

The intermolecular hydrogen-bond distances $O(1)\cdots H(1)O(3)$ and $O(1')\cdots H(1)O(3)$ are 1.70 and 2.04 Å, for the two disordered positions, respectively. The corresponding $O(1)\cdots O(3)$ and $O(1')\cdots O(3)$ distances are 2.75 and 2.98 Å while the $C(15)\cdots H(1)O(3)$ close contact is 2.82 Å.

Conversion of this reductive alkylation product to natural products of possible medicinal interest is currently under way to demonstrate the utility of this reductive alkylation technique.

The authors acknowledge financial support of the Research Institute of Pharmaceutical Sciences of the University of Mississippi in the form of a graduate stipend (ARJ).

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Acta Cryst. (1991). **C47**, 1678–1680

Structure of *trans,endo*-1-Phenyl-2-(4-methoxyphenyl)decahydroquinolin-4-one

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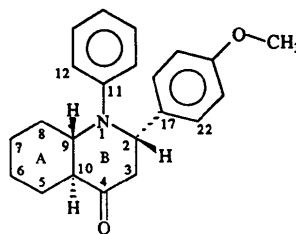
(Received 19 November 1990; accepted 18 December 1990)

Abstract. $C_{22}H_{25}NO_2$, $M_r = 335.45$, monoclinic, $P2_1/c$, $a = 15.306$ (7), $b = 6.779$ (6), $c = 18.004$ (6) Å, $\beta = 91.29$ (2)°, $V = 1867$ (3) Å³, $Z = 4$, $D_x = 1.193$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 7.06$ mm⁻¹, $F(000) = 720$, room temperature, final $R = 0.061$ for 1106 observed reflections [$I > 3\sigma(I)$]. Both the cyclohexane ring and the heterocycle adopt a chair conformation. The N-atom geometry is tetrahedral rather than square planar. The X-ray data provide precise information in good agreement with previously obtained data by ¹H NMR.

Introduction. The determination of the structure of the decahydroquinolin-4-one is necessary to understand its reactivity. For the *trans*-ring-fused 1-phenyl-2-(*p*-methoxyphenyl)decahydroquinolin-4-one, the ¹H NMR data permit the determination of the *trans* relationship between C(9)—H(9) and C(10)—H(10) bonds both of which occupy an axial position in the cyclohexane ring and moreover

indicate a quasi-equatorial or equatorial position for the C(2) phenyl group depending on the *B* ring conformation.

Unambiguous assignment of its structure has to be obtained from a single-crystal X-ray structure analysis.



(1)

The preparation and the study of the reactivity of this compound will be reported further.

Experimental. Crystals suitable for X-ray analysis obtained by slow evaporation from ethyl acetate. Crystal size $0.60 \times 0.40 \times 0.25$ mm. Nonius CAD-4 diffractometer with graphite monochromator; cell parameters measured with 25 reflections in the θ range $8\text{--}12^\circ$; three standard reflections (038, 038, 234) measured every 60 min, loss of 1.4% in 37 h, linearly corrected; scans by $\omega/2\theta$ method, ω -scan width: $(0.8 + 0.35\tan\theta)^\circ$. Data collection for $1 < \theta < 21^\circ$ in index range $-15 \leq h \leq 15$, $0 < k < 6$, $0 < l < 18$. 2311 reflections collected, 2001 unique ($R_{\text{int}} = 0.018$), 1106 with $I > 3\sigma(I)$. Lorentz-polarization corrections applied but non for absorption. Structure solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Heavy atoms were refined with anisotropic thermal parameters, H atoms included using a riding model (C—H 0.95 Å, $B = 6 \text{ \AA}^2$). Full-matrix least-squares refinement based on F_o function minimized $\sum w(\Delta F)^2$ with $w = 1/(\sigma F)^2 = 4F^2/[\sigma I^2 + (pF^2)^2]^{1/2}$; a secondary-extinction parameter g was refined (final value 1.1×10^{-6}); final R values: $R_F = 0.061$, $wR_F = 0.063$, $w = 1$, $S = 1.61$; max. shift/e.s.d. = 0.01, highest peak in the final difference Fourier map = 0.3 e \AA^{-3} . MicroVAX II computer with programs from *SDP* (Frenz, 1985); scattering factors and anomalous components f' , f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1).

