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Structures of (1*SR*,5*SR*,6*SR*)- and (1*SR*,5*SR*,6*RS*)-4-Oxo-3-oxabicyclo[3.2.0]heptane-6-carbonitrile, C7H7NO2

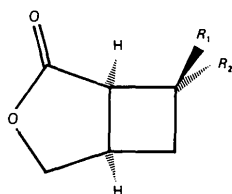BY FREDERICK W. B. EINSTEIN AND ANTHONY C. WILLIS

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Abstract. (1*SR*,5*SR*,6*SR*)(*endo*): $M_r = 137 \cdot 14$, monoclinic, $P2_1/n$, $a = 11 \cdot 134\,(2)$, $b = 8 \cdot 339\,(1)$, $c = 6 \cdot 826\,(2)$ Å, $\beta = 92 \cdot 05\,(2)°$, $V = 633 \cdot 36$ Å^3, $Z = 4$, $F(000) = 288$, $D_x = 1 \cdot 438$ Mg m^{-3}, Mo $K\alpha$ radiation $[\lambda(\alpha_1) = 0 \cdot 7093$ Å$]$, $\mu = 0 \cdot 100$ mm^{-1}, $T = 290$ K, $R = 0 \cdot 035$, 905 observed reflections. (1*SR*,5*SR*,6*RS*)(*exo*): $M_r = 137 \cdot 14$, orthorhombic, *Pbca*, $a = 11 \cdot 267\,(1)$, $b = 11 \cdot 095\,(2)$, $c = 10 \cdot 625\,(1)$ Å, $V = 1328 \cdot 2$ Å^3, $Z = 8$, $F(000) = 576$, $D_x = 1 \cdot 371$ Mg m^{-3}, Mo $K\alpha$ radiation, $\mu = 0 \cdot 095$ mm^{-1}, $T = 290$ K, $R = 0 \cdot 035$, 773 observed reflections. In both compounds the cyclobutane ring is attached to the lactone in a *cis* manner. The different configurations give rise to only minor differences in bond lengths and angles. All rings are non-planar but are flatter in the *exo* form than in the *endo*.

Introduction. In a study on [2+2] photocycloadditions of α,β-unsaturated lactones, presently underway at this University, a number of products were isolated (Oehlschlager & Lafontaine, 1983). The structures of two of the products were investigated by crystal structure analysis in order to determine unambiguously whether the cyclizations in these compounds were *cis* or *trans* and to locate the carbonitrile group.

The analysis showed that the two compounds are:

endo: R_1 = CN; R_2 = H
exo: R_1 = H; R_2 = CN

In the *endo* form the carbonitrile group is on the same side of the cyclobutane ring as the lactone whereas in the *exo* form they are on opposite sides.

Experimental. *Endo*: white needle, $0 \cdot 24 \times 0 \cdot 30 \times 0 \cdot 48$ mm; Picker FACS I diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; lattice parameters from setting angles of 18 reflections, $23 < 2\theta < 31°$; absorption ignored; θ–2θ scan; $\pm h,k,l$ with $4 < 2\theta < 50°$; variations in intensities of standards $<2\%$; 1117 independent reflections, 905 with $I > 2 \cdot 3\sigma(I)$; Lp correction applied; direct methods, *MULTAN* (Germain, Main & Woolfson, 1971); anisotropic block-diagonal refinement on F, H (from ΔF synthesis) isotropic; final $R = 0 \cdot 035$, $R_w = 0 \cdot 044$, $S = 1 \cdot 69$; $w = \{[\sigma(F)]^2 + 0 \cdot 0004F^2\}^{-1}$; max. $\Delta/\sigma = 0 \cdot 05$; all features in final ΔF synthesis between $\pm 0 \cdot 14\,(4)$ e Å^{-3}; PDP8e computer (Larson & Gabe, 1978); scattering factors from *International Tables for X-ray Crystallography* (1974).

