

Fig. 1. View of the molecule (40% thermal ellipsoids) showing the atom-numbering scheme, the *cis*-fused hydrindane ring system and the *cis* orientation with respect to the six-membered ring of the four hydrogen substituents on C(8), C(10), C(16) and C(18), respectively.

chemistry of the molecule showing the *cis*-fused hydrindane ring skeleton and the orientations of the four hydrogens shown in (I) is depicted in Fig. 1.

Related literature. The stereochemistry observed here is similar to that reported for the hydrindane skele-

Table 3. Selected torsion angles ($^{\circ}$)

C(3)—C(4)—C(7)—O(2)	3 (1)	C(25)—C(20)—C(19)—O(4)	-8 (1)
C(7)—O(2)—C(8)—C(18)	144.2 (7)	C(19)—O(4)—C(18)—C(8)	-101.5 (7)
O(2)—C(8)—C(9)—C(10)	-178.5 (6)	O(4)—C(18)—C(17)—C(16)	72.0 (8)
C(8)—C(9)—C(10)—C(11)	168.4 (7)	C(18)—C(17)—C(16)—C(15)	-79.8 (9)
C(8)—C(9)—C(10)—C(16)	51.1 (9)	C(18)—C(17)—C(16)—C(10)	39.8 (9)
C(9)—C(10)—C(11)—C(12)	-101.2 (8)	C(17)—C(16)—C(15)—C(12)	166.0 (7)
C(10)—C(11)—C(12)—C(14)	121.3 (7)	C(16)—C(15)—C(12)—C(13)	91.4 (9)

ton of the sesquiterpenoids illudol and marasmic acid (Cradwick & Sim, 1971).

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The Structure of Urea–Oxalic Acid (2/1) Determined by Neutron Diffraction at 100 K*

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Abstract. $2\text{CH}_4\text{N}_2\text{O}\cdot\text{C}_2\text{H}_2\text{O}_4$, $M_r = 210.15$, monoclinic, $P2_1/c$, $a = 5.048$ (1), $b = 12.374$ (3), $c = 6.876$ (2) Å, $\beta = 96.40$ (2) $^{\circ}$, $V = 426.8$ (1) Å 3 , $Z = 2$, $D_x = 1.63$ Mg m $^{-3}$, $\lambda = 1.296$ Å, $\mu = 0.231$ mm $^{-1}$, $T = 100.0$ (1) K, $R = 0.045$, $wR = 0.035$ for 895 unique observed reflections with $I > 2\sigma(I)$. The crystal structure contains urea and oxalic acid molecules in a hydrogen-bonded network, complex-

ing in a 2:1 ratio. The determination is in agreement with previous X-ray work [Harkema, Bats, Weyenburg & Feil (1972). *Acta Cryst.* **B28**, 1646–1648] and provides additional information on the H atoms.

Experimental. A solution containing urea and oxalic acid in the correct stoichiometric ratio was slowly evaporated at room temperature, resulting in crystal-line plates of a few millimeters in thickness and an area of several millimeters in the other dimensions. A flat triangular crystal with edges of length *ca* 2.5, 4.2

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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}^*
N(1)	9981 (2)	1509 (1)	4853 (2)	184 (4)
N(2)	6189 (2)	2139 (1)	3026 (2)	192 (4)
O(1)	7046 (4)	333 (1)	3297 (3)	159 (5)
O(2)	1580 (4)	1253 (1)	373 (3)	173 (5)
O(3)	2874 (4)	-382 (1)	1513 (3)	160 (5)
C(1)	7716 (3)	1304 (1)	3717 (2)	137 (4)
C(2)	1251 (3)	276 (1)	520 (2)	136 (4)
H(1)	4593 (7)	15 (3)	2252 (6)	284 (10)
H(2)	4587 (8)	1973 (3)	2069 (7)	356 (11)
H(3)	6790 (8)	2907 (3)	3298 (7)	380 (12)
H(4)	10511 (8)	2276 (3)	5145 (7)	352 (12)
H(5)	11044 (7)	877 (3)	5477 (6)	338 (11)

* Defined according to Willis & Pryor (1975).

