

(±)-5-Ethyl-1-methyl-5-phenylpyrimidine-2,4,6-trione at 163 K**William Lewis,* Robert H. McKeown and Ward T. Robinson**

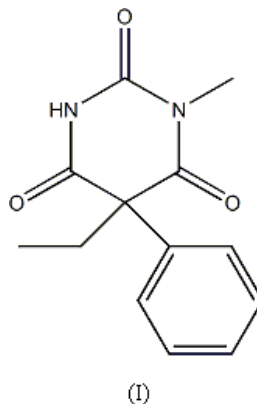
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Correspondence e-mail:
wle11@student.canterbury.ac.nz**Key indicators**Single-crystal X-ray study
T = 163 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
Disorder in main residue
R factor = 0.042
wR factor = 0.127
Data-to-parameter ratio = 12.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

A low-temperature [163 (2) K] redetermination of the crystal structure of the title compound, $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_3$ [originally determined by Bideau, Marly & Housty (1969). *C. R. Acad. Sci. Ser. C*, **269**, 549–551], has been performed. Although no gross structural differences have been observed, H atoms have been located and the extended structure examined.

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The preliminary crystal structure of the title compound, (I), was first described by Bideau *et al.* (1969). The original structure determination and refinement were performed from 551 structure factors using the method of symbolic addition (Karle, 1970), and involved refinement of non-H-atom positions and displacement parameters, with $R = 0.107$. Although this paper mentioned future refinement and location of H atoms, this was never published. We now present a low-temperature [163 (2) K] redetermination of this structure, locating the H atoms and examining the extended structure.



The structure shows no gross structural differences from the original determination, with the cell parameters, at most, different by 1%. The title compound crystallizes in space group $P2_1/c$, with four molecules in the unit cell (see Fig. 1 for the asymmetric unit). The individual molecules form centrosymmetric hydrogen-bonded dimers, which are cross-linked by weak $\text{C}-\text{H}\cdots\text{O}$ interactions (see Table 1), expanding the extended structure into a two-dimensional network (see Fig. 2).

Experimental

Compound (I) was generously donated by Kempthorne, Prosser and Co., Dunedin, New Zealand Drug Company Ltd, Pharmaceutical Laboratories.

Crystal data

C₁₃H₁₄N₂O₃
M_r = 246.26
 Monoclinic, *P*2₁/*c*
a = 13.636 (6) Å
b = 7.218 (3) Å
c = 12.467 (6) Å
 β = 105.398 (6)°
V = 1183.1 (9) Å³
Z = 4

D_x = 1.383 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 3964 reflections
 θ = 6.2–54.5°
 μ = 0.10 mm⁻¹
T = 163 (2) K
 Plate, colourless
 0.80 × 0.50 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1999)
T_{min} = 0.884, *T_{max}* = 1.000
 15219 measured reflections

2670 independent reflections
 1969 reflections with *I* > 2σ(*I*)
R_{int} = 0.058
 θ_{max} = 27.9°
h = -17 → 17
k = -9 → 5
l = -16 → 16

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.042
wR (*F*²) = 0.127
S = 1.08
 2670 reflections
 216 parameters
 Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + 0.1704P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
 Extinction correction: SHELXTL
 Extinction coefficient: 0.028 (4)

