the five-membered rings may be described, following the notation of Altona & Sundaralingam (1972), as $^3T_2$ with O(4)-*endo* and C(14)-*exo* and $^3T_4$ with C(10)-*endo* and C(11)-*exo*. Atoms O(1) and O(6) are involved in intermolecular hydrogen bonding as indicated by the following parameters: O(1)$\cdots$O(6)$(x + 1,$ $y,$ $z) =$ 2·793 (8) Å, O(1)–H(O1) = 1·05715 Å, H(O1)$\cdots$O(6) = 1·779 (6) Å and angle O(1)–H(O1)–O(6) = 159·2 (3)°.

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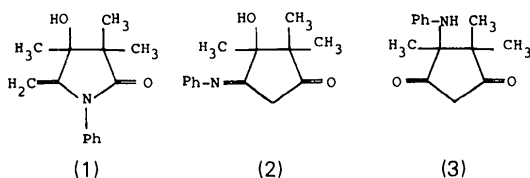
# Structure of 4-Hydroxy-3,3,4-trimethyl-5-methylene-1-phenyl-2-pyrrolidinone

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**Abstract.** $C_{14}H_{17}NO_2$, $M_r = 231\cdot3$, monoclinic, $P2_1/c$, $a = 7\cdot187$ (1), $b = 13\cdot531$ (1), $c = 13\cdot357$ (1) Å, $\beta = 93\cdot75$ (1)°, $V = 1296\cdot2$ (2) Å$^3$, $Z = 4$, $D_x = 1\cdot185$ Mg m$^{-3}$, $\lambda(Cu K\alpha) = 1\cdot54178$ Å, $\mu = 0\cdot599$ mm$^{-1}$, $F(000) = 496$, $T = 296$ K, $R = 0\cdot084$ for 1069 observed reflections. The five-membered ring has a conformation midway between envelope and twist and forms a dihedral angle with the planar six-membered ring of 83·4 (3)°. The distances and angles in the two rings are normal. The cohesion of the crystal is stabilized by hydrogen bonds, O$\cdots$O 2·750 (7) Å, O–H$\cdots$O 159°.

**Introduction.** In the reaction of the monoimine of diacetyl and aniline with the lithium enolate derived from methyl isobutyrate, in molar ratio 1:2 (Pérez-Ossorio, Alcaide, Plumet & López-Mardomingo, 1985), a compound was obtained that from analytical and spectroscopic data (IR, $^1$H NMR, $^{13}$C NMR and mass spectra) and on account of the mechanistic possibilities for the above reaction could be considered as either structure (1), (2) or (3).



(1)            (2)            (3)

The spectroscopic structural assignment becomes complex since both (2) and (3) can exist as enolic tautomers, some of which are favoured by intra-molecular chelation. Structure (1) is related to some enamides used as intermediates in some syntheses of various corrins such as vitamin $B_{12}$. The present investigation was undertaken to clarify which is the true structure.

**Experimental.** Colourless, prismatic, single crystal (0·06 × 0·09 × 0·14 mm) used for X-ray analysis. Cell parameters from setting angles of 53 reflections ($\theta < 40\cdot7$°) by least-squares refinement from a Philips PW 1100 computer-controlled four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Intensity measurement performed up to $\theta = 63$°, $\omega$–2$\theta$ scan technique; range of $hkl$ −9 to 9, 0 to 16 and 0 to 16. Two standard reflections (002, 00$\bar{2}$) measured every 90 min, no significant change in intensities. Total of 2093 independent reflections collected, 1069 for which $I > 2\sigma(I)$ used in further calculations; $R_{int} = 0\cdot014$. Lorentz–polarization factors applied but no absorption correction. Structure solved by direct methods using *MITHRIL*83 (Gilmore, 1983). $E$ map calculated from the set with the highest figure of merit revealed all non-H atoms. H atoms from a difference synthesis and included in the calculation with a fixed isotropic temperature factor. A further refinement cycle gave a final $R = 0\cdot084$, $wR = 0\cdot069$, $(\Delta/\sigma)_{max} = 0\cdot04$, $S =$