Discussion. Final atomic parameters are listed in Table 1; bond lengths, bond angles and selected torsion angles in Table 2.* Fig. 1 shows the molecule and the atom-numbering scheme.

The *A* and *B* rings have a chair conformation as can be seen from either the torsion angles or the distances N(1), C(2), C(3), C(4), C(5), C(6), C(7), C(8), C(9) and C(10) to their least-squares plane [-0.188 (5), 0.280 (6), -0.254 (7), 0.139 (7),

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53850 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

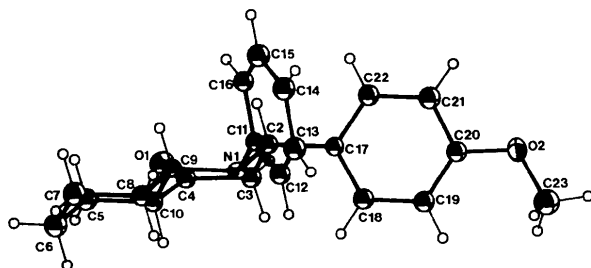


Fig. 1. ORTEP (Johnson, 1976) drawing of the molecular structure and atom-numbering scheme.

Table 1. Positional parameters and equivalent isotropic thermal parameters (\AA^2)

$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
O(1)	0.0292 (2)	0.7715 (7)	-0.0017 (3)	4.9 (1)*
O(2)	-0.4170 (3)	0.6047 (8)	-0.2942 (3)	5.9 (1)*
N(1)	-0.2200 (3)	0.6012 (7)	0.0245 (3)	3.1 (1)*
C(2)	-0.1773 (4)	0.587 (1)	-0.0488 (3)	3.5 (1)
C(3)	-0.1135 (4)	0.761 (1)	-0.0569 (3)	4.3 (1)
C(4)	-0.0490 (4)	0.767 (1)	0.0068 (3)	3.8 (1)
C(5)	-0.0232 (4)	0.764 (1)	0.1457 (3)	4.9 (2)
C(6)	-0.0701 (5)	0.772 (1)	0.2201 (4)	5.7 (2)
C(7)	-0.1357 (4)	0.605 (1)	0.2266 (4)	5.3 (2)
C(8)	-0.2017 (4)	0.610 (1)	0.1616 (3)	4.2 (1)
C(9)	-0.1549 (4)	0.596 (1)	0.0872 (3)	3.5 (1)
C(10)	-0.0911 (4)	0.770 (1)	0.0815 (3)	3.7 (1)
C(11)	-0.2838 (4)	0.4439 (9)	0.0325 (3)	3.1 (1)
C(12)	-0.3689 (4)	0.492 (1)	0.0436 (4)	4.5 (2)
C(13)	-0.4318 (5)	0.344 (1)	0.0555 (4)	5.2 (2)
C(14)	-0.4069 (5)	0.150 (1)	0.0546 (4)	5.3 (2)
C(15)	-0.3209 (4)	0.101 (1)	0.0431 (4)	5.2 (2)
C(16)	-0.2589 (4)	0.248 (1)	0.0320 (3)	4.2 (1)
C(17)	-0.2450 (4)	0.591 (1)	-0.1119 (3)	3.5 (1)
C(18)	-0.3052 (4)	0.742 (1)	-0.1192 (3)	4.3 (1)
C(19)	-0.3644 (4)	0.750 (1)	-0.1787 (3)	4.5 (1)
C(20)	-0.3625 (4)	0.608 (1)	-0.2325 (4)	4.2 (1)
C(21)	-0.3039 (4)	0.455 (1)	-0.2272 (4)	5.0 (2)
C(22)	-0.2451 (4)	0.446 (1)	-0.1669 (3)	4.4 (2)
C(23)	-0.4811 (5)	0.754 (1)	-0.3007 (4)	6.7 (2)

* Atoms refined anisotropically.

