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6-Isopropoxy-5-isopropylpyrimidine-2,4(1*H*,3*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 163 K
Mean σ(C–C) = 0.005 Å
R factor = 0.077
wR factor = 0.198
Data-to-parameter ratio = 16.8

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

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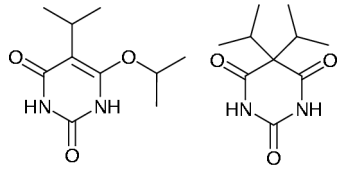
Two molecules of the title compound, $C_{10}H_{16}N_2O_3$, constitute the asymmetric unit. The molecules form a two-dimensional hydrogen-bonded sheet in the *ab* plane.

Comment

5,5-Diisopropylbarbituric acid, (II), is a pivotal structure for structure–reactivity and structure–activity relationship studies of barbituric acid derivatives (McKeown, 1980*a*; McKeown *et al.*, 1986; Wong & McKeown, 1988; Prankerd & McKeown, 1990, 1992*a*,*b*, 1994). The original synthesis of this compound (Preiswerk, 1923) involved the reaction of isopropyl bromide with 5-isopropylbarbituric acid. Later studies (McKeown, 1980*b*) identified the product of this reaction to be (I), for which we now report the crystal structure.



(I) (II)

Compound (I) crystallizes with two molecules in the asymmetric unit (Fig. 1). These molecules differ in the orientation of both the isopropyl and isoproproxy groups (see Table 1). The extended structure of (I) consists of a complex hydrogen-bonded two-dimensional corrugated sheet (Fig. 2). Each molecule of (I) forms two hydrogen bonds with an equivalent molecule related by a centre of symmetry. These two molecule units further extend to other units by single hydrogen bonds, forming a two-dimensional sheet. The two-

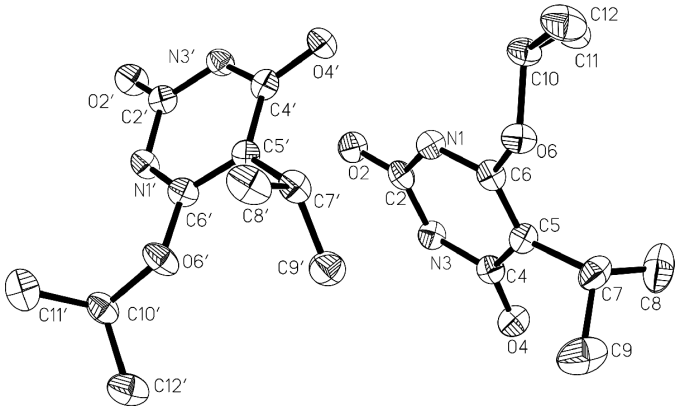


Figure 1
A view of the asymmetric unit of (I), showing displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

dimensional sheets consist of the central hydrogen-bonded layer, covered by two outside layers consisting of the isopropyl and isopropoxy groups. These two-dimensional sheets then stack in the **c** direction (Fig. 3). All N-bound H atoms are involved in hydrogen bonds, while three potential hydrogen-bond acceptors (O2 and the two ether atoms O6 and O6′) do not participate (Table 2).

Experimental

The title compound was prepared by a literature method (Preiswerk, 1923). Recrystallization from ethanol acidified with acetic acid provided crystals suitable for X-ray analysis.

Crystal data

$C_{10}H_{16}N_2O_3$
$M_r = 212.25$
Triclinic, $P\overline{1}$
$a = 8.769$ (6) Å
$b = 11.178$ (8) Å
$c = 12.418$ (9) Å
$\alpha = 71.074$ (9)°
$\beta = 77.684$ (9)°
$\gamma = 88.014$ (9)°
$V = 1124.1$ (13) Å^3
$Z = 4$
$D_x = 1.254$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2889 reflections
$\theta = 4.8$–51.9°
$\mu = 0.09$ mm^{-1}
$T = 163$ (2) K
Needle, colourless
0.70 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.587$, $T_{max} = 1.000$
14 452 measured reflections
4703 independent reflections
2497 reflections with $I > 2\sigma(I)$
$R_{int} = 0.068$
$\theta_{max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 14$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.198$
$S = 0.91$
4703 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1178P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.032 (5)

Table 1
Selected torsion angles (°).

