

Table 1. Final positional parameters and B_{eq} for each atom (all values have been multiplied by 10^4)

The thermal factors have the form $\frac{1}{3}(\sum_i \sum_j \beta_{ij} a_i \cdot a_j)$.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
O(1)	756 (2)	4181 (2)	4139 (3)	3.9
O(2)	779 (2)	4066 (2)	6909 (4)	5.5
N	-896 (2)	3271 (3)	8465 (6)	7.8
C(1)	438 (3)	3909 (2)	2505 (5)	3.6
C(2)	436 (3)	3971 (3)	5614 (5)	4.2
C(3)	-351 (3)	3682 (2)	5546 (5)	4.2
C(4)	-784 (3)	3688 (3)	4149 (6)	5.1
C(5)	-428 (3)	3975 (3)	2563 (6)	4.8
C(6)	785 (3)	4422 (3)	1188 (6)	5.1
C(7)	1646 (3)	4353 (2)	1051 (6)	6.0
C(8)	1889 (3)	3551 (4)	0755 (7)	7.5
C(9)	1576 (3)	3040 (3)	2140 (7)	7.2
C(10)	711 (3)	3110 (3)	2177 (6)	6.0
C(11)	-655 (3)	3448 (3)	7173 (6)	5.5
C(12)	-1625 (3)	3464 (4)	4075 (7)	8.9
C(13)	-2127 (4)	4107 (4)	3883 (11)	12.6

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

C(1)-O(1)	1.483 (5)	O(1)-C(2)	1.342 (5)
C(2)-O(2)	1.195 (6)	C(2)-C(3)	1.467 (6)
C(3)-C(4)	1.335 (7)	C(4)-C(5)	1.486 (7)
C(1)-C(5)	1.515 (7)	C(3)-C(11)	1.448 (7)
C(11)-N	1.146 (7)	C(4)-C(12)	1.522 (8)
C(5)C(1)O(1)	108.8 (4)	C(5)C(1)C(6)	112.1 (4)
C(5)C(1)C(10)	113.1 (4)	C(6)C(1)O(1)	104.7 (4)
C(1)O(1)C(2)	120.5 (3)	C(3)C(2)O(1)	117.0 (4)
O(1)C(2)O(2)	119.5 (4)	C(2)C(3)C(4)	123.9 (4)
C(3)C(4)C(5)	117.4 (4)	C(1)C(5)C(4)	114.6 (4)
C(2)C(3)C(11)	114.2 (4)	C(3)C(11)N	179.3 (5)
C(3)C(4)C(12)	125.1 (5)	C(5)C(4)C(12)	117.4 (4)

Figs. 1 and 2 show a perspective view of the spiro compound and a *c*-axis projection of the unit cell, respectively. The atomic coordinates (Table 1) and essential bond distances and angles (Table 2) are given.*

* Lists of structure factors, anisotropic thermal parameters of the non-hydrogen atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43413 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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with the highest figure of merit revealed all the non-H atoms. H atoms could be located from the difference Fourier maps. Refinement of the structure was performed by the block-diagonal least-squares method, 204 parameters refined. The calculations were carried out on a FACOM M-360 at the Institute for Solid State Physics and a HITAC M200H at the Institute for Molecular Science using the UNICSIII program system (Sakurai & Kobayashi, 1979). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). In the last cycle of the refinement, positional parameter shifts of the non-H atoms were less than one tenth of their standard deviations; the final difference Fourier maps showed no peak greater than $0.3 \text{ e } \text{\AA}^{-3}$. The final *R* and *wR* values converged to 0.065 and 0.082, respectively, $R = (\sum |F_o| - |F_c|) / \sum |F_o|$, $wR = [\sum w(|F_o| - |F_c|) / \sum w(|F_o|)]$ where $w = [\sigma^2(|F_o|) + 0.042(|F_o|)^2]^{-1}$.

