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Structural Investigation of Dihydrooxazinones

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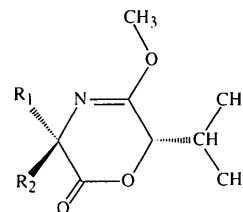
Abstract

The crystal structures of six dihydrooxazinones, (6*S*,3*R*,1'*R*)-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-3-(3'-oxo-1'-phenylbutyl)-2*H*-1,4-oxazin-2-one, C₁₉H₂₅NO₄, (6*S*,3*R*,1'*R*)-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-3-(3'-oxo-1',3'-diphenylpropyl)-2*H*-1,4-oxazin-2-one, C₂₄H₂₇NO₄, (6*S*,3*R*,1'*R*)-3-(1'-cyclohexyl-3'-oxobutyl)-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one, C₁₉H₃₁NO₄, (6*S*,3*S*,1'*R*)-3-[(diethylammonio)(phenyl)methyl]-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one chloride, C₂₀H₃₁N₂O₃⁺.Cl⁻, (6*S*,3*R*,1'*S*)-3,6-dihydro-3-[(hydroxy)(phenyl)methyl]-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one, C₁₆H₂₁NO₄, (6*S*,3*S*,1'*S*)-3,6-dihydro-3-[(hydroxy)(phenyl)methyl]-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one, C₁₆H₂₁NO₄, were determined, and represent the first crystal structures reported containing this type of heterocycle. The geometrical parameters of the heterocycle and the preferred conformations of the substituents are discussed. The characteristic features of the dihydrooxazinones are compared with those of bis-lactim ethers and lactides. An explanation is given for the appearance of the folded conformation by which an aromatic ring shields the heterocycle.

Comment

Dihydrooxazinones play an important role in the course of the asymmetric synthesis of amino acids (Schöllkopf, 1983; Maywald, 1987). The conformation of the heterocycle and the position of side chains, with respect to the heterocycle, are decisive for reactivity and selectivity during the synthesis. In order to obtain a deeper insight into the reaction processes, the crystal structures of six dihydrooxazinones, a type of heterocycle for which no entry exists in the Cambridge Structural Database (version 5.08, October 1994; Allen & Kennard, 1993), were determined. Their structural parameters were compared with those of two similar heterocycles: lactides and bis-lactim ethers; the dihydrooxazinone can be regarded as

a hybrid between these two. All compounds were available as pure enantiomers for which the absolute configuration at C(6) was known from the chemical synthesis.



R ₁	R ₂
(1) —CH ₃	—CH ₂ CH ₂ COCH ₃ Ph
(2) —CH ₃	—CH ₂ CH ₂ COPh Ph
(3) —CH ₃	—CH ₂ CH ₂ COCH ₃ C ₆ H ₁₁
(4) —CH ₂ ^{Ph} N ⁺ (CH ₂ CH ₃) ₂ H ⁺ Cl ⁻	—CH ₃ OH Ph
(5) —CH ₃	—CH ₂ CH ₂ COCH ₃ Ph
(6) —CH ₂ ^{OH} Ph	—CH ₃

(I)

The dihydrooxazinone heterocycle of (6*S*,3*R*,1'*R*)-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-3-(3'-oxo-1'-phenylbutyl)-2*H*-1,4-oxazin-2-one, (1) (Fig. 1), adopts a nearly planar conformation [$Q = 0.099$ Å, $\varphi = 126.9$, $\theta = 86.5^\circ$ (Cremer & Pople, 1975)]. The molecule exhibits the so-called folded conformation by which an aromatic residue shields the heterocycle. This interesting phenomenon was not only found for diketopiperazines in solution (Kopple & Marr, 1967; Kopple & Onishi, 1969) and in the solid state (*e.g.* Lin & Webb, 1973), but also for hydantoines (Fujiwara, Bose, Manhas & van der Veen, 1979) and 1,4-dihydropyridines (Iwasaki, Watanabe & Maeda, 1987). The angle between the phenyl ring and the heterocycle is $47.0(1)^\circ$.

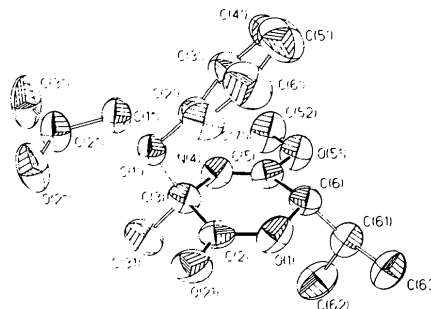


Fig. 1. Perspective view of (1) with the atom numbering and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

The exchange of the terminal methyl group by a phenyl ring leads to (6*S*,3*R*,1'*R*)-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-3-(3'-oxo-1',3'-diphenylpropyl)-2*H*-1,4-oxazin-2-one, (2) (Fig. 2), but this does not markedly alter the conformation of the rest of the molecule. The heterocycle is again nearly planar [$Q = -0.138 \text{ \AA}$, $\varphi = 135.2$, $\theta = 85.0^\circ$ (Cremer & Pople, 1975)] and the angle between it and the shielding phenyl ring is $46.0(1)^\circ$.

