

Sankar Muniappan and Israel  
Goldberg\*School of Chemistry, Sackler Faculty of Exact  
Sciences, Tel-Aviv University, Ramat-Aviv,  
69978 Tel-Aviv, Israel

Correspondence e-mail: goldberg@post.tau.ac.il

## Key indicators

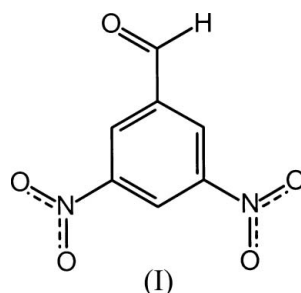
Single-crystal X-ray study  
 $T = 110$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.043  
 $wR$  factor = 0.128  
Data-to-parameter ratio = 16.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.On C—H···O interactions in 3,5-dinitro-  
benzaldehyde

The structure of the title compound,  $\text{C}_7\text{H}_4\text{N}_2\text{O}_5$ , was determined from low-temperature data (at about 110 K). It reveals a stacked-layered organization of the molecules with C—H···O interactions within the layers.

Received 15 November 2006  
Accepted 22 November 2006

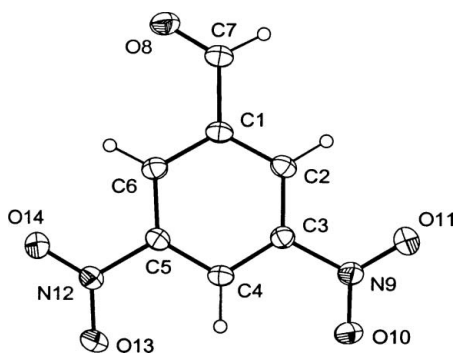
## Comment

The title compound, (I), is an important reagent in the synthesis (by its condensation with pyrrole) of tetrakis(3,5-dinitrophenyl)porphyrin. The latter is a precursor in the preparation of the corresponding porphyrin octacarboxylic acid, an attractive building block for the supramolecular self-assembly of large porphyrin-based arrays (Goldberg, 2005, and references therein). As (I) has not been characterized before by X-ray structure analysis, we report here its structure determined at *ca* 110 K with a resolution of 0.70 Å. The good-quality data allowed refinement of the H-atom parameters, and provide a reliable description of the intermolecular C—H···O contacts. The molecular structure of (I) (Fig. 1) reveals a planar benzaldehyde fragment with small deviations of the nitro groups from the aromatic plane; the dihedral angle between C1/C6 and N9/O11 is 16.13 (6)°, whereas that between C1/C6 and N12/O14 is 3.51 (5)°. In (I), the planar molecular fragments are arranged in layers connected by C—H···O interactions (Fig. 2 and Table 1; CSD, Version 5.27, August 2006 update; Allen, 2002; Desiraju & Steiner, 1999). Each molecule is involved in eight intra-layer interactions (graph-set representation  $R_2^2(8)$ ; Bernstein, *et al.*, 1995) with neighbouring molecules. The corrugated arrays are aligned roughly perpendicular to the *b* axis (Fig. 3).



## Experimental

3,5-Dinitrobenzyl alcohol (Aldrich) was oxidized to 3,5-dinitrobenzaldehyde using quinolinium chlorochromate by the method of Bhyrappa *et al.* (1998). 3,5-Dinitrobenzaldehyde (10 mg, 0.05 mmol) was dissolved in  $\text{CH}_2\text{Cl}_2$  (1 ml) and allowed to stand for slow evaporation. X-ray quality crystals were obtained after 5 d.

**Figure 1**

The molecular structure of (I), showing the atom-labelling scheme. Ellipsoids represent displacement parameters at the 50% probability level at *ca* 110 K.