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j\cos(a_i,a_j).$$

|        | $x$         | $y$         | $z$          | $U_{eq}(\text{Å}^2 \times 10^3)$ |
|--------|-------------|-------------|--------------|------------|
| O(12)  | 0·9181 (6)  | 0·4498 (4)  | 0·2477 (4)   | 85 (2)     |
| O(16)  | 0·2717 (7)  | 0·5208 (3)  | 0·2260 (4)   | 71 (2)     |
| N(1)   | 0·6619 (7)  | 0·3837 (4)  | 0·1622 (4)   | 55 (2)     |
| C(2)   | 0·7522 (9)  | 0·4465 (5)  | 0·2284 (5)   | 59 (3)     |
| C(3)   | 0·6047 (8)  | 0·5151 (5)  | 0·2674 (5)   | 61 (3)     |
| C(4)   | 0·4244 (8)  | 0·4561 (5)  | 0·2476 (5)   | 56 (2)     |
| C(5)   | 0·4673 (9)  | 0·3927 (5)  | 0·1579 (5)   | 55 (2)     |
| C(6)   | 0·7589 (8)  | 0·3198 (6)  | 0·0962 (5)   | 59 (3)     |
| C(7)   | 0·7960 (10) | 0·2241 (6)  | 0·1242 (6)   | 73 (3)     |
| C(8)   | 0·8813 (12) | 0·1608 (7)  | 0·0607 (8)   | 95 (4)     |
| C(9)   | 0·9291 (11) | 0·1959 (9)  | −0·0296 (9)  | 98 (5)     |
| C(10)  | 0·8918 (12) | 0·2902 (9)  | −0·0592 (7)  | 93 (4)     |
| C(11)  | 0·8040 (10) | 0·3562 (7)  | 0·0038 (6)   | 79 (3)     |
| C(13)  | 0·6023 (14) | 0·6070 (7)  | 0·1974 (8)   | 84 (4)     |
| C(14)  | 0·6492 (14) | 0·5467 (8)  | 0·3747 (7)   | 84 (4)     |
| C(15)  | 0·3835 (13) | 0·3863 (7)  | 0·3344 (7)   | 74 (4)     |
| C(17)  | 0·3480 (13) | 0·3520 (7)  | 0·0927 (7)   | 77 (4)     |

Table 2. *Bond distances* (Å) *and angles* (°)

| O(12)—C(2)  | 1·204 (8)  | C(4)—C(5)    | 1·521 (9)  |
|-------------|------------|--------------|------------|
| O(16)—C(4)  | 1·419 (8)  | C(4)—C(15)   | 1·538 (11) |
| N(1)—C(2)   | 1·361 (8)  | C(5)—C(17)   | 1·304 (11) |
| N(1)—C(5)   | 1·401 (8)  | C(6)—C(7)    | 1·369 (11) |
| N(1)—C(6)   | 1·446 (9)  | C(6)—C(11)   | 1·387 (10) |
| C(2)—C(3)   | 1·526 (9)  | C(7)—C(8)    | 1·377 (13) |
| C(3)—C(4)   | 1·531 (9)  | C(8)—C(9)    | 1·361 (16) |
| C(3)—C(13)  | 1·554 (12) | C(9)—C(10)   | 1·358 (17) |
| C(3)—C(14)  | 1·510 (12) | C(10)—C(11)  | 1·405 (14) |