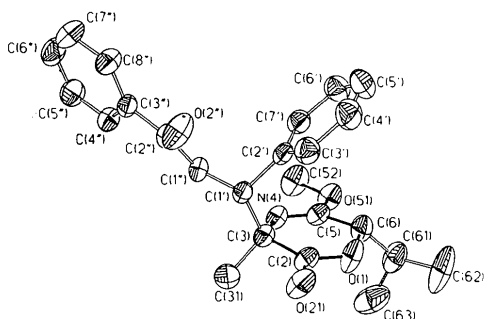


Fig. 2. Perspective view of (2) with the atom numbering and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

In order to discover whether non-aromatic substituents show a similar tendency to shield the heterocycle, the phenyl ring of (1) was replaced by a cyclohexyl residue to give (6*S*,3*R*,1'*R*)-3-(1'-cyclohexyl-3'-oxobutyl)-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one, (3). The result is shown in Fig. 3. Compound (3) crystallizes with two molecules in the asymmetric unit, which differ significantly only in the angular position of the cyclohexyl group [$C(3)-C(1')-C(2')-C(3')$ $79.8(3)$ and $93.0(3)^\circ$]. Slight steric interactions between the cyclohexane and the planar dihydrooxazinone rings [$Q = -0.041$ and -0.068 \AA , $\varphi = 297.5$ and 147.1° , $\theta = 71.6$ and 74.5° , respectively for each molecule (Cremer & Pople, 1975)] lead to a modest increase of the angle $C(3)-C(1')-C(2')$ [$114.2(2)$ and $112.2(2)^\circ$] compared to the angles of $111.0(2)$ in (1) and $111.1(2)^\circ$ in (2).

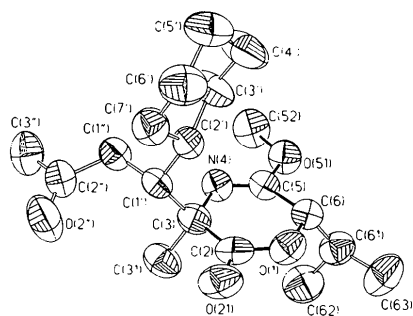


Fig. 3. Perspective view of molecule A of (3) with the atom numbering and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

In contrast to these three structures, in (6*S*,3*S*,1'*R*)-3-[(diethylammonio)(phenyl)methyl]-3,6-dihydro-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one chloride, (4) (Fig. 4), the bulkiest substituents lie on the same side of the heterocycle which, therefore, adopts a flattened-boat conformation with O(1), C(2), N(4) and C(5) in the plane [$Q = 0.304 \text{ \AA}$, $\varphi = 127.5$, $\theta = 89.8^\circ$ (Cremer & Pople, 1975)] while both C(3) and C(6) deviate above the plane by 0.26 \AA . The Cl^- ion is bound via a hydrogen bond to the diethylammonium group [$\text{H(N)} \cdots \text{Cl}$ $2.21(5)$, $\text{N(1'')} \cdots \text{Cl}$ $3.066(4) \text{ \AA}$, $\text{N(1'')} - \text{H(N)} \cdots \text{Cl}$ $158(4)^\circ$].

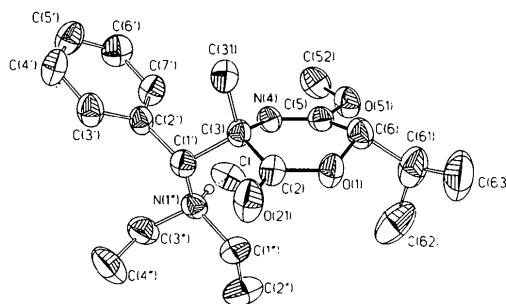


Fig. 4. Perspective view of (4) with the atom numbering and displacement ellipsoids at the 50% probability level. H atoms, except H(N), have been omitted for clarity.

The most interesting structures in this series are the diastereoisomers (6*S*,3*R*,1'*S*)-3,6-dihydro-3-[(hydroxy)(phenyl)methyl]-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one, (5) (Fig. 5), and (6*S*,3*S*,1'*S*)-3,6-dihydro-3-[(hydroxy)(phenyl)methyl]-6-isopropyl-5-methoxy-3-methyl-2*H*-1,4-oxazin-2-one, (6) (Fig. 6). Both heterocycles appear in a flattened-boat conformation with O(1), C(2), N(4) and C(5) forming the base [for (5), $Q = -0.188 \text{ \AA}$, $\varphi = 141.7$, $\theta = 82.3^\circ$; for (6), $Q = 0.183 \text{ \AA}$, $\varphi = 122.1$, $\theta = 87.2^\circ$ (Cremer & Pople, 1975)]. The difference in the chirality of C(3) leads to very different conformations. While (6) adopts a folded conformation, the phenyl ring in (5) is extended towards the N atom of the heterocycle. As a result of this the hydroxy groups form different hydrogen bonds. While in (5) there is an intermolecular hydrogen bond [$\text{H(1'O)} \cdots \text{O(21)}$ $2.26(2)$, $\text{O(1')} \cdots \text{O(21)}$ $3.07(1) \text{ \AA}$, $\text{O(1')} - \text{H(1'O)} \cdots \text{O(21)}$ $159(4)^\circ$; symmetry code: (i) $2 - x, y - 0.5, 1 - z$], in (6) there is an intramolecular attraction between the hydroxy H atom and N(4) [$\text{H(1'O)} \cdots \text{N(4)}$ $2.32(4)$, $\text{O(1')} \cdots \text{N(4)}$ $2.790(4) \text{ \AA}$, $\text{O(1')} - \text{H(1'O)} \cdots \text{N(4)}$ $115(4)^\circ$]. The *cis* arrangement of the phenyl ring and the isopropyl group in (6) results in an increased $C(3)-C(1')-C(2')$ bond angle of $113.7(2)^\circ$ [compared to $111.0(2)$ in (1) and $111.1(2)^\circ$ in (2)] and a larger angle between the aromatic ring and the heterocycle of $72.0(1)^\circ$. The distance between the two ring centres is 4.034 \AA , compared with 3.632 in (1) and 3.618 \AA in (2).