**Crystal data**

$C_7H_4N_2O_5$

$M_r = 196.12$

Monoclinic,  $P2_1/n$

$a = 8.2791$  (2) Å

$b = 6.20910$  (10) Å

$c = 14.9903$  (4) Å

$\beta = 93.5895$  (11)°

$V = 769.08$  (3) Å<sup>3</sup>

$Z = 4$

$D_x = 1.694$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\mu = 0.15$  mm<sup>-1</sup>

$T = 110$  (2) K

Prism, colourless

$0.35 \times 0.30 \times 0.20$  mm

**Data collection**

Nonius KappaCCD diffractometer

$\varphi$  and  $\omega$  scans

Absorption correction: none

6627 measured reflections

2321 independent reflections

1793 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.027$

$\theta_{max} = 30.5^\circ$

**Refinement**

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.127$

$S = 1.06$

2321 reflections

143 parameters

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0766P)^2 + 0.0648P]$  where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.028$

$\Delta\rho_{max} = 0.30$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.34$  e Å<sup>-3</sup>

**Table 1**

Geometry of the C—H...O interactions in (I) (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C2—H2...O13 <sup>i</sup>	0.958 (19)	2.573 (19)	3.5027 (14)	163.7 (15)
C4—H4...O8 <sup>ii</sup>	0.987 (15)	2.381 (15)	3.3382 (14)	163.1 (12)
C7—H7...O10 <sup>iii</sup>	0.967 (17)	2.523 (17)	3.1338 (14)	121.1 (12)
C7—H7...O14 <sup>i</sup>	0.967 (17)	2.621 (17)	3.5340 (15)	157.7 (12)

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$ ; (iii)  $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$ .

Initially, the four H atoms were placed in calculated positions and were constrained to ride on their parent atoms. In the final stages of the least-squares refinement, the coordinates and the displacement parameters of all the H atoms were refined freely without any restraints or constraints.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996) and

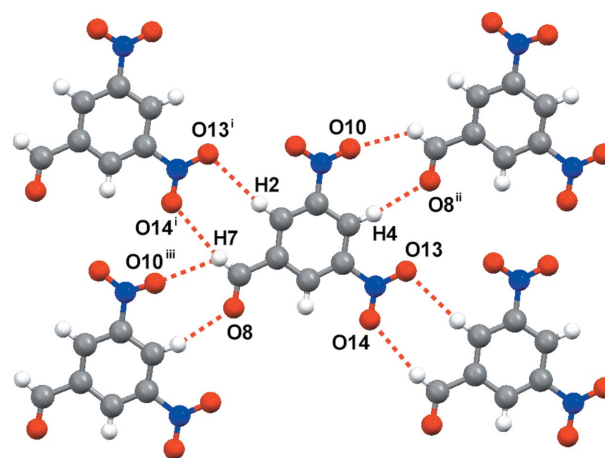
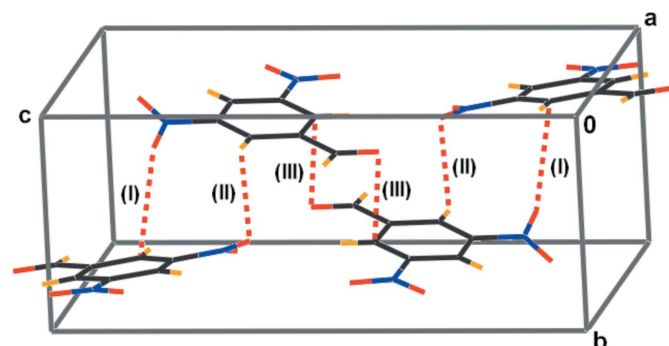
**Figure 2**

Illustration of the weak intermolecular hydrogen bonds within the layers. As a result of the slightly different alignment of adjacent units, these arrays have corrugated surfaces. The C—H...O interactions are indicated by red dotted lines (Table 1). [Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (iii)  $-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$ .]

**Figure 3**

The crystal packing of (I); two pairs of molecules of two adjacent layers are shown. Relatively short contacts observed between molecules in different layers are indicated by dotted red lines. They correspond to the following distances and their symmetry and translation equivalents: (I) H2...O10( $-\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ) 2.648 (2) Å; (II) O11...C2( $-\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ) 3.110 (2) Å; (III) C6...O8( $-x, -y, -z$ ) 3.172 (2) Å.

*Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

This research was supported in part by the Israel Science Foundation (grant No. 254/04).

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