C4—C5—C7—C8	63.6 (4)	C4′—C5′—C7′—C8′	109.8 (3)
C4—C5—C7—C9	−62.8 (4)	C4′—C5′—C7′—C9′	−123.5 (3)
C5—C6—O6—C10	138.6 (3)	C5′—C6′—O6′—C10′	−172.1 (3)
C6—O6—C10—C11	−56.1 (3)	C6′—O6′—C10′—C11′	−91.4 (3)
C6—O6—C10—C12	−177.6 (2)	C6′—O6′—C10′—C12′	146.8 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4′	0.88	2.01	2.814 (3)	150
N3—H3A···O4^{i}	0.88	2.01	2.863 (3)	163
N1′—H1D···O2^{ii}	0.88	1.96	2.814 (3)	165
N3′—H3D···O4′^{iii}	0.88	1.97	2.853 (3)	176

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

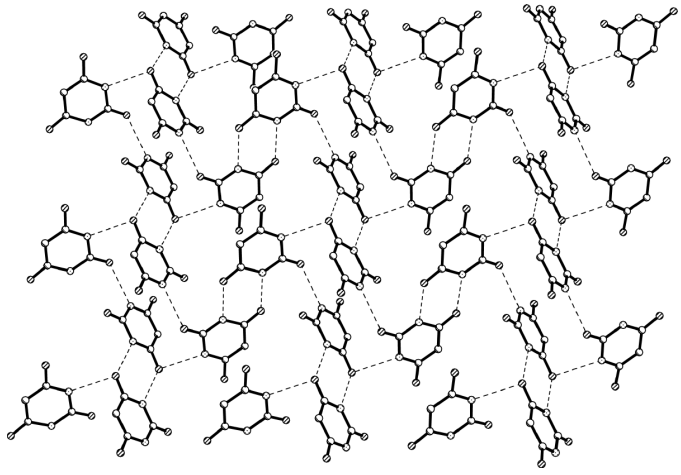


Figure 2
The two-dimensional hydrogen-bonded (dashed lines) sheet. H atoms and isopropyl groups have been omitted for clarity.

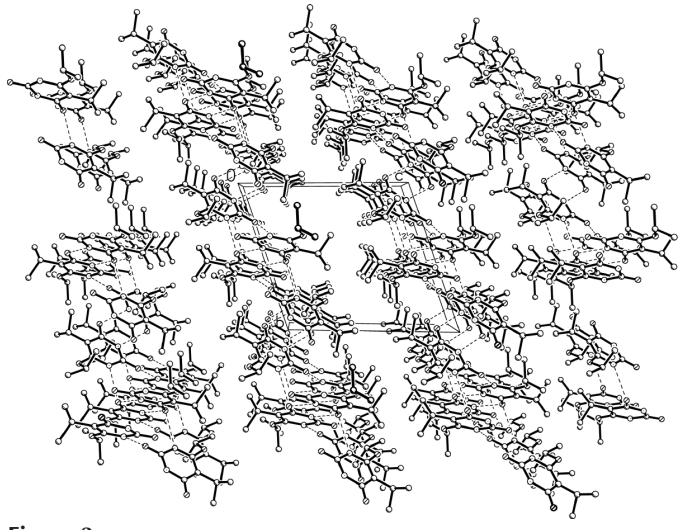


Figure 3
A packing diagram, showing how the two-dimensional hydrogen-bonded (dashed lines) sheets stack in the *ab* plane. H atoms have been omitted for clarity.

All crystals appeared to be multiples or twins and a single crystal could not be isolated. A large needle was mounted for data collection, from which three major domains were identified using *GEMINI* (Bruker, 1999). Data were extracted from all three domains, but the second and third domains displayed poor internal agreement, as well as being weaker data, and so were discarded. All H atoms were placed in calculated positions as riding atoms, with distances of 0.88 (NH), 1.00 (CH) and 0.98 (CH_3), and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $1.2U_{eq}(X)$ where X is the carrier atom.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

A sample of the diethylamine salt of (I) from the original work (Preiswerk, 1923) was kindly provided (in 1969) by Dr A. M. C. Duffus, Roche Products Ltd, Welwyn Garden City,

England. The acid, isolated from the salt, was shown to be the same as (I) obtained in our work.

References

Bruker (1999). *GEMINI*, *SADABS*, *SAINT-Plus* (Version 6.22) and *SMART* (Version 5.045). Bruker AXS Inc., Madison, Wisconsin, USA.

McKeown, R. H. (1980*a*). *J. Chem. Soc. Perkin Trans.* 2, pp. 515–522.

McKeown, R. H. (1980*b*). *J. Chem. Soc. Perkin Trans.* 2, pp. 504–514.

McKeown, R. H., Prankerd, R. J. & Wong, O. (1986). *Development of Drugs in Modern Medicine* edited by J. W. Gorrod, G. G. Gibson & M. Mitchard, pp. 80–89. Horwood: Chichester, England.

Prankerd, R. J. & McKeown, R. H. (1990). *Int. J. Pharm.* **62**, 37–52.

Prankerd, R. J. & McKeown, R. H. (1992*a*). *Int. J. Pharm.* **83**, 25–37.

Prankerd, R. J. & McKeown, R. H. (1992*b*). *Int. J. Pharm.* **83**, 39–45.

Prankerd, R. J. & McKeown, R. H. (1994). *Int. J. Pharm.* **112**, 1–15.

Preiswerk, E. (1923). *Helv. Chim. Acta*, **6**, 192–198.

Sheldrick G. M. (2001). *SHELXTL.* Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Wong, O. & McKeown, R. H. (1988). *J. Pharm. Sci.* **77**, 926–932.