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Structures of 3-Cyano-hexahydronaphth[2,3-*e*][1,2]oxazines. 2

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Abstract. 9-Chloro-2-cyclohexyl-4β-methyl-3,4,4a,5-, 10,10a-hexahydronaphth[2,3-*e*][1,2]oxazine-3β-carbonitrile (1): $C_{20}H_{25}ClN_2O$, m.p. 446-447 K, $M_r = 344.89$, triclinic, $P\bar{1}$, $a = 12.467$ (8), $b = 9.991$ (7),

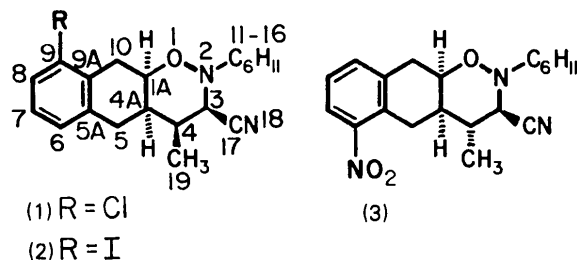
$c = 8.387$ (5) Å, $\alpha = 73.28$ (4), $\beta = 97.04$ (5), $\gamma = 112.79$ (6)°, $V = 922.3$ Å³, $Z = 2$, $D_x = 1.242$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu(\text{Mo } K\alpha) = 1.75$ cm⁻¹, $F(000) = 368$, room temperature, final $R = 0.068$, wR

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= 0.048 for 1552 unique observed [$I > 2\sigma$] reflections. 2-Cyclohexyl-9-iodo-4 β -methyl-3,4,4a α ,5,10,10a α -hexahydronaphth[2,3-*e*][1,2]oxazine-3 β -carbonitrile (2): $C_{20}H_{25}IN_2O$, m.p. 429–432 K, $M_r = 436.33$, monoclinic, $P2_1/c$, $a = 13.693$ (8), $b = 15.424$ (9), $c = 9.564$ (7) Å, $\beta = 104.16$ (5)°, $V = 1958.5$ Å³, $Z = 4$, $D_x = 1.48$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu(\text{Mo } K\alpha) = 15.16$ cm⁻¹, $F(000) = 880$, room temperature, final $R = 0.058$, $wR = 0.048$ for 1947 unique observed [$I > 2\sigma$] reflections. The tetrahydro-1,2-oxazine rings of both compounds are in distorted chair conformations with axial 3 β -cyano groups and equatorial *N*-cyclohexyl and 4 β -methyl substituents. The endocyclic torsion angles about the O(1)–N(2) and C(1A)–O(1) bonds are exceptionally large [–75.3 (5) and 72.8 (6)° for (1) and –75.2 (7) and 73.8 (7)° for (2)].

Experimental. Reaction of *N*-cyclohexyl-*N*-propenyl-nitrosonium ion with 5-chloro- and 5-iodo-1,4-dihydronaphthalene, respectively, followed by work-up with potassium cyanide, yielded in each case a mixture of six isomers (Van Dyk, 1986) from which the title compounds (1) and (2), which are equivalent isomers, were isolated. Both compounds were crystallized (colourless prisms) from dichloromethane–hexane.



Experimental data and structure refinement details are summarized in Table 1. Philips PW 1100 diffractometer, graphite-monochromated Mo $K\alpha$; $\omega/2\theta$ scans; Lorentz–polarization corrections; no corrections for absorption or secondary extinction; structures solved and refined by *SHELX76* (Sheldrick, 1976); (1) solved by direct methods; (2) solved by locating I position from Patterson map and the positions of the remaining non-H atoms from Fourier maps; refinement by full-matrix least-squares procedure minimizing the function $\sum w(|F_o| - |F_c|)^2$ with $w = [\sigma(F)]^{-2}$ treating non-H atoms anisotropically and H atoms isotropically; scattering factors from *International Tables for X-ray Crystallography* (1974); H atoms of (1) located from difference maps; H atoms of (2) included in ideal calculated positions in a riding model (all C–H = 1.08 Å, methyl group refined as a rigid group free to rotate).

Table 1. Summary of intensity measurement and structure refinement details

Compound	(1) ($R=\text{Cl}$)	(2) ($R=\text{I}$)
Crystal size (mm)	0.25 × 0.25 × 0.10	0.25 × 0.25 × 0.20
Unit-cell determination	25 reflections 3 < θ < 8°	25 reflections 3 < θ < 8°
θ_{max} (°)	23	23
hkl range	–13,13; –10,10; 0,9	–14,14; 0,16; 0,10
Number of standard reflections	3	3
Number of measured reflections	2610	2846
Criterion for observed reflections	$I > 2\sigma$	$I > 2\sigma$
Number of unique observed reflections	1552	1947
Hydrogens	From difference Fourier map	Calculated
Common U_{iso} for H (Å ²)	0.048 (3)	0.066 (5)
Max. $\Delta f/\sigma$	0.007	0.075
Max., min. $\Delta\rho$ (e Å ⁻³)	0.27, –0.30	0.87, –0.98
R, wR	0.068, 0.048	0.058, 0.048