Table 2. Bond lengths (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$)

N(1)—C(2)	1.489 (6)	C(9)—C(10)	1.537 (7)
N(1)—C(9)	1.489 (6)	C(10)—C(5)	1.537 (7)
N(1)—C(11)	1.456 (6)	C(5)—C(6)	1.534 (7)
C(2)—C(3)	1.539 (7)	C(6)—C(7)	1.520 (8)
C(2)—C(17)	1.520 (7)	C(7)—C(8)	1.530 (7)
C(3)—C(4)	1.497 (7)	C(8)—C(9)	1.537 (7)
O(1)—C(4)	1.210 (5)	C(4)—C(10)	1.505 (7)
C(17)—C(18)	1.383 (7)	C(11)—C(12)	1.362 (7)
C(18)—C(19)	1.388 (7)	C(12)—C(13)	1.407 (8)
C(19)—C(20)	1.370 (7)	C(13)—C(14)	1.371 (8)
C(20)—C(21)	1.369 (8)	C(14)—C(15)	1.378 (7)
C(21)—C(22)	1.395 (7)	C(15)—C(16)	1.394 (8)
C(22)—C(17)	1.393 (7)	C(16)—C(11)	1.382 (7)
O(2)—C(20)	1.375 (6)	O(2)—C(23)	1.410 (7)
C(11)—N(1)—C(2)	110.5 (4)	N(1)—C(2)—C(17)	110.9 (4)
C(2)—N(1)—C(9)	111.8 (4)	N(1)—C(2)—C(3)	109.2 (4)
C(11)—N(1)—C(9)	110.3 (4)	C(3)—C(2)—C(17)	109.9 (4)
O(1)—C(4)—C(3)	122.8 (5)	C(4)—C(10)—C(9)	109.6 (4)
O(1)—C(4)—C(10)	123.9 (5)	C(4)—C(10)—C(5)	112.1 (4)
C(3)—C(4)—C(10)	113.4 (4)	C(5)—C(10)—C(9)	110.5 (5)
N(1)—C(9)—C(8)	110.0 (4)	C(2)—C(3)—C(4)	111.0 (5)
N(1)—C(9)—C(10)	110.3 (4)	C(10)—C(5)—C(6)	109.5 (5)
C(8)—C(9)—C(10)	108.7 (4)	C(5)—C(6)—C(7)	111.3 (5)
C(20)—O(2)—C(23)	117.7 (5)	C(6)—C(7)—C(8)	110.6 (5)
		C(7)—C(8)—C(9)	110.7 (5)
N(1)—C(2)—C(3)—C(4)	55.1 (6)	C(10)—C(5)—C(6)—C(7)	56.8 (8)
C(2)—C(3)—C(4)—C(10)	-54.1 (7)	C(5)—C(6)—C(7)—C(8)	-56.5 (7)
C(3)—C(4)—C(10)—C(9)	53.7 (7)	C(6)—C(7)—C(8)—C(9)	57.7 (7)
C(4)—C(10)—C(9)—N(1)	-55.5 (6)	C(7)—C(8)—C(9)—C(10)	-59.1 (7)

0.227 (7), -0.240 (8), 0.244 (7), -0.224 (7), 0.278 (6) and -0.263 (7) Å, respectively]. The relative configuration of C(2)—H(2) and C(9)—H(9) is that of a *cis* (*endo*) relationship, the distances to the least-squares

plane previously defined of the atoms H(2) and H(9) being 1.230 (7) and 1.228 (8) Å respectively. On the other hand, the distances of the atoms H(9) and H(10) to this plane, 1.228 (8) and -1.212 (7) Å respectively, confirm the *trans* relationship between the C(9)—H(9) and C(10)—H(10) bonds. The C(2) phenyl group occupies an equatorial position as shown by the distance of C(17) to this least-squares plane of -0.194 (6) Å.