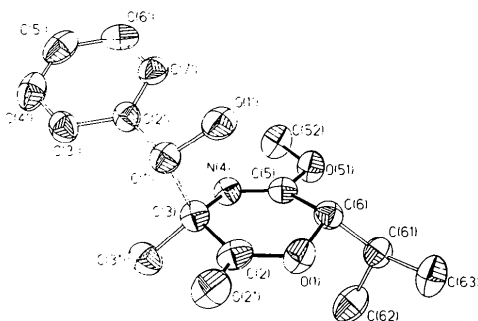


Fig. 5. Perspective view of (5) with the atom numbering and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

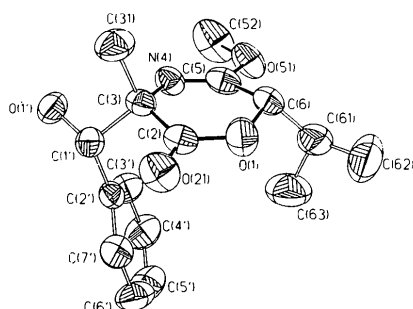


Fig. 6. Perspective view of (6) with the atom numbering and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

Average values for bond lengths and angles of the dihydrooxazinone ring show very small standard deviations (Table 7). In each compound the methoxy group lies in the plane of the heterocycle. The rigidity of the C=N double bond prevents this type of heterocycle from adopting a well pronounced boat conformation, as in lactides (Bolte, Beck, Nieger & Egert, 1994). As a result of forcing the carboxylic ester group into a common plane with the lactim ether moiety, the intra-annular angles C(6)—O(1)—C(2) and O(1)—C2—C(3) are widened, while the extra-annular angles O(1)—C(2)—O(21) and C(3)—C(2)—O(21) are decreased compared to those of pure lactides.

The geometric parameters of the lactim-ether moiety are in excellent agreement with those of the five published bis-lactim ethers (Schöllkopf, Grüttner, Anderskewitz, Egert & Dyrbusch, 1987; Schöllkopf, Hupfeld, Küper, Egert & Dyrbusch, 1988; Schöllkopf, Kühnle, Egert & Dyrbusch, 1987; Schöllkopf, Pettig, Busse, Egert & Dyrbusch, 1986; Schöllkopf, Pettig, Schulze, Klinge, Egert, Benecke & Noltemeyer, 1988).

The isopropyl group adopts nearly the same conformation in each of the six structures. The torsion angle

C(5)—C(6)—C(61)—H(61) has a mean value of $60(2)^\circ$ and the average H(61)···O(51) distance is $2.51(4) \text{ \AA}$. The isopropyl group prefers an arrangement where the small H atom is in the direct vicinity of the methoxy O atom, a conformational preference also found in the bis-lactim ethers. A similar arrangement was found for lactides (Mathieson & Taylor, 1963; Bolte *et al.*, 1994) where a tertiary C atom takes a position in which its attached H atom is in close proximity to the carbonyl O atom of the heterocycle. This conformation was not only found for the isopropyl group but also for the H atom, H(1'), in all of the six dihydrooxazinones. The mean of the absolute value of the torsion angle C(2)—C(3)—C(1')—H(1') is $56(10)^\circ$ and the mean H(1')···O(21) distance is $2.6(1) \text{ \AA}$.

This preferred arrangement helps us to understand the conformation of the molecules without regarding attractive interactions between the heterocycle and the aromatic residue. Whenever a tertiary C atom is attached to the heterocycle the H atom is found in the vicinity of the carbonyl O atom. In spite of some small distortions [widening of the angle C(3)—C(1')—C(2')] even (6) adopts this conformation.

Experimental

Compounds (1), (3) and (6) were crystallized from cyclohexane, (2) from cyclohexane/2-propanol, (4) from acetone/cyclohexane and (5) from cyclohexane/ether.

Compound (1)

Crystal data

C₁₉H₂₅NO₄
M_r = 331.4
 Orthorhombic
*P*2₁2₁2₁
a = 8.863 (1) Å
b = 13.429 (1) Å
c = 15.791 (1) Å
V = 1879.5 (5) Å³
Z = 4
D_x = 1.171 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 40 reflections
 θ = 10–12.5°
 μ = 0.08 mm⁻¹
T = 293 K
 Block
 0.8 × 0.5 × 0.4 mm
 Colourless

Data collection

Stoe Siemens four-circle diffractometer
 $\omega/2\theta$ profile fitting (Clegg, 1981)
 Absorption correction: none
 1900 measured reflections
 1900 independent reflections
 1717 observed reflections [*F* > 3σ(*F*)]

θ_{\max} = 25°
h = -10 → 10
k = 0 → 15
l = -16 → 18
 3 standard reflections monitored every 100 reflections
 intensity decay: negligible

Refinement

Refinement on *F*
R = 0.041

$\Delta\rho_{\max}$ = 0.13 e Å⁻³
 $\Delta\rho_{\min}$ = -0.12 e Å⁻³

$wR = 0.045$
 $S = 1.42$
 1717 reflections
 233 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F) + 0.0005F^2]$
 $(\Delta/\sigma)_{\max} = 0.001$

Extinction correction: see
 below
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

$wR = 0.037$
 $S = 1.42$
 1782 reflections
 275 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F) + 0.0005F^2]$
 $(\Delta/\sigma)_{\max} = 0.07$