Table 2. Atomic fractional coordinates ($\times 10^4$, $\times 10^5$ for I) and U_{eq} (Å² × 10³, × 10⁴ for I) for the non-H atoms with e.s.d.'s in parentheses

U_{eq} is defined as the geometric mean of the diagonal elements of the diagonalized matrix of U_{ij} .

Compound (1) ($R=\text{Cl}$)	x	y	z	U_{eq}
O(1)	973 (3)	8540 (3)	7632 (4)	35 (2)
N(2)	0 (3)	7128 (4)	7682 (5)	34 (2)
C(3)	465 (4)	6160 (6)	7207 (7)	40 (3)
C(4)	1390 (5)	5844 (6)	8514 (7)	41 (3)
C(4A)	2295 (5)	7294 (6)	8850 (7)	43 (4)
C(5)	3234 (5)	8179 (7)	7542 (8)	50 (4)
C(5A)	4021 (5)	9699 (6)	7728 (6)	41 (3)
C(6)	5133 (5)	10324 (7)	7025 (7)	48 (4)
C(7)	5823 (5)	11780 (9)	7088 (8)	62 (5)
C(8)	5461 (6)	12571 (8)	7857 (8)	57 (4)
C(9)	4395 (5)	11904 (7)	8574 (7)	49 (4)
C(9A)	3644 (4)	10454 (6)	8534 (6)	38 (3)
C(10)	2489 (5)	9770 (7)	9364 (7)	49 (4)
C(1A)	1668 (4)	8244 (6)	9099 (7)	41 (3)
C(11)	–873 (4)	7575 (6)	6515 (7)	38 (3)
C(12)	–1315 (5)	8468 (7)	7269 (8)	49 (4)
C(13)	–2318 (5)	8833 (7)	6221 (8)	58 (4)
C(14)	–3292 (5)	7426 (7)	5978 (8)	55 (4)
C(15)	–2839 (5)	6574 (7)	5203 (8)	53 (4)
C(16)	–1865 (5)	6165 (6)	6254 (7)	43 (4)
C(17)	869 (4)	6729 (6)	5461 (8)	40 (3)
N(18)	1116 (4)	7108 (5)	4092 (6)	58 (3)
C(19)	1886 (6)	4843 (8)	8064 (9)	60 (5)
Cl	4002 (1)	12907 (2)	9607 (2)	82 (1)

Compound (2) ($R=\text{I}$)	x	y	z	U_{eq}
O(1)	6363 (3)	2628 (3)	905 (5)	34 (3)
N(2)	5473 (4)	2195 (4)	1152 (7)	36 (3)
C(3)	5693 (5)	1259 (4)	1275 (8)	39 (4)
C(4)	6596 (5)	1117 (4)	2614 (8)	41 (4)
C(4A)	7506 (5)	1713 (4)	2561 (8)	35 (4)
C(5)	8088 (5)	1454 (4)	1461 (9)	37 (4)
C(5A)	8864 (5)	2105 (5)	1279 (8)	34 (4)
C(6)	9653 (6)	1835 (5)	723 (9)	48 (5)
C(7)	10381 (6)	2434 (6)	514 (10)	59 (6)
C(8)	10322 (7)	3275 (6)	886 (10)	59 (6)
C(9)	9524 (6)	3547 (5)	1421 (9)	44 (5)
C(9A)	8746 (5)	2982 (4)	1635 (8)	32 (4)
C(10)	7893 (5)	3305 (5)	2185 (9)	40 (5)
C(1A)	7100 (5)	2633 (4)	2279 (8)	37 (4)
C(11)	4661 (5)	2468 (5)	–109 (8)	36 (5)
C(12)	3710 (5)	1927 (5)	–166 (9)	44 (5)
C(13)	2836 (6)	2261 (5)	–1392 (10)	57 (6)
C(14)	2620 (6)	3216 (6)	–1211 (11)	64 (6)
C(15)	3566 (6)	3751 (5)	–1110 (10)	57 (6)
C(16)	4447 (6)	3426 (5)	104 (9)	45 (5)
C(17)	5823 (5)	860 (5)	–91 (10)	36 (5)
N(18)	5888 (5)	554 (4)	–1140 (9)	55 (4)
C(19)	6881 (6)	155 (5)	2789 (9)	54 (5)
I	94720 (5)	48652 (4)	19941 (9)	703 (4)