Table 2. Bond distances (\AA) and angles ($^\circ$) and hydrogen-bond parameters ($\text{\AA}, ^\circ$)

N(1)—C(1)	1.335 (2)	O(1)—C(1)	1.273 (2)
N(1)—H(4)	1.000 (4)	O(3)—C(2)	1.294 (2)
N(1)—H(5)	1.016 (4)	O(3)—H(1)	1.074 (4)
N(2)—C(1)	1.344 (2)	O(2)—C(2)	1.226 (2)
N(2)—H(2)	1.005 (4)	C(2)—C(2')	1.540 (2)
N(2)—H(3)	1.009 (4)		
C(1)—N(1)—H(4)	119.4 (3)	N(1)—C(1)—O(1)	120.1 (1)
C(1)—N(1)—H(5)	118.6 (2)	N(2)—C(1)—O(1)	121.2 (1)
H(4)—N(1)—H(5)	121.9 (3)	C(2)—O(3)—H(1)	113.1 (2)
C(1)—N(2)—H(2)	117.5 (2)	C(2')—C(2)—O(2)	120.7 (1)
C(1)—N(2)—H(3)	120.7 (3)	C(2')—C(2)—O(3)	113.8 (1)
H(2)—N(2)—H(3)	121.2 (3)	O(2)—C(2)—O(3)	125.5 (2)
N(1)—C(1)—N(2)	118.7 (1)		
X—H...Y	d(H...Y)	d(X...Y)	$\angle(X-H...Y)$
O(3)—H(1)...O(1)	1.416 (4)	2.479 (3)	168.9 (4)
N(1)—H(5)...O(1 ^{iv})	1.924 (4)	2.939 (2)	178.1 (4)
N(2)—H(2)...O(2)	2.016 (4)	2.999 (2)	165.5 (3)
N(2 ⁱⁱⁱ)—H(3 ⁱⁱⁱ)...O(3)	2.127 (4)	3.115 (2)	165.8 (3)
N(1 ^{iv})—H(4 ^{iv})...O(2)	1.900 (4)	2.896 (2)	173.0 (4)

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

and 3.6 mm and a thickness of 1.2 mm (volume *ca* 4.9 mm³) was cut from a larger one and mounted on an Al needle.

Neutron data were collected on a four-circle diffractometer at the HFR reactor at Petten at a temperature of 100.0 (1) K. Cooling device: liquid-He continuous-flow system (Herbert & Campbell, 1977). The wavelength used was 1.296 \AA monochromatized by the (220) planes of a Cu double-crystal monochromator; lattice parameters by fitting θ settings of 10 reflections in the θ range 25 to 41°. 1183 reflections were measured in $\omega/2\theta$ step-scan mode (0.0526° step⁻¹), $-6 \leq h \leq 6$, $0 \leq k \leq 15$, $0 \leq l \leq 8$. The scan width was 2.8° for reflections with $\theta < 45^\circ$ and (due to an increase in mosaicity on account of a failure in the liquid-helium unit and the subsequent heating up of the crystal) 3.1° for reflections with $45 < \theta < 57.5^\circ$ (max. θ). The reference reflections used were 0,12,0 and $\bar{2}04$ with a maximum intensity variation of 10%. Corrections using a scaling function

were performed according to Dam, Harkema & Feil (1983).

An absorption correction was applied with $\mu = 2.31 \text{ cm}^{-1}$, experimentally determined by transmission measurement on a crystal plate, with the aid of program ACXR (Harkema, 1978) to give minimum and maximum calculated transmission factors of 0.566 for the $\bar{1}02$ reflection and 0.817 for the 1,15,1 reflection. 99 multiply measured reflections were averaged ($R_{int} = 0.022$).

Full-matrix least-squares refinement (Busing, Martin & Levy, 1962) on F_o for 110 parameters [positional and anisotropic temperature parameters on all independent atoms, scale factor and an isotropic secondary-extinction correction (Larson, 1969)] resulted in an R factor of 0.045 ($wR = 0.035$), starting with values of Harkema, Bats, Weyenberg & Feil (1972). 895 unique reflections were used with $I > 2\sigma(I)$. The weighting scheme was $w = [\sigma(F_o)]^{-2}$, where $\sigma(F_o)$ for every reflection was calculated according to Hamilton (1964); scattering lengths from Koester & Yelon (1982); shift-to-e.s.d. ratio in final cycle < 0.01 ; largest correction for extinction (yF_c), $y = 0.68$. Final positional parameters are given in Table 1.* Intermolecular distances and hydrogen bonds are presented in Table 2, while Fig. 1 gives an overview of the structure in the $10\bar{2}$ plane.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53344 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