Extinction correction: see
 below
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (1)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(1)	0.2749 (2)	0.7718 (1)	0.6580 (1)	0.065 (1)
C(2)	0.1268 (3)	0.7750 (2)	0.6738 (1)	0.053 (1)
C(3)	0.0552 (3)	0.8695 (2)	0.7082 (2)	0.053 (1)
N(4)	0.1527 (2)	0.9564 (1)	0.7121 (1)	0.056 (1)
C(5)	0.2904 (3)	0.9468 (2)	0.6974 (2)	0.053 (1)
C(6)	0.3753 (3)	0.8546 (2)	0.6728 (2)	0.052 (1)
O(21)	0.0537 (2)	0.7008 (1)	0.6595 (1)	0.073 (1)
C(31)	-0.0769 (4)	0.8960 (2)	0.6501 (2)	0.080 (1)
O(51)	0.3882 (2)	1.0236 (1)	0.7014 (1)	0.072 (1)
C(52)	0.3225 (4)	1.1205 (2)	0.7179 (3)	0.095 (1)
C(61)	0.4703 (3)	0.8674 (2)	0.5934 (2)	0.063 (1)
C(62)	0.3769 (4)	0.9054 (3)	0.5207 (2)	0.090 (1)
C(63)	0.5523 (4)	0.7715 (2)	0.5700 (2)	0.079 (1)
C(1')	-0.0009 (2)	0.8436 (2)	0.7993 (2)	0.050 (1)
C(2')	0.1272 (2)	0.8044 (2)	0.8534 (1)	0.051 (1)
C(3')	0.2288 (3)	0.8669 (2)	0.8942 (2)	0.064 (1)
C(4')	0.3475 (3)	0.8285 (3)	0.9409 (2)	0.082 (1)
C(5')	0.3679 (4)	0.7272 (3)	0.9470 (2)	0.101 (1)
C(6')	0.2680 (4)	0.6651 (2)	0.9082 (2)	0.095 (1)
C(7')	0.1488 (3)	0.7019 (2)	0.8620 (2)	0.068 (1)
C(1'')	-0.0851 (3)	0.9309 (2)	0.8409 (2)	0.061 (1)
O(2'')	-0.3205 (2)	0.8584 (1)	0.8022 (2)	0.094 (1)
C(2'')	-0.2542 (3)	0.9272 (2)	0.8340 (2)	0.069 (1)
C(3'')	-0.3369 (3)	1.0149 (2)	0.8700 (3)	0.110 (2)

Compound (2)

Crystal data

$\text{C}_{24}\text{H}_{27}\text{NO}_4$
 $M_r = 393.5$
 Monoclinic
 $P2_1$
 $a = 8.363 (1) \text{\AA}$
 $b = 10.453 (1) \text{\AA}$
 $c = 12.270 (1) \text{\AA}$
 $\beta = 94.91 (1)^\circ$
 $V = 1068.7 (3) \text{\AA}^3$
 $Z = 2$
 $D_x = 1.223 \text{ Mg m}^{-3}$

Data collection

Stoe Siemens four-circle
 diffractometer
 $\omega/2\theta$ profile fitting (Clegg,
 1981)
 Absorption correction:
 none
 1989 measured reflections
 1900 independent reflections
 1782 observed reflections
 $[F > 3\sigma(F)]$

Refinement

Refinement on F
 $R = 0.034$

Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{\AA}$
 Cell parameters from 50
 reflections
 $\theta = 10\text{--}12.5^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Block
 $0.9 \times 0.6 \times 0.3 \text{ mm}$
 Colourless

$R_{\text{int}} = 0.031$
 $\theta_{\max} = 25^\circ$
 $h = -10 \rightarrow 3$
 $k = 0 \rightarrow 14$
 $l = 0 \rightarrow 18$
 3 standard reflections
 monitored every 100
 reflections
 intensity decay: negligible

$\Delta\rho_{\max} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.12 \text{ e \AA}^{-3}$

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(1)	0.8677 (2)	0.3657 (2)	0.0963 (1)	0.058 (1)
C(2)	0.7449 (3)	0.3219 (2)	0.1479 (2)	0.043 (1)
C(3)	0.6841 (3)	0.3978 (2)	0.2423 (2)	0.038 (1)
N(4)	0.7560 (2)	0.5233 (2)	0.2620 (1)	0.042 (1)
C(5)	0.8760 (3)	0.5548 (2)	0.2136 (2)	0.043 (1)
C(6)	0.9585 (3)	0.4784 (2)	0.1306 (2)	0.049 (1)
O(21)	0.6835 (2)	0.2237 (2)	0.1153 (1)	0.057 (1)
C(31)	0.5029 (3)	0.4157 (3)	0.2143 (2)	0.057 (1)
O(51)	0.9543 (2)	0.6657 (2)	0.2324 (1)	0.058 (1)
C(52)	0.8979 (5)	0.7454 (3)	0.3164 (3)	0.075 (1)
C(61)	0.9912 (4)	0.5529 (3)	0.0283 (2)	0.064 (1)
C(62)	1.0795 (6)	0.4687 (4)	-0.0488 (3)	0.109 (2)
C(63)	0.8408 (5)	0.6084 (4)	-0.0273 (3)	0.091 (1)
C(1')	0.7186 (3)	0.3134 (2)	0.3472 (2)	0.036 (1)
C(2')	0.8966 (3)	0.2820 (2)	0.3668 (2)	0.036 (1)
C(3')	0.9524 (3)	0.1628 (2)	0.3375 (2)	0.047 (1)
C(4')	1.1158 (3)	0.1341 (3)	0.3499 (2)	0.059 (1)
C(5')	1.2231 (3)	0.2241 (3)	0.3906 (2)	0.062 (1)
C(6')	1.1709 (3)	0.3423 (3)	0.4211 (2)	0.056 (1)
C(7')	1.0082 (3)	0.3713 (2)	0.4096 (2)	0.044 (1)
C(1'')	0.6518 (3)	0.3701 (2)	0.4484 (2)	0.039 (1)
C(2'')	0.6430 (3)	0.2730 (2)	0.5388 (2)	0.039 (1)
O(2'')	0.6682 (3)	0.1602 (2)	0.5231 (1)	0.062 (1)
C(3'')	0.6014 (3)	0.3173 (2)	0.6483 (2)	0.037 (1)
C(4'')	0.5185 (3)	0.4305 (2)	0.6631 (2)	0.044 (1)
C(5'')	0.4767 (3)	0.4635 (3)	0.7667 (2)	0.055 (1)
C(6'')	0.5182 (4)	0.3850 (3)	0.8542 (2)	0.061 (1)
C(7'')	0.6024 (4)	0.2745 (3)	0.8413 (2)	0.063 (1)
C(8'')	0.6444 (3)	0.2400 (3)	0.7383 (2)	0.051 (1)

Compound (3)

Crystal data

$\text{C}_{19}\text{H}_{31}\text{NO}_4$
 $M_r = 337.5$
 Monoclinic
 $P2_1$
 $a = 8.712 (1) \text{\AA}$
 $b = 12.848 (2) \text{\AA}$
 $c = 17.971 (2) \text{\AA}$
 $\beta = 90.39 (1)^\circ$
 $V = 2011.5 (5) \text{\AA}^3$
 $Z = 4$
 $D_x = 1.114 \text{ Mg m}^{-3}$