Table 2 gives the positional and thermal parameters and Table 3 bond distances and selected bond and torsion angles for the non-H atoms. The ring puckering parameters (Cremer & Pople, 1975) describing the conformation of the *B* and *C* rings are shown in Table 4.* Fig. 1 shows stereoviews of the two compounds.

* Lists of structure factors, anisotropic thermal parameters, positional and isotropic thermal parameters for the H atoms and lists of bond lengths and angles [including H atoms for compound (1)] have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43450 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Bond distances (Å) and selected bond and torsion angles (°) for the non-H atoms with *e.s.d.*'s in parentheses

	(1) (R=Cl)	(2) (R=I)
O(1)—N(2)	1.454 (4)	1.458 (7)
O(1)—C(1A)	1.443 (5)	1.448 (8)
N(2)—C(3)	1.463 (6)	1.473 (8)
N(2)—C(11)	1.485 (6)	1.487 (8)
C(3)—C(4)	1.551 (6)	1.563 (9)
C(3)—C(17)	1.484 (7)	1.493 (11)
C(4)—C(4A)	1.530 (7)	1.558 (9)
C(4)—C(19)	1.510 (8)	1.533 (9)
C(4A)—C(5)	1.520 (7)	1.521 (10)
C(4A)—C(1A)	1.511 (7)	1.524 (9)
C(5)—C(5A)	1.496 (7)	1.503 (9)
C(5A)—C(6)	1.406 (7)	1.380 (10)
C(5A)—C(9A)	1.376 (7)	1.415 (9)
C(6)—C(7)	1.387 (8)	1.408 (10)
C(7)—C(8)	1.372 (8)	1.354 (11)
C(8)—C(9)	1.361 (7)	1.380 (11)
C(9)—C(9A)	1.395 (7)	1.429 (10)
C(9)—R	1.726 (6)	2.112 (8)
C(9A)—C(10)	1.495 (7)	1.479 (9)
C(10)—C(1A)	1.534 (7)	1.520 (9)
C(11)—C(12)	1.513 (7)	1.536 (9)
C(11)—C(16)	1.525 (7)	1.531 (9)
C(13)—C(14)	1.507 (8)	1.521 (11)
C(14)—C(15)	1.499 (8)	1.519 (11)
C(15)—C(16)	1.527 (7)	1.539 (10)
C(17)—N(18)	1.141 (6)	1.132 (9)
C(1A)—O(1)—N(2)	105.8 (3)	106.3 (5)
O(1)—N(2)—C(3)	106.2 (3)	107.4 (5)
O(1)—N(2)—C(11)	104.1 (3)	103.1 (5)
C(11)—N(2)—C(3)	115.7 (4)	116.1 (5)
N(2)—C(3)—C(4)	109.3 (4)	107.9 (5)
C(3)—C(4)—C(4A)	111.8 (4)	111.7 (6)
C(4)—C(4A)—C(1A)	108.6 (4)	107.2 (6)
C(4A)—C(1A)—O(1)	108.6 (4)	107.4 (5)
C(10)—C(1A)—C(4A)	113.6 (4)	110.6 (6)
C(1A)—C(4A)—C(5)	111.7 (5)	114.3 (6)
C(4A)—C(5)—C(5A)	115.1 (5)	114.2 (6)
C(5)—C(5A)—C(9A)	120.3 (5)	119.1 (7)
C(9A)—C(10)—C(1A)	116.5 (5)	115.4 (6)
C(1A)—C(4A)—C(5)—C(5A)	47.5 (8)	49.9 (9)
C(4A)—C(5)—C(5A)—C(9A)	-25.2 (9)	-24 (1)
C(5)—C(5A)—C(9A)—C(10)	4.6 (9)	0 (1)
C(5A)—C(9A)—C(10)—C(1A)	-7.2 (9)	-3 (1)
C(9A)—C(10)—C(1A)—C(4A)	30.1 (8)	30 (1)
C(10)—C(1A)—C(4A)—C(5)	-49.6 (7)	-53.3 (9)
C(1A)—O(1)—N(2)—C(3)	-75.3 (5)	-75.2 (7)
O(1)—N(2)—C(3)—C(4)	63.1 (6)	62.4 (7)
N(2)—C(3)—C(4)—C(4A)	-50.5 (7)	-51.9 (8)
C(3)—C(4)—C(4A)—C(1A)	46.7 (7)	50.4 (8)
C(4)—C(4A)—C(1A)—O(1)	-58.0 (6)	-60.6 (7)
C(4A)—C(1A)—O(1)—N(2)	72.8 (6)	73.8 (7)
C(12)/C(16)—C(11)—N(2)—O(1)	-68.0 (6)	-71.6 (7)
C(16)/C(12)—C(11)—N(2)—C(3)	55.3 (6)	52.0 (8)