Exo: white plate, $0 \cdot 52 \times 0 \cdot 41 \times 0 \cdot 19$ mm; Nonius CAD-4F diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; lattice parameters from setting angles of 25 reflections, $18 < 2\theta < 26°$; absorption ignored; θ–2θ scan; h,k,l with $0 < 2\theta < 48°$; standards' variation $<3\%$; 1043 independent reflections, 773 with $I > 2 \cdot 3\sigma(I)$; Lp correction applied; direct methods, *MULTAN*; anisotropic full-matrix refinement on F, H (from ΔF synthesis) isotropic; final $R = 0 \cdot 035$, $R_w = 0 \cdot 044$, $S = 1 \cdot 65$; $w = \{[\sigma(F)]^2 + 0 \cdot 0004F^2\}^{-1}$; max. $\Delta/\sigma = 0 \cdot 01$; all features in final ΔF synthesis between $\pm 0 \cdot 15\,(4)$ e Å^{-3}; VAX 11/750 computer; scattering factors from *International Tables* (1974).

Discussion. *ORTEP* diagrams (Johnson, 1970) of the two molecules displaying the atom labelling are shown as Figs. 1 and 2. The final atomic coordinates are given in Table 1 and selected bond lengths and angles in Table 2.*

The structures reveal that the cyclobutane ring is attached to *cis* sites of the five-membered ring in both compounds. The respective bond lengths and angles are very similar with a few small differences in the angles at C(7) and C(8) arising from the transposition of the carbonitrile group and the H atom at C(7). The dimensions are in general agreement with those in

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* Lists of structure factors, anisotropic temperature factors, distances and angles involving H atoms, torsion angles, and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39578 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
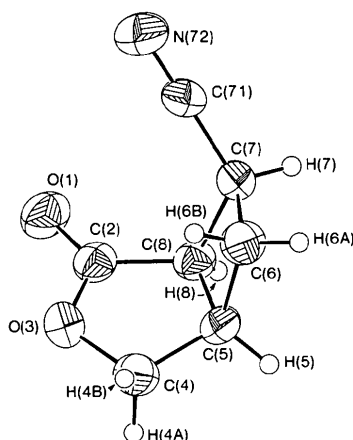

Fig. 1. *ORTEP* projection of the *endo* form showing the atom-labelling scheme. Ellipsoids indicate 50% probability levels and H atoms are drawn as arbitrarily small circles.
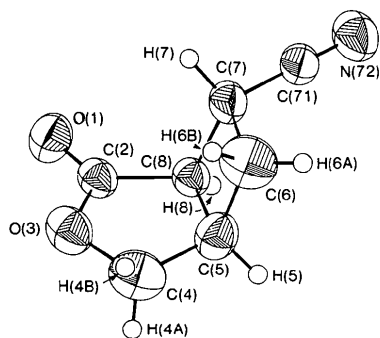

Fig. 2. *ORTEP* projection of the *exo* form showing the atom-labelling scheme. Ellipsoids indicate 50% probability levels. The coordinates listed in Table 1 have been transformed by $(1-x, 1-y, 1-z)$.

Table 1. *Final fractional atomic coordinates and isotropic thermal parameters* (Å²)

For non-H atoms $B_{eq} = \frac{8}{3}\pi^2(U_{11} + U_{22} + U_{33})$.

