Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Triethylammonium 3,5-dinitrobenzoate 1,4-dihydroxybenzene hemisolvate

Jian-Wen Tian* and Yan-Yan Liu

Experimental Chemistry Center, Nanchang University, Nanchang 330031, People's Republic of China

Correspondence e-mail: wen820@sohu.com

Received 5 January 2007
Accepted 16 January 2007

Key indicators

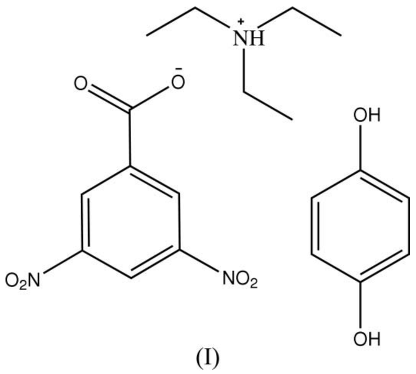
Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.005 Å
R factor = 0.064
wR factor = 0.227
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_6H_{16}N^+\cdot C_7H_3N_2O_6^-\cdot 0.5C_6H_6O_2$, the asymmetric unit contains one cation, one anion and one-half of the neutral molecule. The dihydroxybenzene molecule lies on a centre of inversion. Intermolecular O—H···O and N—H···O hydrogen bonds are effective in the stabilization of the crystal structure.

Comment

We planned to synthesize *p*-phenylenebis(3,5-dinitrobenzoate), but by accident, we obtained the title compound, (I). The crystal structure determination of (I) has been carried out in order to elucidate the molecular conformation. We report here the crystal structure of (I).



(I)

The asymmetric unit of (I) contains one $C_6H_{16}N^+$ cation, one $C_7H_3N_2^-$ anion and one-half of the neutral $C_6H_6O_2$ molecule (Fig. 1). The dihydroxybenzene molecule lies on a centre of inversion. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

The C1–C6 and C8–C10/C8*A*–C10*A* rings are, of course, planar and the dihedral angle between them is 85.26 (6)°.

As can be seen from the packing diagram (Fig. 2), intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) link the components, and may be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

3,5-Dinitrobenzoyl chloride (0.5 g, 2.2 mmol) and 1,4-dihydroxybenzene (0.12 g, 1.1 mmol) were mixed in a flask; triethylamine (3 ml) was poured into the mixture and it was stirred for 6 h. The solution was evaporated to give a brown solid, which was then dissolved in acetone/petroleum ether (1:1). Single crystals suitable for X-ray analysis were obtained by evaporation of the solvent over several days (Emen *et al.*, 2006; Wardell *et al.*, 2006) (yield 0.41 g, 46%).

Crystal data

$C_6H_{16}N^+ \cdot C_7H_3N_2O_6^- \cdot 0.5C_6H_6O_2$
$M_r = 368.37$
Monoclinic, $P2_1/c$
$a = 10.8033\ (13)$ Å
$b = 13.7485\ (16)$ Å
$c = 13.3250\ (16)$ Å
$\beta = 108.863\ (2)^\circ$
$V = 1872.9\ (4)$ Å^3
$Z = 4$
$D_x = 1.306$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 291\ (2)$ K
Block, white
$0.28 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.972$, $T_{\max} = 0.980$
16350 measured reflections
4291 independent reflections
2170 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.227$
$S = 1.05$
4291 reflections
239 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1033P)^2 + 0.4311P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7···O2$^{\rm i}$	0.82	1.90	2.717 (3)	174
N3—H3N···O1$^{\rm i}$	0.91	1.76	2.668 (3)	175

Symmetry code: (i) $-x + 1, -y + 2, -z + 1$.

H atoms were positioned geometrically, with O—H = 0.82 Å, N—H = 0.91 Å and C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = xU_{\rm eq}$(C,N,O), where $x = 1.2$ for NH, aromatic and methylene H, and $x = 1.5$ for all other H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Thanks are expressed to Dr Hui He for her help with this work.

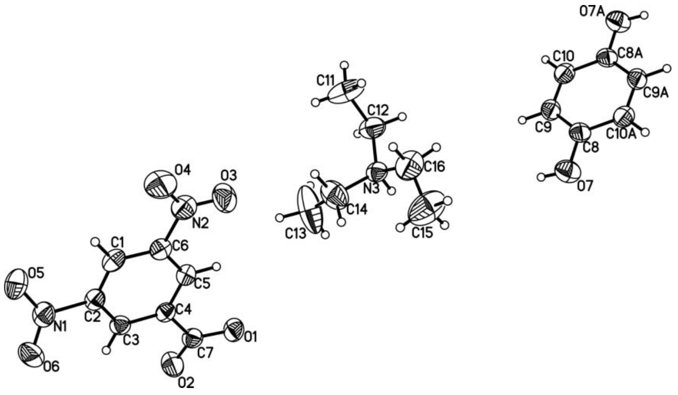


Figure 1
The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A) $2 - x, 2 - y, -z$.]

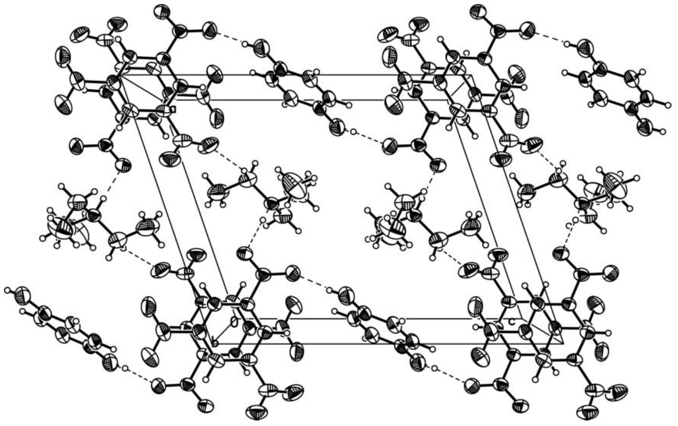


Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker, (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Emen, F. M., Flörke, U., Külcü, N. & Arslan, H. (2006). *Acta Cryst.* E**62**, o3814–o3815.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wardell, S. M. S. V., Souza, M. V. N. de, Wardell, J. L., Low, J. N. & Glidewell, C. (2006). *Acta Cryst.* E**62**, o2589–o2591.