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## Key indicators

Single-crystal X-ray study

 $T = 297$  KMean  $\sigma(\text{C}-\text{C}) = 0.004$  Å

Disorder in main residue

 $R$  factor = 0.076 $wR$  factor = 0.204

Data-to-parameter ratio = 12.9

For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.5-(Anilinomethylene)-2,2-dimethyl-1,3-  
dioxane-4,6-dione

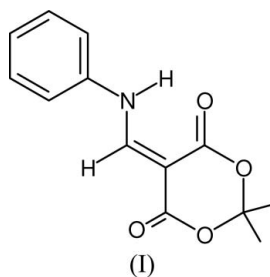
In the title enaminone,  $\text{C}_{13}\text{H}_{13}\text{NO}_4$ , the 1,3-dioxane-4,6-dione ring exhibits a distorted boat conformation. The amino H atom has one intramolecular contact to a carbonyl O atom, forming a six-membered ring. Molecules related by an inversion center are linked into dimers by  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

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## Comment

Recently, we reported the structure of an enaminone derived from Meldrum's acid (Vencato *et al.*, 2006). The synthesis of functionalized enaminones is a theme of ongoing interest owing to their potential in the synthesis of heterocyclic compounds (Negri *et al.*, 2004; Lue & Greenhill, 1997; Kuckländer, 1994; Ferraz & Pereira, 2004). A particular class of such compounds is derived from Meldrum's acid and the solid-state structural study of such derivatives has been described (Cunha *et al.*, 2003, 2005; Vencato *et al.*, 2004; da Silva *et al.*, 2005a,b, 2006; Joussef *et al.*, 2005a,b).



In the title compound, (I) (Fig. 1 and Table 1), the Meldrum's acid group has a distorted boat conformation, indicated by the Cremer and Pople parameters (Cremer & Pople, 1975):  $\text{O}5 \rightarrow \text{C}4 \rightarrow \dots \rightarrow \text{C}6$  [ $Q = 0.419$  (2) Å,  $\theta = 63.4$  (3)° and  $\varphi = 300.5$  (3)°]. As atom O10 is disordered, the angle  $\text{O}10-\text{C}8-\text{C}3$  of  $129.9$  (4)° is somewhat enlarged, and short contacts are observed for  $\text{H}2\cdots\text{O}10$  and  $\text{H}2\cdots\text{O}10'$  (2.539 and 2.285 Å, respectively). The torsion angle  $\text{C}13-\text{N}1-\text{C}2-\text{C}3$  is  $178.4$  (2)° and the  $\text{N}1-\text{C}13$  and  $\text{C}2-\text{C}3$  distances indicate delocalization.

Two bifurcated hydrogen bonds are observed in the structure of (I). The amino  $\text{N}1-\text{H}1$  group acts as a bifurcated hydrogen-bond donor, intramolecularly to O9 and intermolecularly to another O9<sup>i</sup> atom from an adjacent centrosymmetrically related molecule [symmetry code: (i)  $1 - x, 1 - y, 1 - z$ ] (Table 2). Another bifurcated intermolecular hydrogen bond (non-classical),  $\text{C}18-\text{H}18\cdots\text{O}5^i$  and  $\text{C}18-\text{H}18\cdots\text{O}9^i$ , forms a dimer about an inversion center. This particular arrangement results in a short  $\text{O}9\cdots\text{O}9^i$  contact of

2.856 (3) Å. The non-classical intermolecular hydrogen bond C15—H15<sup>i</sup>···O10<sup>ii</sup> [symmetry code: (ii)  $2 - x, 2 - y, 1 - z$ ] connects the dimers, forming endless ribbons along the [110] direction and close to the plane ( $\bar{1}12$ ) (Fig. 2). A close contact C12···O10<sup>iii</sup> [3.556 (6) Å; symmetry code: (iii)  $2 - x, 1 - y, 2 - z$ ] connects neighbouring ribbons.

## Experimental

A solution of 144 mg (1.0 mmol) of Meldrum's acid in 1.5 ml (19.6 mmol) of trimethyl orthoformate was heated under reflux for 3 h after which time the solvent was evaporated. To the resulting yellow solid was added 165 mg (2.0 mmol) of aniline in 3 ml of MeOH and the solution was allowed to stand at room temperature with stirring for 24 h. The solvent was evaporated and the crude solid was recrystallized from EtOH/petroleum ether (4:1), affording (I) as light-yellow crystals (205 mg; 87% yield; mp 424–425 K). Single crystals were obtained by diffusion of petroleum ether into a solution of (I) in CH<sub>3</sub>Cl at room temperature.