Data collection

Stoe Siemens four-circle
 diffractometer
 $\omega/2\theta$ profile fitting (Clegg,
 1981)
 Absorption correction:
 none
 7957 measured reflections
 3713 independent reflections
 3225 observed reflections
 $[F > 3\sigma(F)]$

Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{\AA}$
 Cell parameters from 55
 reflections
 $\theta = 10\text{--}12.5^\circ$
 $\mu = 0.07 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Block
 $0.8 \times 0.7 \times 0.5 \text{ mm}$
 Colourless

$R_{\text{int}} = 0.021$
 $\theta_{\max} = 25^\circ$
 $h = -10 \rightarrow 10$
 $k = -15 \rightarrow 15$
 $l = 0 \rightarrow 21$
 3 standard reflections
 monitored every 100
 reflections
 intensity decay: negligible

Refinement

Refinement on F $R = 0.051$ $wR = 0.053$ $S = 1.402$

3225 reflections

463 parameters

H atoms: see below

 $w = 1/[\sigma^2(F) + 0.0008F^2]$ $(\Delta/\sigma)_{\max} = 0.08$ $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors
from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV)

Orthorhombic

 $P2_12_1$ $a = 8.228 (1) \text{ \AA}$ $b = 15.310 (2) \text{ \AA}$ $c = 17.027 (1) \text{ \AA}$ $V = 2144.9 (7) \text{ \AA}^3$ $Z = 4$ $D_x = 1.184 \text{ Mg m}^{-3}$ Cell parameters from 40
reflections $\theta = 10\text{--}12.5^\circ$ $\mu = 0.20 \text{ mm}^{-1}$ $T = 293 \text{ K}$

Block

 $0.5 \times 0.5 \times 0.4 \text{ mm}$

Colourless

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (3)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(1)	0.3010 (3)	0.3525 (2)	0.4171 (1)	0.076 (1)
C(2)	0.1577 (4)	0.3417 (2)	0.4422 (1)	0.065 (1)
C(3)	0.0827 (3)	0.4287 (2)	0.4867 (2)	0.065 (1)
N(4)	0.1805 (3)	0.5179 (2)	0.5037 (1)	0.065 (1)
C(5)	0.3132 (3)	0.5209 (2)	0.4780 (2)	0.059 (1)
C(6)	0.3948 (3)	0.4428 (2)	0.4309 (2)	0.064 (1)
O(21)	0.0912 (3)	0.2626 (2)	0.4270 (1)	0.090 (1)
C(31)	-0.0512 (4)	0.4682 (3)	0.4387 (2)	0.088 (1)
O(51)	0.4094 (2)	0.6015 (2)	0.4920 (1)	0.077 (1)
C(52)	0.3504 (5)	0.6833 (3)	0.5381 (3)	0.098 (1)
C(61)	0.4503 (4)	0.4863 (3)	0.3575 (2)	0.084 (1)
C(62)	0.3206 (5)	0.5349 (4)	0.3122 (2)	0.114 (2)
C(63)	0.5397 (5)	0.4053 (4)	0.3153 (2)	0.119 (2)
C(1')	0.0222 (3)	0.3811 (2)	0.5604 (2)	0.059 (1)
C(2')	0.1438 (3)	0.3202 (3)	0.6053 (1)	0.063 (1)
C(3')	0.2562 (4)	0.3826 (3)	0.6524 (2)	0.100 (1)
C(4')	0.3718 (4)	0.3134 (4)	0.6915 (2)	0.114 (2)
C(5')	0.2943 (5)	0.2329 (3)	0.7389 (2)	0.096 (1)
C(6')	0.1809 (4)	0.1689 (3)	0.6934 (2)	0.090 (1)
C(7')	0.0684 (4)	0.2366 (3)	0.6528 (2)	0.071 (1)
C(1'')	-0.0629 (3)	0.4637 (3)	0.6074 (2)	0.071 (1)
C(2'')	-0.2341 (3)	0.4648 (3)	0.6020 (2)	0.077 (1)
O(2'')	-0.3072 (3)	0.4014 (3)	0.5675 (2)	0.116 (1)
C(3'')	-0.3115 (4)	0.5504 (4)	0.6425 (3)	0.105 (2)
O(1a)	0.7768 (2)	0.3230 (2)	0.1315 (1)	0.075 (1)
C(2a)	0.6299 (3)	0.3218 (2)	0.1096 (1)	0.064 (1)
C(3a)	0.5593 (3)	0.2204 (2)	0.0807 (2)	0.066 (1)
N(4a)	0.6616 (3)	0.1300 (2)	0.0806 (2)	0.072 (1)
C(5a)	0.7991 (4)	0.1409 (2)	0.0996 (2)	0.065 (1)
C(6a)	0.8793 (3)	0.2369 (2)	0.1245 (2)	0.069 (1)
O(21a)	0.5587 (3)	0.4001 (2)	0.1138 (1)	0.082 (1)
C(31a)	0.4252 (4)	0.1956 (3)	0.1329 (2)	0.094 (1)
O(51a)	0.8996 (2)	0.0607 (2)	0.0979 (2)	0.088 (1)
C(52a)	0.8375 (5)	-0.0382 (3)	0.0731 (3)	0.117 (2)
C(61a)	0.9642 (4)	0.2242 (4)	0.1997 (2)	0.096 (1)
C(62a)	0.8584 (6)	0.1883 (4)	0.2600 (2)	0.126 (2)
C(63a)	1.0475 (5)	0.3241 (4)	0.2203 (3)	0.131 (2)
C(1'a)	0.5005 (3)	0.2429 (2)	0.0004 (2)	0.063 (1)
C(2'a)	0.6311 (3)	0.2789 (2)	-0.0516 (2)	0.061 (1)
C(3'a)	0.7127 (4)	0.1946 (3)	-0.0975 (2)	0.077 (1)
C(4'a)	0.8423 (4)	0.2389 (3)	-0.1433 (2)	0.087 (1)
C(5'a)	0.7895 (5)	0.3257 (4)	-0.1932 (2)	0.102 (2)
C(6'a)	0.7056 (4)	0.4085 (3)	-0.1514 (2)	0.094 (1)
C(7'a)	0.5769 (4)	0.3639 (3)	-0.1040 (2)	0.076 (1)
C(1''a)	0.4063 (3)	0.1526 (3)	-0.0326 (2)	0.082 (1)
C(2''a)	0.2364 (3)	0.1601 (2)	-0.0269 (2)	0.067 (1)
O(2''a)	0.1716 (2)	0.2372 (2)	-0.0048 (2)	0.091 (1)
C(3''a)	0.1498 (4)	0.0676 (3)	-0.0537 (3)	0.102 (2)