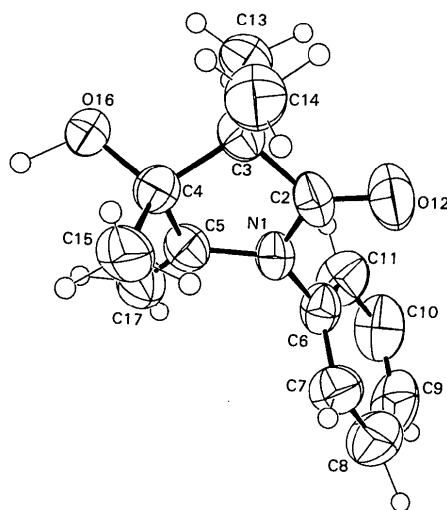| C(5)—N(1)—C(6)    | 123·2 (5) | O(16)—C(4)—C(15)   | 110·4 (6) |
|-------------------|-----------|--------------------|-----------|
| C(2)—N(1)—C(6)    | 122·8 (5) | O(16)—C(4)—C(5)    | 112·4 (5) |
| C(2)—N(1)—C(5)    | 113·9 (5) | C(5)—C(4)—C(15)    | 107·8 (6) |
| O(12)—C(2)—N(1)   | 125·9 (6) | N(1)—C(5)—C(4)     | 105·7 (5) |
| N(1)—C(2)—C(3)    | 106·8 (5) | C(4)—C(5)—C(17)    | 127·3 (7) |
| O(12)—C(2)—C(3)   | 127·2 (6) | N(1)—C(5)—C(17)    | 127·0 (7) |
| C(2)—C(3)—C(14)   | 112·8 (6) | N(1)—C(6)—C(11)    | 118·7 (7) |
| C(2)—C(3)—C(13)   | 105·2 (6) | N(1)—C(6)—C(7)     | 119·5 (6) |
| C(2)—C(3)—C(4)    | 102·9 (5) | C(7)—C(6)—C(11)    | 121·7 (7) |
| C(13)—C(3)—C(14)  | 109·8 (7) | C(6)—C(7)—C(8)     | 120·5 (8) |
| C(4)—C(3)—C(14)   | 115·8 (6) | C(7)—C(8)—C(9)     | 118·5 (9) |
| C(4)—C(3)—C(13)   | 109·8 (6) | C(8)—C(9)—C(10)    | 122·0 (9) |
| O(16)—C(4)—C(3)   | 110·3 (5) | C(9)—C(10)—C(11)   | 120·7 (9) |
| C(3)—C(4)—C(15)   | 113·1 (6) | C(6)—C(11)—C(10)   | 116·6 (8) |
| C(3)—C(4)—C(5)    | 102·5 (5) |                    |           |



Fig. 1. Perspective view of the molecule with atom labelling.

3·5; unit weights were used. Final difference Fourier map showed a maximum peak of 0·20 e Å$^{-3}$; largest hole −0·18 e Å$^{-3}$. Repeated attempts at crystallization under various conditions failed and the determination was carried out with the available crystals; the poor quality of these small crystals restricted the accuracy of the structure determination. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *PARST* (Nardelli, 1983a) on a VAX 11/750 computer.

**Discussion.** Final atomic parameters are given in Table 1.* The structure is shown in Fig. 1. The important bond distances and angles are given in Table 2. The six-membered ring does not deviate significantly from planarity [maximum deviation C(9) −0·008 (8) Å], while the five-membered ring deviates significantly, having a conformation midway between envelope and twist: $Q_2 = 0·284$ (7) Å, $\varphi_2 = 85$ (1)°, in terms of Cremer & Pople (1975) puckering parameters, and $\Delta C_s^4 = 0·0500$ (0·0032), $\Delta C_2^{N(1)} = 0·069$ (0·0017) and $\Delta C_2^2 = 0·087$ (0·0020), in terms of Nardelli (1983b) asymmetry parameters. The dihedral angle between the five- and six-membered rings is 83·4 (3)°. The distances and the angles in the phenyl ring and in the five-membered rings are normal. The cohesion of the crystal is stabilized by one hydrogen bond between O(16) and O(12), the O···O distance being 2·750 (7) Å and the O—H···O angle 159°.

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