (cryst., yellow dimorph) at 298.15 K

	1	2	3	4	5	6	7
m, sample [a] (g)	1.391578	1.391418	1.435944	1.374323	1.410242	1.424178	1.403334
m, fuse (g)	0.00328	0.00370	0.00298	0.00362	0.00307	0.00308	0.00290
m, Pt wire (g)	0.00802	0.00706	0.00846	0.00750	0.00756	0.00784	0.00696
m, crucible [b] (g)	5.65793	5.65795	5.65797	5.65795	5.65791	5.65794	5.65791
m, soot (g)	0	0	0	0	0	0	0
m, H_2O (g)	10.8	10.8	10.8	10.8	10.8	10.8	10.8
q_i (kJ)	0.059197	0.066543	0.053950	0.065144	0.055524	0.055699	0.052251
q_n (kJ)	0.074053	0.076442	0.078233	0.074650	0.075844	0.079428	0.075844
q_c (kJ)	0	0	0	0	0	0	0
q_w (kJ)	0.045576	0.045540	0.047108	0.044954	0.046219	0.046672	0.045973
$\Delta\theta$ (K)	3.3232	3.3252	3.4300	3.2836	3.3688	3.4016	3.3513
ϵ_{cf} (kJ K^{-1})	0.061376	0.061376	0.061457	0.061345	0.061410	0.061435	0.061397
$-\Delta u_c^0$(298.15 K)(kJ g^{-1})	20.5947	20.6024	20.6037	20.5982	20.6037	20.5991	20.5994

$\langle -\Delta u_c^0,\ 298.15\ \mathrm{K} \rangle = 20.6002 \pm 0.0039$ kJ g^{-1} [c].

[a] Corrected for buoyancy ($\rho = 1.479$ g cm^{-3}) and for water content (0.096 mass%), see Experimental section.

[b] Fused silica.

[c] Includes error in ϵ_{sf}(benzoic acid).

TABLE 5

Results for the combustion calorimetry of 1,2-dihydroxybenzene (cryst.) at 298.15 K

	1	2	3	4	5	6	7	8
m, sample [a] (g)	1.017683	0.989172	1.042736	1.014864	1.018383	1.003568	1.010036	0.986463
m, fuse (g)	0.00286	0.00398	0.00253	0.00282	0.00326	0.00302	0.00233	0.00263
m, Pt wire (g)	0.00762	0.00656	0.00864	0.00778	0.00804	0.00734	0.00703	0.00661
m, crucible [b] (g)	3.337837	5.657810	3.337847	3.337937	3.337937	3.337797	5.658110	3.337737
m, soot (g)	0.00017	0.00051	0.00018	0.00048	0.00009	0.00020	0.00032	0.00006
m, H_2O (g)	1.08	1.08	1.08	1.08	1.08	1.08	1.08	1.08
q_i(kJ)	0.051851	0.071440	0.046080	0.051152	0.058847	0.054650	0.042582	0.047829
q_n(kJ)	0.004778	0.004180	0.005375	0.005375	0.005375	0.004778	0.004778	0.004778
q_c(kJ)	0.00561	0.01683	0.00594	0.01584	0.00297	0.00660	0.01056	0.00198
q_w(kJ)	0.019608	0.018992	0.020151	0.019537	0.019614	0.019298	0.01944	0.018924
$\Delta\theta$(K)	3.0748	2.9881	3.1482	3.0653	3.0760	3.0317	3.0482	2.9784
ϵ_{cf}(kJ K^{-1})	0.018899	0.020594	0.018959	0.018892	0.018901	0.018865	0.020644	0.018824
$-\Delta u_c^0$(298.15 K) (kJ g^{-1})	26.0210$_5$	26.0121	26.0087	26.0227	26.0033	26.0146	26.0101$_5$	26.0016

$\langle -\Delta u_c^0, 298.15 \rangle = 26.0018 \pm 0.0067$ kJ g^{-1} [c].

[a] Corrected for buoyancy ($\rho = 1.604$ g cm^{-3}) and for water content (0.012%), see Experimental section.

[b] Fused silica.

[c] Includes error in ϵ_{sf} (benzoic acid).

339

material, not positively identified, at about the same concentration. If the 1,2-nitrophenol in the vapour burns at the time of firing then the thermal correction for the vapourized material is negligible; if it does not, it is significant. Initial experiments with 1,2-nitrophenol gave conflicting results. Sample A gave $\langle - \Delta u_c^o, 298.15 \text{ K} \rangle = 20.8629 \pm 0.0088$ kJ g^{-1} ($n = 9$, where n is the number of experiments). Subsequent experiments with another batch of the same material (sample B) gave 20.6787 ± 0.0082 kJ g^{-1} ($n = 6$). At this stage, the calibration with benzoic acid and combustion of 1,4-nitrophenol were repeated; both experiments gave results within the error limits of earlier experiments. In view of the difference between samples A and B, and the volatility of the material, a new sample (sample C) from a different source (B.D.H.) was purified as before (99.95% by DSC; 0.065% H$_2$O by Karl Fischer) and combusted with each aliquot encapsulated in polythene; $\langle - \Delta u_c^o, 298.15 \text{ K} \rangle$, sample C $= 20.6713 \pm 0.0062$ kJ g^{-1} ($n = 8$), a value not significantly different from that for sample B. The uncertainty intervals quoted include the uncertainty in ϵ_{sf} and, for sample C, the uncertainty in Δu_c^o (polythene). The combustion results for samples B and C are collected in Tables 2 and 3; a mean value was used to calculate ΔH_f^0. The difference in the energies of combustion of samples A and B is due to an unidentified systematic error. Corresponding results for 1,4-nitrophenol and 1,2-dihydroxybenzene are given in Tables 4 and 5, respectively.