Related literature. This article forms part of a series on the structure and conformation of 6- or 9- halo- or nitro-substituted 3-cyano-2-cyclohexyl-4-methylhexahydronaphth-1,2-oxazines (Holzapfel, Kruger & Van Dyk, 1987). The cycloaddition reactions of *N*-cyclohexyl-*N*-propenylnitrosonium ions with 5-halo or -nitro-substituted 1,4-dihydronaphthalenes followed by treatment with potassium cyanide yielded in every case six isomers of the correspondingly substituted naphthoxazines of which the regio- and stereochemistry had to be established (Holzapfel, Koekemoer & Van Dyk, 1985; Van Dyk, 1986). The conformations of these two 3β-cyano-4β-methyl isomers are similar to that of the 6-nitro-substituted 3β-cyano-4α-methyl isomer (3) which also has the cyano group in an axial position (Holzapfel, Kruger & Van Dyk, 1987). These results provide evidence that the stereochemistry of the related cyano-substituted tetrahydrooxazines (Petrzilka, Felix & Eschenmoser, 1973; Riediker & Graf, 1979) have been correctly assigned on the basis of ¹H NMR data.

Table 4. Puckering parameters describing the conformation of the *B* and *C* rings

Compound	(1) (R=Cl)		(2) (R=I)	
	<i>B</i>	<i>C</i>	<i>B</i>	<i>C</i>
Ring				
<i>Q</i> (Å)	0.41	0.64	0.44	0.65
<i>θ</i> (°)	48.0	14.3	52.5	11.7
<i>φ</i> (°)	53.5	11.1	54.2	1.6
Conformation	<i>E</i> ₂	¹ <i>C</i> ₄	<i>E</i> ₂	¹ <i>C</i> ₄

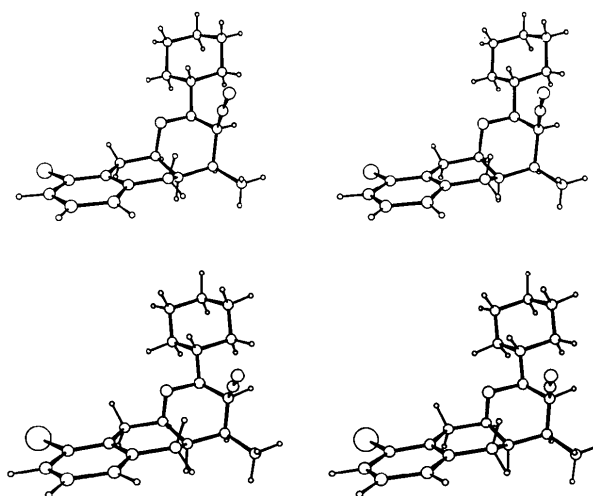


Fig. 1. Stereopairs showing the similarity in the conformations of compounds (1) (top) and (2) (bottom).

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2-Ethyl-1,3-diphenyl-1,3-propanedione

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Abstract. $C_{17}H_{16}O_2$, $M_r = 252.31$, monoclinic, $P2_1/a$, $a = 10.260$ (4), $b = 9.998$ (4), $c = 13.739$ (3) Å, $\beta = 92.15$ (3)°, $V = 1408.3$ Å³, $Z = 4$, $D_x = 1.19$ Mg m⁻³, λ (Mo $K\alpha$) = 0.71073 Å, $\mu = 0.072$ mm⁻¹, $F(000) = 536$, $T = 288$ K, final $R = 0.037$ for 926 unique observed reflections with $I > 3\sigma(I)$. The carbonyl functions which are oriented in a *cis* arrangement are non-coplanar (89.3°), indicating a lack of hydrogen-bonded enol formation. Details of the structural content and important bond distances and angles are reported. The bond lengths and angles are internally consistent.