Compound (4)

Crystal data

 $\text{C}_{20}\text{H}_{31}\text{N}_2\text{O}_3^+ \cdot \text{Cl}^-$ $M_r = 382.9$ Mo $K\alpha$ radiation $\lambda = 0.71069 \text{ \AA}$

Data collection

Stoe Siemens four-circle

diffractometer

 $\omega/2\theta$ profile fitting (Clegg,
1981)

Absorption correction:

none

3475 measured reflections

2182 independent reflections

2176 observed reflections

 $[F > 3\sigma(F)]$ $R_{\text{int}} = 0.024$ $\theta_{\text{max}} = 25^\circ$ $h = -9 \rightarrow 3$ $k = 0 \rightarrow 18$ $l = 0 \rightarrow 20$

3 standard reflections

monitored every 100

reflections

intensity decay: negligible

Refinement

Refinement on F $R = 0.055$ $wR = 0.053$ $S = 1.361$

2176 reflections

254 parameters

H atoms: see below

 $w = 1/[\sigma^2(F) + 0.0005F^2]$ $(\Delta/\sigma)_{\max} = 0.008$ $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors
from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV)Table 4. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (4)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(1)	0.9256 (3)	0.1499 (1)	0.6143 (1)	0.048 (1)
C(2)	0.8720 (5)	0.0900 (2)	0.5636 (2)	0.043 (1)
C(3)	0.7884 (5)	0.0091 (2)	0.5965 (2)	0.038 (1)
N(4)	0.7249 (4)	0.0169 (2)	0.6760 (1)	0.040 (1)
C(5)	0.7891 (5)	0.0737 (2)	0.7199 (2)	0.041 (1)
C(6)	0.9184 (5)	0.1377 (2)	0.6992 (2)	0.048 (1)
O(21)	0.8943 (4)	0.1017 (2)	0.4948 (1)	0.065 (1)
C(31)	0.9238 (5)	-0.0588 (2)	0.5956 (2)	0.051 (1)
O(51)	0.7477 (3)	0.0787 (2)	0.7965 (1)	0.054 (1)
C(52)	0.6404 (6)	0.0111 (3)	0.8243 (2)	0.066 (2)
C(61)	0.8983 (7)	0.2295 (2)	0.7338 (2)	0.073 (2)
C(62)	0.7405 (7)	0.2727 (3)	0.7044 (3)	0.094 (2)
C(63)	1.0452 (8)	0.2847 (3)	0.7170 (3)	0.106 (2)
C(1')	0.6463 (5)	-0.0210 (2)	0.5415 (2)	0.041 (1)
C(2')	0.6170 (5)	-0.1188 (2)	0.5495 (2)	0.042 (1)
C(3')	0.6612 (5)	-0.1726 (2)	0.4869 (2)	0.052 (1)
C(4')	0.6541 (6)	-0.2619 (2)	0.4943 (2)	0.060 (2)
C(5')	0.6037 (6)	-0.2986 (2)	0.5629 (2)	0.061 (1)
C(6')	0.5551 (5)	-0.2463 (2)	0.6245 (2)	0.059 (1)
C(7')	0.5607 (5)	-0.1565 (2)	0.6178 (2)	0.051 (1)
N(1'')	0.4911 (4)	0.0302 (2)	0.5542 (2)	0.044 (1)
C(1'')	0.5162 (6)	0.1278 (2)	0.5460 (2)	0.060 (2)
C(2'')	0.3603 (6)	0.1799 (3)	0.5552 (3)	0.086 (2)
C(3'')	0.3564 (5)	-0.0022 (2)	0.5020 (2)	0.060 (1)
C(4'')	0.3871 (7)	0.0081 (3)	0.4153 (2)	0.084 (2)
Cl	0.2852 (1)	0.0144 (1)	0.7036 (1)	0.071 (1)

Compound (5)*Crystal data*

C₁₆H₂₁NO₄
M_r = 291.3
 Monoclinic
*P*2₁
a = 8.039 (1) Å
b = 11.097 (1) Å
c = 8.498 (1) Å
 β = 98.92 (1)°
V = 748.9 (4) Å³
Z = 2
D_x = 1.292 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 45 reflections
 θ = 10–12.5°
 μ = 0.06 mm⁻¹
T = 293 K
 Block
 0.6 × 0.4 × 0.4 mm
 Colourless

Data collection

Stoe Siemens four-circle diffractometer
 $\omega/2\theta$ profile fitting (Clegg, 1981)
 Absorption correction: none
 1428 measured reflections
 1252 independent reflections
 1170 observed reflections
 $[F > 3\sigma(F)]$

*R*_{int} = 0.014
 θ_{\max} = 25°
 $h = -9 \rightarrow 9$
 $k = -3 \rightarrow 5$
 $l = 0 \rightarrow 10$
 3 standard reflections monitored every 100 reflections
 intensity decay: negligible