Combining the heats of combustion with enthalpies of formation for CO$_2$(g) (-393.51 ± 0.13 kJ mole^{-1}) [19] and H$_2$O(l) (-285.830 ± 0.042 kJ mole^{-1}) [19], we obtain enthalpies of formation for the title compounds. The enthalpies of sublimation of these compounds have been determined [20] by a torsion-effusion method and enthalpies of formation in the gas phase may be calculated; these are collected in Table 6 with a literature value [1] for ΔH_f^0 [1,4-dihydroxybenzene (g)].

The only literature reference to the combustion thermochemistry of the nitrophenols is contained in an early review by Kharasch [22] who quotes three independent determinations of the enthalpies of combustion for each nitrophenol. A mean of these values yields -195 ± 2 kJ mole^{-1} and -198 ± 8 kJ mole^{-1} for the enthalpies of formation of the crystalline 1,2- and 1,4-nitrophenols, respectively. These values differ markedly from those in Table 6.

A reliable value for the enthalpy of formation of 1,4-dihydroxybenzene is quoted by Cox and Pilcher [1] (see Table 6). Zwolinski and co-workers [23] give a corresponding figure for the 1,2 isomer (-353.13 kJ mole^{-1} for the solid) which derives from nineteenth century studies by Stohman and Langbein [24]; the agreement between this figure and our work is fortuitous.

In the absence of specific electronic and steric effects, the gas phase enthalpies of formation of the 1,2, the 1,3 and the 1,4 isomers of any disubstituted benzene will be identical. Experimentally observed differences may thus be ascribed to specific electronic and steric effects. The foregoing

TABLE 6

Enthalpies of combustion and formation at 298.15 K for nitrophenols and dihydroxybenzenes

Compounds	$-\Delta H_c^0$ (kJ mole^{-1})	$-\Delta H_f^0(c)$ (kJ mole^{-1})	$\Delta H^0(c \rightarrow g)$ [b] (kJ mole^{-1})	$-\Delta H_f^0(g)$ (kJ mole^{-1})
1,2-Nitrophenol	2873.27 ± 0.62	202.4 ± 1.0	73.6 ± 1.3	128.8 ± 1.6
1,4-Nitrophenol [a]	2863.21 ± 0.54	212.4 ± 1.0	94.7 ± 1.7	117.7 ± 2.0
1,2-Dihydroxybenzene	2865.49 ± 0.74	353.1 ± 1.1	81.5 ± 1.7	271.6 ± 2.0
1,4-Dihydroxybenzene				261.7 ± 2.1

[a] Yellow dimorph
[b] These values corrected to 298 K using Watson's [21] equation.

results are interpreted assuming (i) that the resonance stabilization energy of the aromatic nucleus is the same in the 1,2 and 1,4 isomers and (ii) that there is no steric interaction between substituents in the 1 and 4 positions. Hence, the differences in the experimental $\Delta H_f^0(g)$ are rationalized in terms of special electronic and steric interactions between substituents in the 1 and 2 positions. The dominant effect in the title compounds is likely to be a stabilizing effect due to the formation of an intramolecular H-bond; this effect is offset by a destabilizing steric interaction, a destabilizing steric inhibition of resonance plus a longer range dipole–dipole interaction arising from the adjacent polar substituents. Thus, it is probably not correct to identify the differences in $\Delta H_f^0(g)$ of the isomers with the strength of the

TABLE 7

Differences (Δ) between the enthalpies of formation of gaseous 1,2 and 1,4 disubstituted benzenes at 298.15 K

Substituent		Δ [a] (kJ mole^{-1})
X	Y	
NO_2	OH	-11.1 ± 2.6
OH	OH	-9.9 ± 2.9
CH_3	OH	-3.3 ± 2.1
COOH	NH_2	-2.3 ± 4.4
C_2H_5	OH	-1.1 ± 2.2
CH_3	CH_3	0.0 ± 1.0
CH_3	C_2H_5	4.5 ± 1.6
Cl	Cl	7.7 ± 2.4
CH_3	COOH	12.0 ± 1.4
F	F	12.8 ± 1.5
Cl	COOH	16.0 ± 4.8
OCH_3	COOH	18.1 ± 1.8
C_2H_5	COOH	22.6 ± 2.0

[a] The first two entries are results from this work, the remainder from ref. 25.

intramolecular H-bond.

$$\Delta = \Delta H_f^0[1,2\text{-}C_6H_4XY(g)] - \Delta H_f^0[1,4\text{-}C_6H_4XY(g)]$$

Rather, this difference $(-\Delta)$ of ~ 10 kJ mole^{-1} for the title compounds represents a lower limit for the intramolecular H-bond strength. Table 7 compares the function, Δ, for the title compounds with corresponding values [25] for other disubstituted benzenes.

ACKNOWLEDGEMENTS

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