The N-atom geometry is tetrahedral rather than planar. The position of the N(1) phenyl plane is given by the torsion angle C(2)—N(1)—C(11)—

C(12) of -120.4 (6)°. The dihedral angle between the N(1) phenyl ring and the C(2) phenyl ring is 68 (1)°.

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Acta Cryst. (1991). C47, 1680–1683

Structure of the 4-Oxo-2-butenic Acid Alkyl Ester Moiety. III. Structures of Diethyl *N,N'*-(Ethylenediamino)bis(4-oxo-2-butenate) and Propyl 4-Oxo-4-ureido-2-butenate

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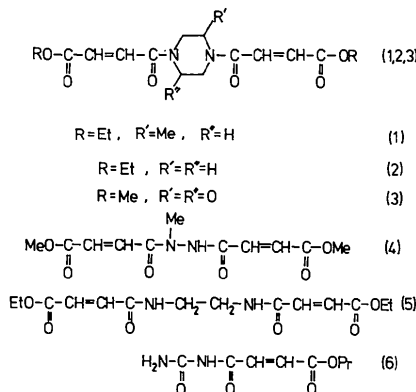
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(Received 17 August 1990; accepted 17 December 1990)

Abstract. (5), C₁₄H₂₀N₂O₆, *M_r* = 312.3, monoclinic, *P*2₁/*c*, *a* = 11.348 (1), *b* = 4.854 (1), *c* = 14.629 (1) Å, β = 99.87 (1)°, *V* = 793.9 Å³, *Z* = 2, *D_x* = 1.306 Mg m⁻³, λ(Cu Kα) = 1.54178 Å, Ni filter, μ = 0.83 mm⁻¹, *F*(000) = 332, *T* = 293 K, *R* = 0.047 for 755 reflections. (6), C₈H₁₂N₂O₄, *M_r* = 200.2, orthorhombic, *Pccn*, *a* = 39.365 (2), *b* = 10.147 (1), *c* = 9.937 (1) Å, *V* = 3968.9 Å³, *Z* = 16, *D_x* = 1.340 Mg m⁻³, λ(Cu Kα) = 1.54178 Å, Ni filter, μ = 0.93 mm⁻¹, *F*(000) = 1696, *T* = 293 K, *R* = 0.043 for 2119 reflections. The conjugated 4-amino-4-oxo-2-butenate fragments are approximately planar, with the ester group *anti* to the double bond. Amide type NH...O hydrogen bonds join molecules into chains in the first structure. Crystals of the urea derivative contain a complex hydrogen-bonding network which includes an intramolecular hydrogen bond.

Introduction. In our studies of alkyl 4-oxo-2-butenate structures (Główka & Iwanicka, 1990; Główka, Iwanicka & Najman, 1991) we try to analyse conformation and to estimate the flexibility of the chain fragment with a view to attaining a better understanding of the potential cytostatic activity shown by similar compounds. The most pronounced inhibition of transplantable neoplasms L1210, P388 and Sa180 is shown by 4,4'-(2-methyl-1,4-piperazinediyl)bis(4-oxo-2-butenic acid diethyl ester) (1) (Graczyk, Pakulska, Groszkowski &

Najman, 1980; Groszkowski & Najman, 1983) (see Scheme). As part of these studies we now describe the structures of the linear compounds (5) and (6).



Experimental. (5), (6). Data collection: CAD-4, ω/2θ scan, θ_{max} = 75°, three standards monitored every hour, absorption correction according to Walker & Stuart (1983), structure solution by direct methods, full-matrix least squares (non-H atoms anisotropic, H atoms isotropic), function minimized Σw(|*F_o* - *F_c*)²; atomic scattering factors from analytical approximation in *SHELX76*; programs used: *SHELX76* (Sheldrick, 1976), *SHELXS86* (Sheldrick, 1986) and *ORTEP* (Johnson, 1976).