### Crystal data

C <sub>13</sub> H <sub>13</sub> NO <sub>4</sub>	$V = 619.5 (2) \text{ \AA}^3$
$M_r = 247.24$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.325 \text{ Mg m}^{-3}$
$a = 5.495 (1) \text{ \AA}$	Cu $K\alpha$ radiation
$b = 10.594 (2) \text{ \AA}$	$\mu = 0.83 \text{ mm}^{-1}$
$c = 11.589 (3) \text{ \AA}$	$T = 297 (2) \text{ K}$
$\alpha = 71.42 (2)^\circ$	Block, light yellow
$\beta = 82.24 (2)^\circ$	$0.33 \times 0.28 \times 0.28 \text{ mm}$
$\gamma = 76.10 (2)^\circ$	

### Data collection

Enraf–Nonius CAD-4 diffractometer	1893 reflections with $I > 2\sigma(I)$
non-profiled $\omega/2\theta$ scans	$R_{\text{int}} = 0.018$
Absorption correction: none	$\theta_{\text{max}} = 67.0^\circ$
2268 measured reflections	2 standard reflections
2158 independent reflections	frequency: 120 min
	intensity decay: 1%

### Refinement

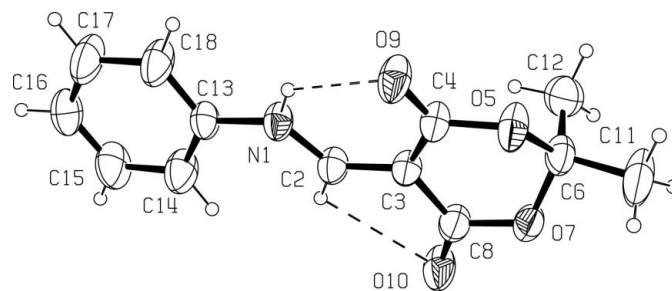
Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.1162P)^2 + 0.1316P]$
$R[F^2 > 2\sigma(F^2)] = 0.076$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.204$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.10$	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
2158 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
167 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.217 (16)

**Table 1**

Selected geometric parameters (Å, °).

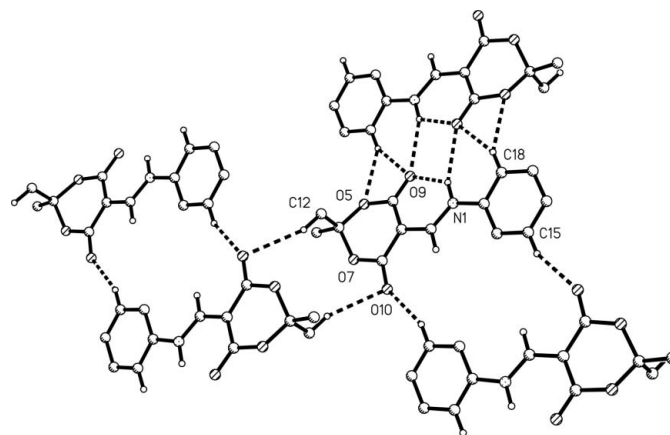
N1—C2	1.311 (3)	O5—C6	1.432 (3)
N1—C13	1.420 (2)	C6—O7	1.423 (3)
C2—C3	1.373 (3)	O7—C8	1.354 (2)
C4—O5	1.353 (2)	O9—O9 <sup>i</sup>	2.856 (3)
C2—N1—C13	125.03 (17)	O5—C6—C11	105.9 (2)
C13—N1—C2—C3	178.4 (2)	C2—N1—C13—C14	9.3 (4)
N1—C2—C3—C4	3.7 (4)		

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



**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond and short contact are shown as dashed lines. The disordered atom O10' is not shown.



**Figure 2**

The hydrogen-bonding (dashed lines) scheme in (I), viewed approximately perpendicular to the plane ( $\bar{1}11$ ). Only the H atoms involved in hydrogen bonds are shown. Only one disorder component is shown.

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 <sup>i</sup> ···O9	0.86	2.14	2.760 (2)	129
N1—H1 <sup>i</sup> ···O9 <sup>i</sup>	0.86	2.46	3.259 (2)	155
C18—H18···O5 <sup>i</sup>	0.93	2.60	3.432 (3)	150
C18—H18···O9 <sup>i</sup>	0.93	2.44	3.288 (3)	151
C15—H15···O10 <sup>ii</sup>	0.93	2.45	3.310 (10)	154

Symmetry codes: (i)  $-x + 1, -y + 1, -z + 1$ ; (ii)  $-x + 2, -y + 2, -z + 1$ .

Some non-H atoms show abnormally high displacement parameters, but all these atoms were refined with anisotropic displacement parameters with a positive definite thermal tensor. All H atoms were placed in calculated positions ( $C-H = 0.96$  and  $0.97 \text{ \AA}$ ) and treated as riding atoms [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$  or  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$ ]. Atom O10 was found to be disordered between two positions, their site-occupation factors were refined but later fixed at 0.70 and 0.30.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; 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: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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