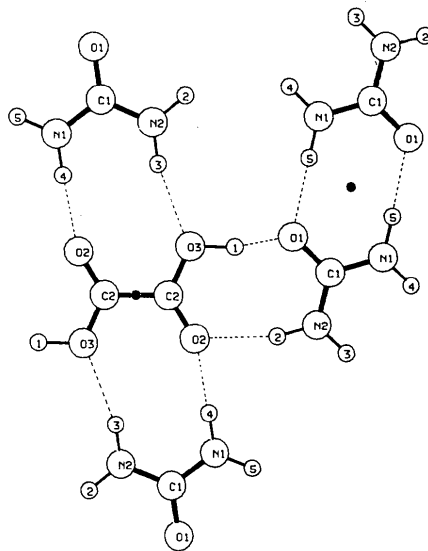


Fig. 1. View of the structure of urea-oxalic acid (2/1). Hydrogen bonds are indicated by dashed lines. The H atoms are labeled with digits only, ● denotes an inversion centre.

Related literature. Urea–oxalic acid (2/1) is the second known structure of complexes between urea and oxalic acid (Harkema, ter Brake & Helmholtz, 1979) and was studied in the framework of the accurate electron density of oxalic acid complexes (Coppens, 1984). The aim is to determine the accurate hydrogen positions in combination with the thermal parameters. Non-H-atom positions and thermal parameters are in good agreement with those obtained by a new X-ray diffraction experiment (Harkema, van Hummel, Peerdeman, Feil & Helmholtz, 1990).

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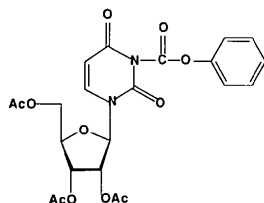
Structure of 2',3',5'-Tri-*O*-acetyl-*N*³-phenoxycarbonyluridine

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Abstract. C₂₂H₂₂N₂O₁₁, *M_r* = 490.41, monoclinic, *P*2₁, *a* = 9.870 (1), *b* = 15.421 (2), *c* = 8.059 (1) Å, β = 110.28 (1)°, *V* = 1150.6 (3) Å³, *Z* = 2, *D_x* = 1.42 g cm⁻³, Cu *Kα*, λ = 1.54184 Å, μ = 9.42 cm⁻¹, *F*(000) = 512, *T* = 293 K, *wR* = 0.057 for 2345 observed reflections with *I* > 3σ(*I*). The sugar conformation and puckering parameters are ³*E* (C3'-endo), *P* = 16.0° and τ_{*m*} = 38.8°. The C4'-C5' side-chain orientation is *gauche-gauche* [φ_{OO} = -57.5 (2); φ_{OC} = 59.7 (2)°]. The glycosidic torsion angle is -160.0 (2)° corresponding to the *anti* conformation. The dihedral angle between the pyrimidine and benzene rings is 15.1 (3)°.



Experimental. Crystal dimensions 0.40 × 0.30 × 0.25 mm. Enraf–Nonius CAD-4 diffractometer,

graphite-monochromated Cu *Kα* radiation. Cell dimensions from setting angles of 25 independent reflections with 47 ≤ θ ≤ 55°. ω–2θ scans. Reflections surveyed in the range 2 ≤ 2θ ≤ 150° (Cu *Kα*); -12 ≤ *h* ≤ 11, 0 ≤ *k* ≤ 19, 0 ≤ *l* ≤ 9; 2352 reflections were unique, 2345 observed with *I* > 3σ(*I*). Three reference reflections monitored periodically showed no significant variation in intensity. Absorption correction was not applied. Structure solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier-map recycling. Refinement using the *SDP* package (Frenz, 1985), full-matrix least-squares refinement on *F*, with non-H atoms having anisotropic temperature factors. Most of the H atoms were located from difference Fourier syntheses and were refined with isotropic temperature parameters. *w* = 4*F_o*²/[σ²(*I_o*) + (0.04*I_o*)²]^{1/2}/Lp, final *R* = 0.040, *wR* = 0.057, *S* = 2.22, maximum shift/e.s.d. in the final least-squares cycle 0.30, maximum peak in the final difference map 0.27 (4) e Å⁻³. Secondary-extinction coefficient refined (final value of 9.5 × 10⁻⁶). Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final fractional coordinates and