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## 4-(2-Methoxy-4-nitrophenyl)morpholine-3,5-dione

M. Delower H. Bhuiyan,<sup>a</sup> Paul Jensen,<sup>b</sup> Peter Turner<sup>b</sup> and Andrew C. Try<sup>a\*</sup>

<sup>a</sup>Department of Chemistry and Biomolecular Sciences, Building F7B, Macquarie University, NSW 2109, Australia, and <sup>b</sup>Crystal Structure Analysis Facility, School of Chemistry, F11, The University of Sydney, NSW 2006, Australia

Correspondence e-mail: andrew.try@mq.edu.au

## Key indicators

Single-crystal X-ray study

$T = 150$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å

$R$  factor = 0.032

$wR$  factor = 0.092

Data-to-parameter ratio = 14.1

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

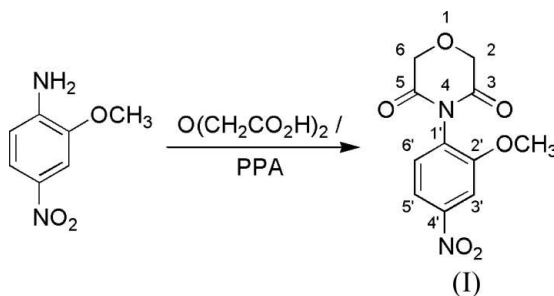
The crystal structure of the title compound,  $\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}_6$ , is stabilized by  $\text{C}-\text{H} \cdots \pi$  interactions.

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## Comment

The reaction between anilines, or substituted anilines, and either succinic or glutaric acid in polyphosphoric acid (PPA) to afford *N*-phenyl succinimides and glutarimides was recently reported (Mederski *et al.*, 2003). An analogous reaction between 4-nitroaniline and diglycolic acid failed to afford a cyclic diamide but unexpectedly yielded the first synthesis of 2,8-dinitro Tröger's base. In contrast, the synthesis of (I) was achieved by reacting 2-methoxy-4-nitroaniline with diglycolic acid in PPA, *i.e.* the anticipated cyclic diamide product.

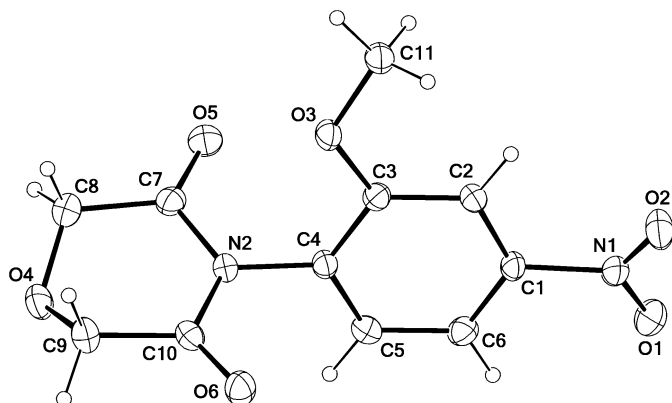


We were interested in preparing a range of dinitro Tröger's base compounds and isolated (I) as a by-product in one of these reactions.

A short contact of 2.838 (2) Å exists between N1 and O6 of an adjacent molecule, as shown in Fig. 2. There are two  $\text{C}-\text{H} \cdots \pi$  interactions which put this close contact in perspective. H9A is 2.94 Å from the centroid of the benzene ring attached to N1; however, H11A which is in contact with the opposite side of the benzene ring is able to approach more closely at 2.76 Å from the centroid (symmetry codes for O6 and H9A:  $x, y - 1, z$ ; for H11A:  $1 - x, -y, -z$ ). Atoms H9A and H11A are, respectively, 2.92 and 2.75 Å from the plane of the benzene ring and the close proximity of atom O6 also forces N1 to be pushed 0.104 (2) Å away from this plane.