Experimental. Crystals of the title compound (I) synthesized by a previously reported method (Kalyanam, Karban & McAtee, 1979); colorless rectangular crystal, 0.32 × 0.35 × 0.84 mm, glass fiber mount, Enraf-Nonius CAD-4F four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; cell constants from setting angles of 22 reflections ($4.0 \leq \theta \leq 13.0^\circ$); correction for Lorentz, polarization effects, no absorption correction; 288K; intensity data collected by ω -2 θ scan technique, variable scan rate of

Table 1. Atomic positional parameters ($\times 10^4$) and equivalent isotropic thermal parameters (Å² × 10³)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
O1	9811 (2)	3188 (2)	1700 (2)	779 (8)
O3	10237 (2)	2178 (2)	3875 (2)	971 (9)
C1	8808 (3)	3612 (3)	2039 (2)	55 (1)
C2	8449 (3)	3266 (3)	3072 (2)	51 (1)
C3	9214 (3)	2037 (3)	3407 (2)	62 (1)
C11	7980 (3)	4578 (3)	1473 (2)	53 (1)
C12	6660 (3)	4708 (4)	1605 (2)	64 (1)
C13	5931 (3)	5618 (4)	1049 (3)	79 (1)
C14	6519 (4)	6395 (4)	372 (3)	87 (1)
C15	7834 (4)	6277 (4)	243 (3)	91 (1)
C16	8555 (3)	5368 (4)	784 (2)	75 (1)
C21	8718 (3)	4477 (4)	3724 (2)	69 (1)
C22	8247 (4)	4311 (5)	4742 (3)	101 (2)
C31	8742 (3)	680 (3)	3146 (2)	55 (1)
C32	7641 (3)	478 (3)	2539 (2)	72 (1)
C33	7260 (4)	-802 (4)	2311 (3)	88 (1)
C34	7920 (4)	-1878 (4)	2675 (3)	96 (1)
C35	9001 (4)	-1682 (4)	3287 (3)	88 (1)
C36	9398 (3)	-419 (4)	3519 (2)	70 (1)

* Equivalent isotropic thermal parameter (U_{eq}) defined as $\frac{1}{3}$ the trace of the orthogonalized U_{ij} tensor.

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

O1–C1	1.221 (4)	O1–C1–C2	120.9 (3)
O3–C3	1.218 (4)	O1–C1–C11	120.0 (3)
C1–C2	1.519 (4)	C2–C1–C11	118.9 (3)
C1–C11	1.486 (4)	C1–C2–C3	109.1 (2)
C2–C3	1.520 (4)	C1–C2–C21	108.9 (3)
C2–C21	1.526 (5)	C3–C2–C21	112.5 (3)
C3–C31	1.480 (5)	O3–C3–C2	119.4 (3)
C11–C12	1.379 (4)	O3–C3–C31	120.1 (3)
C11–C16	1.382 (5)	C2–C3–C31	120.4 (3)
C12–C13	1.388 (5)	C1–C11–C12	122.5 (3)
C13–C14	1.370 (5)	C1–C11–C16	118.6 (3)
C14–C15	1.372 (5)	C12–C11–C16	118.9 (3)
C15–C16	1.373 (5)	C11–C12–C13	120.1 (3)
C21–C22	1.505 (5)	C12–C13–C14	120.2 (3)
C31–C32	1.393 (4)	C13–C14–C15	120.0 (3)
C31–C36	1.377 (5)	C14–C15–C16	119.9 (3)
C32–C33	1.371 (5)	C11–C16–C15	120.9 (3)
C33–C34	1.357 (5)	C2–C21–C22	113.7 (3)
C34–C35	1.380 (5)	C3–C31–C32	121.9 (3)
C35–C36	1.361 (5)	C3–C31–C36	119.3 (3)
		C32–C31–C36	118.8 (3)
		C31–C32–C33	119.3 (3)
		C32–C33–C34	121.4 (3)
		C33–C34–C35	119.4 (3)
		C34–C35–C36	120.1 (3)
		C31–C36–C35	121.0 (3)