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3,3-Dimethylpyrrolidine-2,4-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.123
Data-to-parameter ratio = 16.2

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

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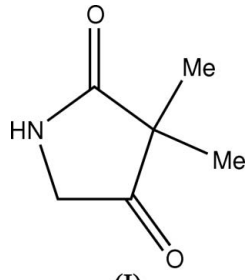
In the title compound, $C_6H_9NO_2$, the asymmetric unit consists of three molecules. All the five-membered rings are essentially planar. The molecule is stabilized by intermolecular N—H···O and C—H···O interactions, forming a two-dimensional network parallel to (100).

Comment

Five-membered nitrogen-heterocyclic compounds, such as piracetam (Gouliaev *et al.*, 1994) and clausenamide (Hartwig *et al.*, 1987), are known to possess diverse medicinal properties, which include antibiotic, antibacterial, antifungal and cytotoxic effects. The title compound, (I), was prepared as an intermediate for synthesizing more complex biologically active natural products.



(I)

The asymmetric unit of (I) consists of three molecules (Fig. 1). The absence of bulky groups allows the five-membered rings, *viz*. N1/C1–C4, N2/C7–C10 and N8/C13–C16, to be approximately planar, with a maximum deviation of 0.077 (2) Å for atom C16. This contrasts with the situation in methyl 3-benzyl-4-hydroxy-2-oxopyrrolidine-3-carboxylate (Hamzah *et al.*, 2003). The bond lengths and angles are in normal ranges (Allen *et al.*, 1987). The molecule is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds (Table 2), forming a two-dimensional network parallel to (100) (Fig. 2).

Experimental

The synthetic approach to the title compound began with the condensation between glycine methyl ester (3.77 g, 30 mmol) and methyl malonate potassium salt (4.68 g, 30 mm mol) to give a diester (5.22 g, 92% yield). Dickmann cyclization of this diester (3.78 g, 20 mmol) with sodium/methanol in 50 ml toluene under reflux gave a β,β-diketoester, methyl 2,4-dioxopyrrolidine-3-carboxylate (2.84 g, 90% yield). Dimethoxycarbonylation of this β,β-diketoester (1.00 g, 6.37 mmol) was successfully carried out in 30 ml acetonitrile to give an intermediate with a basic pyrrolidinone ring skeleton (0.62 g, 98%

yield). Dialkylation of this intermediate (0.50 g, 5.05 mmol) was then carried out in 50 ml tetrahydrofuran solution containing excess methyl iodide (3.5 g, 25 mmol) and 25 ml of 1 *M* tetrabutylammonium fluoride (25 mmol) to give compound (I) (0.20 g, 32% yield). Crystals suitable for X-ray investigation were obtained by slow evaporation of a diethyl ether solution (m.p. 141.8–415.9 K).

Crystal data

$C_6H_9NO_2$
M_r = 127.14
Monoclinic, $C2/c$
a = 28.745 (6) Å
b = 12.653 (2) Å
c = 11.588 (2) Å
β = 101.966 (3)°
V = 4122.9 (14) Å^3
Z = 24
D_x = 1.229 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colorless
0.44 × 0.36 × 0.26 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.960, T_{max} = 0.976
10969 measured reflections
4053 independent reflections
3168 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
S = 1.03
4053 reflections
250 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.9776P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2317 (19)	N1—C1	1.329 (2)
O2—C3	1.2031 (18)	N1—C2	1.445 (2)
O3—C9	1.2018 (18)	N2—C7	1.334 (2)
O4—C7	1.2270 (18)	N2—C8	1.4503 (19)
O5—C15	1.2016 (19)	N3—C13	1.3318 (19)
O6—C13	1.2326 (18)	N3—C14	1.451 (2)
N1—C1—C4—C3	−2.31 (17)	N3—C13—C16—C15	−12.06 (17)
N2—C7—C10—C9	−5.01 (18)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	1.99	2.8433 (19)	171
N2—H2C···O6^{ii}	0.86	2.18	3.0023 (18)	161
N3—H3A···O4^{iii}	0.86	2.04	2.8814 (18)	166
C5—H5C···O3^{iv}	0.96	2.58	3.511 (3)	163
C8—H8A···O5^{v}	0.97	2.56	3.337 (2)	137
C17—H17C···O2^{vi}	0.96	2.54	3.369 (3)	145

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

All H atoms were located in a difference map, but they were repositioned geometrically, with C—H = 0.96–0.97 Å and N—H = 0.86 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

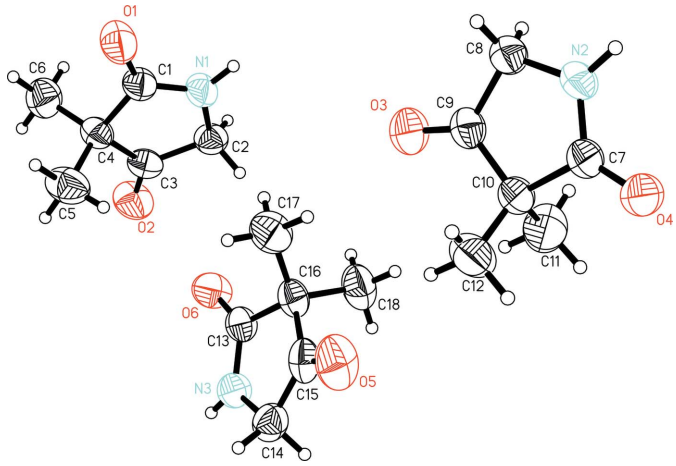


Figure 1
The asymmetric unit of (I), shown with 50% probability displacement ellipsoids

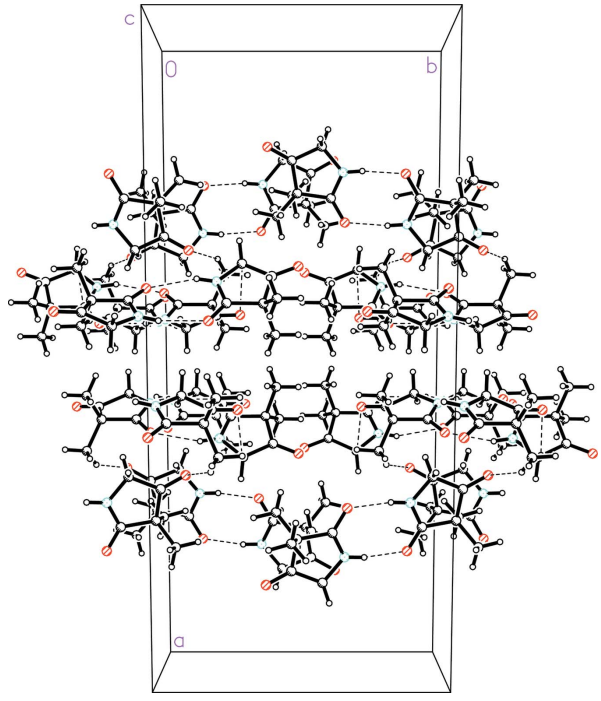


Figure 2
The molecular packing of (I), viewed down the *c* axis. The dashed lines indicate N—H···O and C—H···O hydrogen bonds.

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