Refinement

Refinement on *F*
R = 0.027
wR = 0.033
S = 1.37
 1170 reflections
 205 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F) + 0.003F^2]$
 $(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.14 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.13 \text{ e \AA}^{-3}$
 Extinction correction: see below
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (6)*Crystal data*

C₁₆H₂₁NO₄
M_r = 291.3
 Monoclinic
*P*2₁
a = 8.512 (1) Å
b = 9.375 (1) Å
c = 10.607 (1) Å
 β = 105.49 (1)°
V = 815.7 (5) Å³
Z = 2
D_x = 1.186 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 40 reflections
 θ = 10–12.5°
 μ = 0.05 mm⁻¹
T = 293 K
 Block
 0.5 × 0.4 × 0.2 mm
 Colourless

Data collection

Stoe Siemens four-circle diffractometer
 $\omega/2\theta$ profile-fitted (Clegg, 1981)
 Absorption correction: none
 1917 measured reflections
 1821 independent reflections
 1441 observed reflections
 $[F > 3\sigma(F)]$

*R*_{int} = 0.023
 θ_{\max} = 25°
 $h = -10 \rightarrow 9$
 $k = -11 \rightarrow 2$
 $l = 0 \rightarrow 12$
 3 standard reflections monitored every 100 reflections
 intensity decay: negligible

Refinement

Refinement on *F*
R = 0.046
wR = 0.042
S = 1.37
 1441 reflections
 205 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F) + 0.0003F^2]$
 $(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$
 Extinction correction: see below
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 5. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (5)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
O(1)	0.6616 (1)	0.4688	0.5809 (1)	0.046 (1)
C(2)	0.7593 (2)	0.5078 (3)	0.4769 (2)	0.044 (1)
C(3)	0.7269 (2)	0.4618 (3)	0.3054 (2)	0.040 (1)
N(4)	0.5768 (2)	0.3872 (3)	0.2632 (2)	0.038 (1)
C(5)	0.5039 (2)	0.3472 (3)	0.3723 (2)	0.037 (1)
C(6)	0.5448 (2)	0.3713 (4)	0.5482 (2)	0.039 (1)
O(21)	0.8668 (2)	0.5804 (3)	0.5214 (2)	0.059 (1)
C(31)	0.7103 (3)	0.5693 (4)	0.1949 (3)	0.054 (2)
O(51)	0.3724 (2)	0.2712 (3)	0.3429 (2)	0.047 (1)
C(52)	0.3241 (3)	0.2336 (4)	0.1800 (2)	0.055 (2)
C(61)	0.3896 (2)	0.4009 (4)	0.6232 (2)	0.044 (1)
C(62)	0.3005 (3)	0.5114 (4)	0.5473 (3)	0.062 (2)
C(63)	0.4353 (3)	0.4161 (5)	0.8042 (3)	0.065 (2)
C(1')	0.8833 (2)	0.3843 (4)	0.2854 (2)	0.040 (1)
O(1')	0.8967 (2)	0.2976 (3)	0.4098 (2)	0.052 (1)
C(2')	0.8749 (2)	0.3291 (4)	0.1221 (2)	0.037 (2)
C(3')	0.9402 (3)	0.3884 (5)	0.0019 (3)	0.045 (2)
C(4')	0.9352 (3)	0.3379 (5)	-0.1475 (3)	0.058 (2)
C(5')	0.8642 (3)	0.2271 (5)	-0.1795 (3)	0.059 (2)
C(6')	0.7947 (3)	0.1666 (5)	-0.0631 (3)	0.053 (2)
C(7')	0.8021 (3)	0.2184 (5)	0.0873 (3)	0.045 (2)

Table 6. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (6)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
O(1)	0.6908 (2)	0.3379	0.4698 (2)	0.063 (1)
C(2)	0.5808 (3)	0.2757 (3)	0.3722 (3)	0.044 (1)
C(3)	0.4603 (3)	0.3661 (3)	0.2746 (3)	0.043 (1)
N(4)	0.4959 (3)	0.5180 (3)	0.2750 (2)	0.048 (1)
C(5)	0.6015 (3)	0.5677 (3)	0.3712 (3)	0.051 (1)
C(6)	0.7012 (3)	0.4912 (4)	0.4882 (3)	0.052 (1)
O(21)	0.5789 (3)	0.1481 (3)	0.3684 (2)	0.064 (1)
C(31)	0.2949 (3)	0.3502 (5)	0.3054 (3)	0.065 (1)
O(51)	0.6351 (3)	0.7082 (3)	0.3808 (2)	0.068 (1)
C(52)	0.5449 (6)	0.7974 (4)	0.2759 (4)	0.098 (2)
C(61)	0.8819 (4)	0.5273 (5)	0.5258 (4)	0.078 (1)
C(62)	0.9717 (6)	0.4485 (6)	0.6504 (4)	0.122 (2)
C(63)	0.9577 (6)	0.5039 (7)	0.4133 (5)	0.114 (2)
C(1')	0.4493 (3)	0.3131 (4)	0.1355 (3)	0.046 (1)
O(1')	0.3226 (2)	0.3861 (3)	0.0439 (2)	0.059 (1)
C(2')	0.6087 (3)	0.3235 (4)	0.0978 (3)	0.042 (1)
C(3')	0.6550 (4)	0.4485 (4)	0.0503 (3)	0.057 (1)
C(4')	0.7999 (4)	0.4577 (5)	0.0153 (3)	0.072 (2)
C(5')	0.8990 (4)	0.3416 (7)	0.0268 (3)	0.081 (2)
C(6')	0.8550 (4)	0.2158 (6)	0.0748 (3)	0.076 (2)
C(7')	0.7112 (4)	0.2074 (6)	0.1106 (3)	0.059 (1)

Table 7. Selected bond lengths (Å), bond angles (°) and torsion angles (°)

