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5-(Aminomethylene)-2,2-dimethyl-1,3-dioxane-4,6-dione

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.103
Data-to-parameter ratio = 10.4

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

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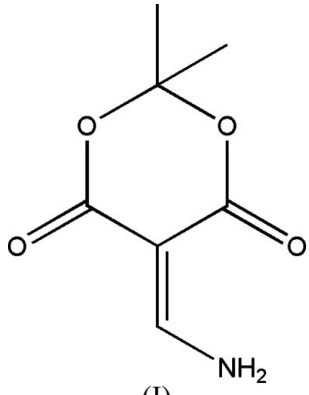
In the title compound $C_7H_9NO_4$, the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation. Intermolecular hydrogen bonds connect the molecules into chains. One intramolecular N—H···O hydrogen bond to a carbonyl O atom is also observed, forming a six-membered ring.

Comment

Meldrum's acid and its derivatives serve as key intermediates for the synthesis of heterocyclic compounds with pharmacological activity (Gaber & McNab, 2001). However, their biological properties have scarcely been investigated (Herzog & Reinshagen, 1976; Lukevics *et al.*, 2003). On the other hand, several X-ray studies with Meldrum's acid have been described (Blake *et al.*, 1995, 2003; Low *et al.*, 2002). In the light of these interests (da Silva *et al.*, 2005*a*,*b*), we report here the crystal structure of the title compound, (I).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation, with C4 in the flap position. The torsion angle N1—C1—C2—C5 is 176.7 (2)°. One H atom of the NH_2 group

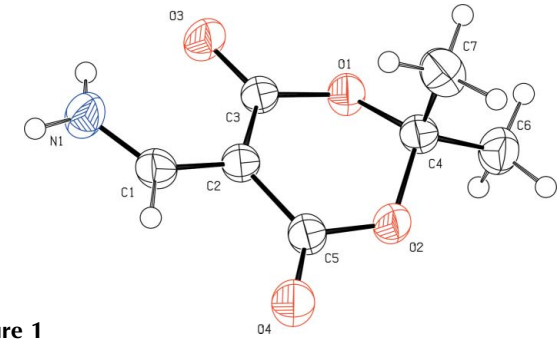


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

has an intramolecular contact to O3, with a H···O distance of 2.24 (2) Å, forming a six-membered ring. Three intermolecular hydrogen bonds of types N—H···O and C—H···O connect the molecules into chains. Details of the hydrogen bonding are given in Table 2 and shown in Fig. 2.

Experimental

The title compound was prepared according to the literature procedure of Chen (1991). Single crystals of (I) suitable for X-ray data collection were obtained by recrystallization from methanol.

Crystal data

$C_7H_9NO_4$
M_r = 171.15
Monoclinic, $C2/c$
a = 9.830 (1) Å
b = 9.609 (1) Å
c = 16.819 (2) Å
β = 90.95 (1)°
V = 1588.4 (3) Å^3
Z = 8
D_x = 1.431 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 1.02 mm^{-1}
T = 299 (2) K
Prism, colourless
0.20 × 0.15 × 0.10 mm

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1667 measured reflections
1420 independent reflections
1063 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 66.9°
3 standard reflections
frequency: 120 min
intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.103
S = 1.05
1420 reflections
137 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.1561P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0016 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11*N*···O3	0.84 (2)	2.24 (2)	2.818 (2)	126 (2)
N1—H11*N*···O3^{i}	0.84 (2)	2.30 (2)	3.045 (2)	146 (2)
N1—H12*N*···O4^{ii}	0.93 (2)	2.12 (2)	2.929 (2)	145 (2)
C1—H1···O2^{ii}	0.96 (2)	2.50 (2)	3.398 (2)	156.7 (17)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

The H atoms were located in difference map, and their positional parameters were refined freely [N—H = 0.84 (2)–0.93 (2) Å and C—H = 0.94 (2)–1.01 (2) Å]. Isotropic displacement parameters were set equal to $1.2U_{eq}$(parent atom).

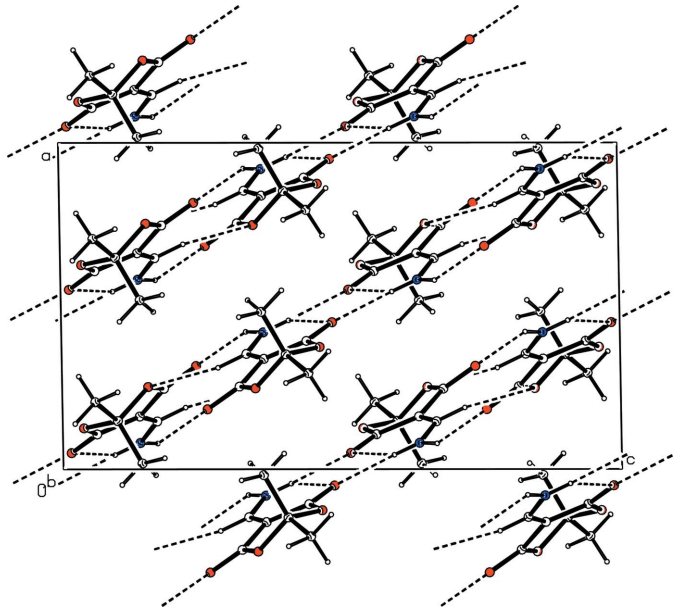


Figure 2
The molecular packing of (I), with hydrogen bonds shown as dashed lines.

Data collection: *CAD-4 Software* (Nonius, 1996); cell refinement: *CAD-4 Software*; data reduction: *REDU4* (Stoe & Cie, 1987); 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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