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5-[(4-Methoxyphenylamino)methylene]-2,2-dimethyl-1,3-dioxane-4,6-dione

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.111
Data-to-parameter ratio = 12.8

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

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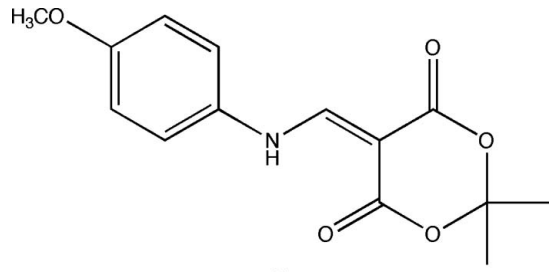
In the title compound, $C_{14}H_{15}NO_5$, the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The amino H atom has one intra- and one intermolecular contact to carbonyl O atoms, with O···H distances of 2.11 (2) and 2.31 (2) Å.

Comment

The condensation of arylidene derivatives of Meldrum's acid with cyclic monoamines has led to the efficient preparation of a diverse range of cyclic compounds such as quinolinone, 3-hydroxythiophene, naphthols, azepin-3(2*H*)-ones or pyrrolizin-3-ones (Gaber & McNab, 2001). Moreover, various derivatives of Meldrum's acid have been investigated by X-ray crystallography (Gould *et al.*, 1998; Blake *et al.*, 1997; Blake, Gould *et al.*,1994; Blake & McNab, 1995; Blake, McNab & Morrow, 1994). As part of a continuing study of the conformation in the solid state of 5-aminomethylene Meldrum's acid derivatives (Joussef *et al.*, 2005*a*,*b*), we report here the structure of the title compound, (I).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The dihedral angle C6—N7—C8—C9 is −177.5 (2)° and the distances C6—N7 and C8—C9 indicate delocalization. The H atom of the NH group has one intra- and one intermolecular contact to atom O10; details are given in Table 1. The delocalization of the N-atom lone pair into the Meldrum's acid ring may be favoured in the direction of one of

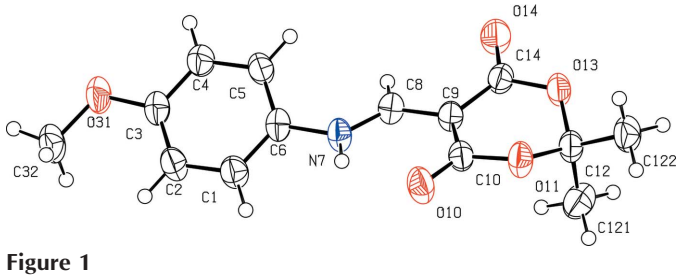


Figure 1
The molecular structure of (I), showing the atom labeling, with displacement ellipsoids drawn at the 50% probability level.

the two carbonyl groups (Blake *et al.*, 2003). The bond lengths and angles are in good agreement with values retrieved from the Cambridge Structural Database (Version 5.25; Allen, 2002).

Experimental

The title compound was prepared according to a literature procedure (Cassis *et al.*, 1985) and was recrystallized from methanol.

Crystal data

$C_{14}H_{15}NO_5$
M_r = 277.27
Monoclinic, $P2_1/c$
a = 13.007 (3) Å
b = 7.1317 (14) Å
c = 14.743 (3) Å
β = 102.14 (3)°
V = 1337.0 (5) Å^3
Z = 4
D_x = 1.377 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 5.1–18.6°
μ = 0.11 mm^{-1}
T = 193 (2) K
Irregular block, colorless
0.43 × 0.33 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2464 measured reflections
2362 independent reflections
1615 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.1°
$h = -15 \rightarrow 15$
$k = -8 \rightarrow 0$
$l = -17 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.111
S = 1.03
2362 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.2139P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7···O10	0.86 (2)	2.11 (2)	2.729 (2)	128 (2)
N7—H7···O10^{i}	0.86 (2)	2.31 (2)	3.088 (2)	151 (2)

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

The amino H atom was located in a difference map and was refined freely. C-bound H atoms were positioned with idealized geometry and were refined as riding, with C—H = 0.93 Å (0.96 Å for methyl groups) and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

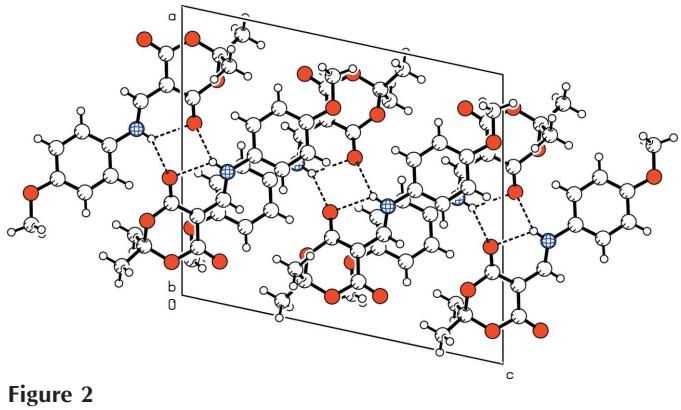


Figure 2
The molecular packing of (I), viewed along the b axis, with hydrogen bonds shown as dashed lines.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4/PC Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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