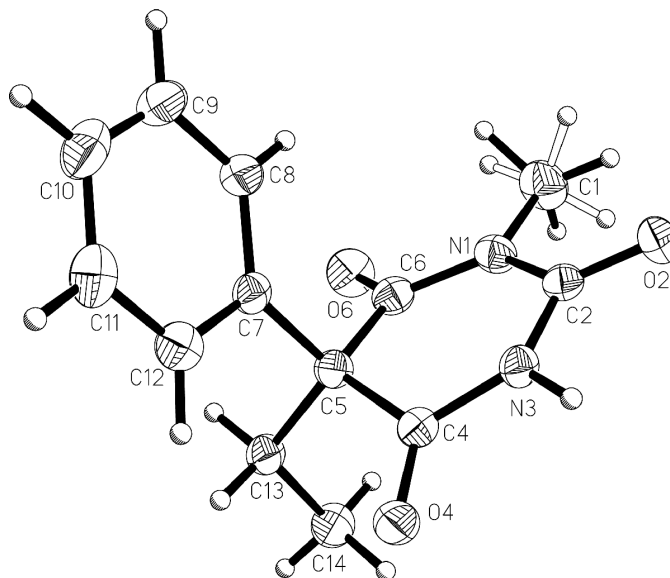


Figure 1
 The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius. The two sets of H atoms of the disordered methyl group are distinguished using solid and unfilled bonds.

Table 1

Geometric parameters for the hydrogen bond and the weak C–H···O interaction (Å, °).

| <i>D</i> –H··· <i>A</i> | <i>D</i> –H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> –H··· <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| N3–H3A···O4 ⁱ | 0.876 (18) | 2.003 (19) | 2.8787 (19) | 178.8 (17) |
| C12–H12A···O6 ⁱⁱ | 0.963 (19) | 2.48 (2) | 3.371 (2) | 154.1 (15) |

Symmetry codes: (i) 1 – *x*, –*y*, –*z*; (ii) *x*, –½ – *y*, *z* – ½.

All H atoms were located in a difference map and their coordinates were freely refined. Atomic displacement parameters were set at *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl C atoms and *U*_{iso}(H) = 1.2*U*_{eq}(*X*), where *X* is any other bonded carrier atom. The H atoms of the methyl group were disordered over two positions, with a 55 (4):45 (4) ratio of occupancies.

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.

References

Bideau, J.-P., Marly, L. & Housty, J. (1969). *C. R. Acad. Sci. Ser. C*, **269**, 549–551.
 Bruker (1999). SADABS (Version 2.03), SAINT-Plus (Version 5.045) and SMART (Version 6.22). Bruker AXS Inc., Madison, Wisconsin, USA.
 Karle, I. L. (1970). *Crystallographic Computing*, edited by S. R. Hall, pp. 19–25. Copenhagen: Munksgaard.
 Sheldrick, G. M. (2001). SHELXTL. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

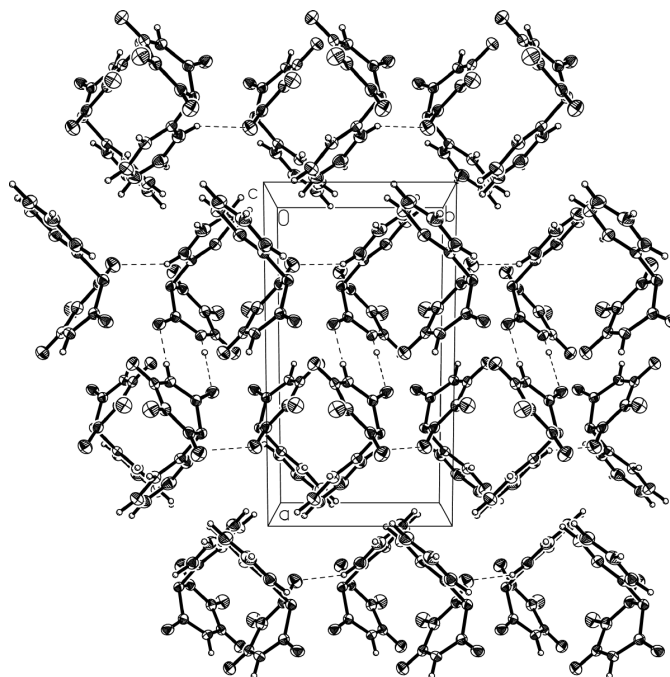


Figure 2
 A packing diagram of (I), showing the hydrogen-bonded dimer, as well as the weak C–H···O interactions (dashed lines). Methyl H atoms and the ethyl group have been omitted for clarity.