	(1)	(2)	(3) Molecule A	(3) Molecule B	(4)	(5)	(6)	Mean
O(1)—C(2)	1.336 (3)	1.332 (3)	1.337 (4)	1.337 (4)	1.334 (4)	1.342 (3)	1.331 (3)	1.336 [4]
C(2)—C(3)	1.520 (3)	1.527 (3)	1.524 (4)	1.530 (4)	1.524 (5)	1.528 (3)	1.509 (4)	1.523 [7]
C(3)—N(4)	1.453 (3)	1.454 (3)	1.459 (4)	1.463 (4)	1.454 (4)	1.461 (3)	1.456 (4)	1.457 [4]
N(4)—C(5)	1.249 (3)	1.253 (3)	1.248 (4)	1.251 (4)	1.263 (4)	1.253 (3)	1.256 (4)	1.253 [5]
C(5)—C(6)	1.500 (3)	1.507 (3)	1.495 (4)	1.484 (4)	1.488 (5)	1.503 (3)	1.487 (4)	1.495 [9]
C(6)—O(1)	1.443 (3)	1.444 (3)	1.440 (4)	1.428 (4)	1.459 (4)	1.432 (3)	1.450 (3)	1.44 [1]
O(21)—C(2)	1.209 (3)	1.201 (3)	1.201 (4)	1.184 (4)	1.200 (4)	1.199 (4)	1.197 (4)	1.199 [7]
O(51)—C(5)	1.349 (3)	1.343 (3)	1.354 (3)	1.352 (4)	1.350 (4)	1.346 (3)	1.346 (4)	1.349 [4]
O(51)—C(52)	1.449 (3)	1.436 (4)	1.436 (5)	1.450 (4)	1.441 (5)	1.440 (3)	1.439 (5)	1.442 [6]
O(1)—C(2)—C(3)	120.2 (2)	120.1 (2)	120.4 (3)	119.5 (3)	118.0 (3)	119.3 (2)	119.8 (2)	119.6 [8]
O(1)—C(2)—C(3)	116.0 (2)	115.8 (2)	115.7 (2)	115.6 (2)	116.0 (3)	115.8 (2)	116.4 (2)	115.9 [3]
O(1)—C(2)—C(3)	119.3 (2)	119.7 (2)	119.2 (2)	119.5 (3)	117.1 (3)	118.8 (2)	118.0 (2)	118.8 [9]
O(1)—C(2)—C(3)	128.6 (2)	127.8 (2)	129.3 (3)	128.5 (3)	127.8 (3)	128.2 (2)	128.6 (3)	128.4 [5]
O(1)—C(2)—C(3)	111.6 (2)	111.7 (2)	111.5 (2)	112.2 (2)	110.3 (3)	111.3 (2)	111.4 (2)	111.4 [6]
O(1)—C(2)—C(3)	123.5 (2)	123.3 (2)	123.7 (2)	124.2 (2)	122.7 (3)	123.3 (2)	122.9 (2)	123.4 [5]
O(1)—C(2)—C(3)	117.7 (2)	117.4 (2)	117.4 (3)	118.2 (3)	118.6 (3)	118.4 (2)	117.7 (2)	117.9 [5]
O(1)—C(2)—C(3)	122.1 (2)	122.4 (2)	122.2 (3)	122.4 (3)	123.4 (3)	122.3 (2)	122.5 (2)	122.5 [4]
O(1)—C(2)—C(3)	122.7 (2)	123.0 (2)	121.9 (3)	121.8 (3)	120.4 (3)	121.9 (2)	121.1 (3)	121.8 [9]
O(1)—C(2)—C(3)	108.7 (2)	109.2 (2)	108.8 (2)	109.6 (2)	111.8 (3)	109.9 (2)	110.3 (2)	110 [1]
O(1)—C(2)—C(3)	115.9 (2)	116.0 (2)	116.3 (2)	115.7 (2)	115.6 (2)	116.6 (2)	116.9 (2)	116.1 [5]
O(1)—C(2)—C(3)—N(4)	7.3 (3)	6.5 (3)	-4.3 (4)	0.8 (4)	20.3 (5)	6.5 (3)	14.1 (4)	
O(1)—C(2)—C(3)—N(4)	-8.4 (3)	-10.1 (3)	4.1 (4)	-3.7 (4)	-24.0 (4)	-13.2 (3)	-14.5 (3)	
O(1)—C(2)—C(3)—N(4)	1.9 (4)	2.0 (3)	-1.3 (4)	0.7 (5)	3.2 (5)	3.7 (3)	1.1 (3)	
O(1)—C(2)—C(3)—N(4)	6.1 (4)	9.4 (3)	-1.6 (4)	4.9 (5)	20.5 (5)	12.1 (3)	12.6 (4)	
O(1)—C(2)—C(3)—N(4)	-7.0 (3)	-12.8 (3)	1.3 (3)	-7.9 (4)	-23.6 (4)	-18.9 (3)	-12.5 (3)	
O(1)—C(2)—C(3)—N(4)	0.8 (3)	5.5 (3)	1.5 (4)	5.4 (4)	4.8 (5)	10.5 (3)	0.2 (4)	
O(1)—C(2)—C(3)—N(4)	-4.3 (3)	3.1 (3)	0.1 (4)	-0.3 (5)	5.6 (5)	2.7 (3)	-0.1 (3)	

The data were corrected for Lorentz and polarization effects. An extinction correction was made for compounds (1), (2), (5) and (6) [$F^* = F(1 + 0.002\chi F^2/\sin 2\theta)^{-1/4}$, where $\chi = 0.011$ (1), 0.009 (1), 0.009 (1) and 0.014 (1), respectively]. The structures were solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined with a modified version of *SHELX76* (Sheldrick, 1976). All H atoms were located by difference synthesis and refined with fixed individual displacement parameters using a riding model [with the exceptions that the hydroxy H atoms in (5) and in (6) were refined isotropically with the O—H distance restrained to 0.85 Å, and H(N) in (4) was refined isotropically without any constraints or restraints] or refining methyl groups as rigid groups; non-H atoms were refined anisotropically by blocked-cascade least-squares methods. Molecular graphics were prepared using *SHELXTL* (Sheldrick, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

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