## Experimental

A mixture of 2-methoxy-4-nitroaniline (1.00 g, 5.95 mmol), diglycolic acid (1.28 g, 9.58 mmol) and polyphosphoric acid (86%, d 1.9, 10.0 g) was heated at 353 K for 12 h under a drying tube. After cooling, water (50 ml) was added and the reaction mixture was neutralized with sodium hydroxide (3 M). The mixture was extracted with dichloromethane (3 × 50 ml) and the combined organic layers were washed with brine (50 ml), dried over anhydrous sodium sulfate, filtered and



**Figure 1**  
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

evaporated to dryness to yield a pale-yellow solid. The crude material was chromatographed (silica gel, dichloromethane) to afford (I) (546 mg, 34%) as a white solid. A second compound (the 4,10-dimethoxy-2,8-dinitro Tröger's base analogue, 224 mg, 20%) was subsequently eluted from the column.

#### Crystal data

$C_{11}H_{10}N_2O_6$	$V = 555.3 (3) \text{ \AA}^3$
$M_r = 266.21$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.592 \text{ Mg m}^{-3}$
$a = 7.526 (2) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.554 (2) \text{ \AA}$	$\mu = 0.13 \text{ mm}^{-1}$
$c = 10.259 (3) \text{ \AA}$	$T = 150 (2) \text{ K}$
$\alpha = 100.460 (4)^\circ$	Prism, yellow
$\beta = 102.539 (4)^\circ$	$0.54 \times 0.32 \times 0.29 \text{ mm}$
$\gamma = 94.732 (4)^\circ$	

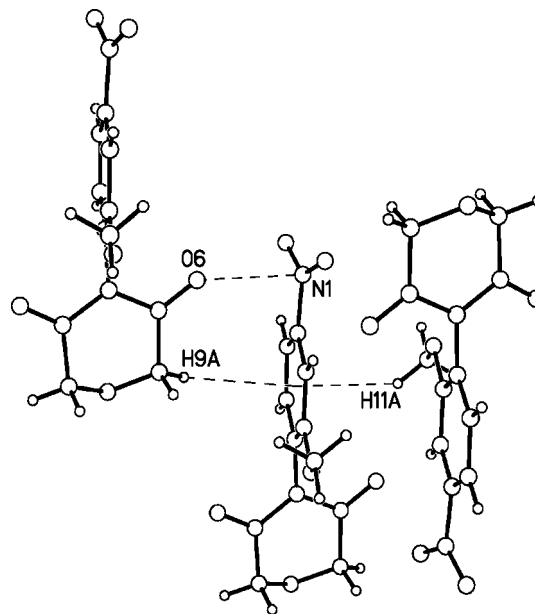
#### Data collection

Bruker SMART 1000 CCD diffractometer	(Siemens, 1995)
$\omega$ scans	$T_{\min} = 0.939, T_{\max} = 0.972$
Absorption correction: Gaussian [GAUSSIAN (Coppens <i>et al.</i> , 1965) and XPREP	5320 measured reflections
	2433 independent reflections
	2109 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.030$
	$\theta_{\text{max}} = 27.1^\circ$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.1137P]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.092$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
2433 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
173 parameters	
H-atom parameters constrained	

H atoms were positioned geometrically, with C—H = 0.95, 0.99 and 0.98 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$ , where  $x = 1.5$  for methyl H and  $x = 1.2$  for all other H atoms. The



**Figure 2**  
A schematic representation of the interaction between N1 and O6 and also two C—H... $\pi$  interactions (dashed lines). (Symmetry code for O6 and H9A:  $x, y - 1, z$ ; for H11A:  $1 - x, -y, -z$ .)

methyl group was free to rotate about the C—O bond in the refinement.

Data collection: SMART (Siemens, 1995); cell refinement: SMART; data reduction: SAINT (Siemens, 1995) and XPREP (Siemens, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: Xtal3.6 (Hall *et al.*, 1999), ORTEPII (Johnson, 1976), and WinGX (Farrugia, 1999); software used to prepare material for publication: WinGX.

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