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2,2-Dimethyl-5-[(6-methylpyridin-2-ylamino)-methylene]-1,3-dioxane-4,6-dione

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.129
Data-to-parameter ratio = 13.6

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

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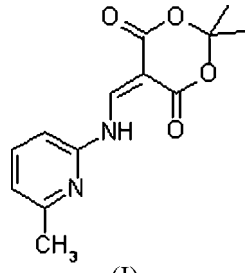
In the title compound $C_{13}H_{14}N_2O_4$, the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation. The amino H atom forms intra- and intermolecular contacts to carbonyl O atoms. One intermolecular C—H···O hydrogen bond is also observed.

Comment

Meldrum's acid and its derivatives serve as key intermediates for the synthesis of heterocyclic compounds with pharmacological activity (Chen, 1991; Delfourne *et al.*, 2000). Thus, the present X-ray crystallographic study of the title compound, (I), is part of our ongoing search for biological compounds based on 5-aminomethylene Meldrum's acid derivatives (da Silva *et al.*, 2005*a*,*b*, 2006).



(I)

In compound (I), the 1,3-dioxane-4,6-dione ring exhibits an envelope conformation with atom C9 in the flap position. The C1—N2—C6—C7 torsion angle and the C1—N2 and C6—C7 distances (Table 1) indicate electron delocalization between the two rings. The delocalization of the N-atom lone pair into the ring of Meldrum's acid may be favoured in the direction of

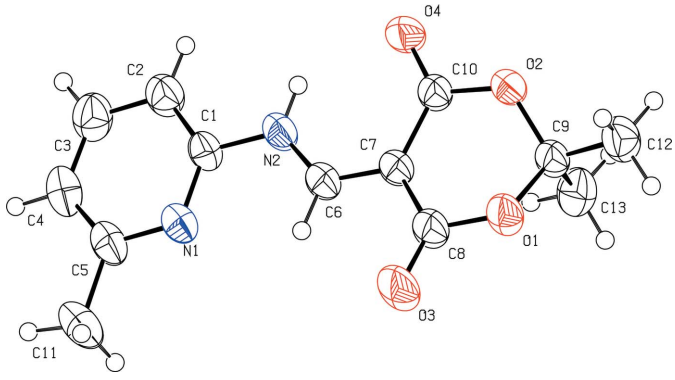


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Only one of the two disordered sets of H atoms of the methyl group C11 is shown.

one of the two available carbonyl groups, C8═O3 and C10═O4 (Blake *et al.*, 2003).

The H atom of the NH group forms one intra- and one intermolecular hydrogen bond to the carbonyl atom O4. One intermolecular C—H···O hydrogen bond is also observed. 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 Cannon *et al.* (2001) and was recrystallized from methanol (m.p. 434 K; yield 80%).

Crystal data

$C_{13}H_{14}N_2O_4$
M_r = 262.26
Monoclinic, $C2/c$
a = 17.101 (1) Å
b = 7.354 (1) Å
c = 21.375 (2) Å
β = 91.868 (9)°
V = 2686.7 (5) Å^3
Z = 8
D_x = 1.297 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 0.82 mm^{-1}
T = 299 (2) K
Needle, colourless
0.50 × 0.10 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3300 measured reflections
2394 independent reflections
1901 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 66.9°
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.07
2394 reflections
176 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2 + 0.4614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.040
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0045 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.328 (2)	N2—C1	1.423 (2)
N1—C5	1.344 (2)	C6—C7	1.372 (2)
C1—N2—C6—C7	178.15 (15)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O4	0.86	2.15	2.7683 (18)	128
N2—H2*N*···O4^{i}	0.86	2.46	3.2652 (18)	155
C2—H2···O4^{i}	0.93	2.51	3.335 (2)	148

Symmetry code: (i) $-x, -y+2, -z$.

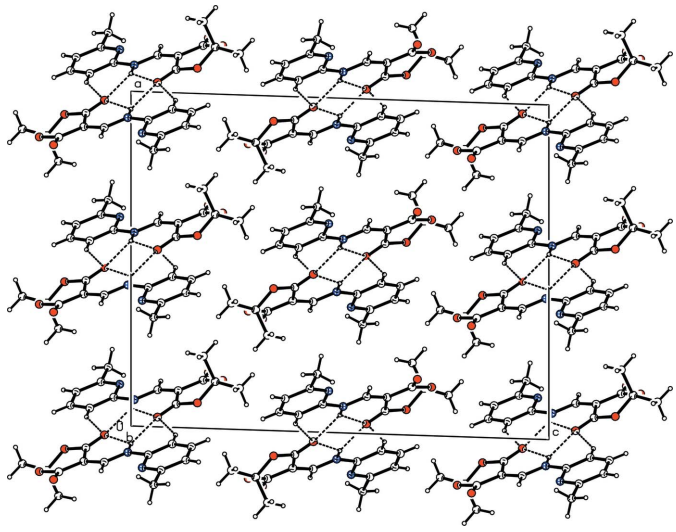


Figure 2
The molecular packing of (I), with hydrogen bonds shown as dashed lines. Only one of the two disordered sets of H atoms of the methyl group C11 is shown.

All H atoms were included in the riding-model approximation, with N—H = 0.86 Å and C—H in the range 0.93–0.96 Å. Isotropic displacement parameters were set equal to $1.2U_{eq}$(parent atom). The methyl group C11 is disordered and was refined with two equally occupied positions rotated from each other by 60°.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC 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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