	x	y	z	B_{eq} or B
Endo				
O(1)	0.39417(9)	0.20033(15)	0.42330(16)	4.73
C(2)	0.48430(12)	0.25736(20)	0.36242(19)	3.27
O(3)	0.48452(9)	0.40564(13)	0.28622(15)	3.86
C(4)	0.60195(13)	0.44888(20)	0.21775(22)	3.84
C(5)	0.68733(12)	0.32186(17)	0.29510(19)	3.26
C(6)	0.73568(13)	0.34272(21)	0.50765(22)	3.92
C(7)	0.67519(13)	0.18346(18)	0.56349(20)	3.41
C(8)	0.60729(13)	0.18437(17)	0.36103(20)	3.09
C(71)	0.60429(13)	0.18134(18)	0.73848(20)	3.55
N(72)	0.54799(14)	0.18350(18)	0.87421(19)	4.90
H(4A)	0.5945(13)	0.4514(19)	0.0805(23)	4.7(4)
H(4B)	0.6223(14)	0.5574(19)	0.2647(23)	4.7(4)
H(5)	0.7476(14)	0.2962(16)	0.2061(21)	3.7(3)
H(6A)	0.8193(16)	0.3446(19)	0.5293(25)	5.0(4)
H(6B)	0.6979(13)	0.4406(17)	0.5669(22)	4.4(3)
H(7)	0.7294(12)	0.1003(15)	0.5703(20)	3.2(3)
H(8)	0.6057(13)	0.0841(18)	0.2900(23)	4.3(3)
Exo				
O(1)	0.13574(14)	0.30824(15)	0.30733(13)	4.96
C(2)	0.14067(16)	0.21528(18)	0.24999(22)	3.54
O(3)	0.10621(14)	0.11111(13)	0.30211(12)	4.64
C(4)	0.11342(29)	0.01225(24)	0.21252(28)	5.64
C(5)	0.17596(21)	0.05883(20)	0.09826(22)	4.51
C(6)	0.31121(26)	0.04960(24)	0.09725(34)	6.10
C(7)	0.32225(18)	0.18704(21)	0.10936(20)	3.87
C(8)	0.18442(17)	0.19480(18)	0.11853(18)	3.25
C(71)	0.37152(16)	0.24861(23)	0.00070(22)	3.97
N(72)	0.40844(17)	0.29584(19)	−0.08530(18)	5.51
H(4A)	0.0349(27)	−0.0075(23)	0.1961(22)	6.9(7)
H(4B)	0.1580(22)	−0.0497(25)	0.2593(29)	6.6(6)
H(5)	0.1421(19)	0.0330(22)	0.0243(24)	5.9(6)
H(6A)	0.3485(24)	0.0145(27)	0.0288(29)	8.2(8)
H(6B)	0.3404(25)	0.0079(29)	0.1732(28)	7.9(8)
H(7)	0.3633(18)	0.2156(18)	0.1816(20)	4.3(5)
H(8)	0.1469(16)	0.2485(17)	0.0625(17)	3.2(4)

related complexes such as 5,6-dimethyl-4-(1-oxo-2-phenylethyl)-1,4,5,7a-tetrahydroisobenzofuran-3(3a*H*)-one (Jones & Kennard, 1978), *trans*-2-(2,4-dihydroxy-3-methoxybenzyl)-3-(4-hydroxy-3-methoxybenzyl)-butyrolactone (Bryan & Shen, 1978), and 10α-*tert*-butyl-3,3aα,4,5,6,6aα,7,8,9,10-decahydro-7α-methyl-1*H*-naphtho[1,8a-*c*]furan-1,8-dione (Rubin, Danoff & Brooks, 1982). Those differences that do occur result from the differing nature of the substituents on the ring, especially when extra ring strain is imposed by adjacent rings.

The five-membered rings assume configurations intermediate between an envelope and a half-chair (Bucourt, 1974) with C(4) and C(5) being the atoms most distant from the least-squares plane of the five ring atoms, by almost equal distances on opposite sides of the plane. The ring is a little flatter in the *exo* form than in the *endo* ($\sum$|torsion angles| = 48·8° for *endo*, 32·0° for *exo*). γ-Butyrolactones without the constraints of attached rings tend to prefer an envelope configuration with four of the ring atoms approximately coplanar and the other typically 0·5 Å out of this plane (Glusker, Minkin & Casciato, 1971; Glusker, Minkin & Soule, 1972; Berman, Carrell & Glusker, 1973; Bryan & Shen, 1978; Sikirica, Vicković & Viterbo, 1979; Bruvo, Sikirica & Vicković, 1981; Brownbridge, Egert, Hunt, Kennard & Warren, 1981).

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

	Endo	Exo		Endo	Exo		Endo	Exo
O(1)–C(2)	1·198 (2)	1·199 (3)	C(5)–C(6)	1·539 (2)	1·527 (4)	C(7)–C(8)	1·551 (2)	1·558 (3)
C(2)–O(3)	1·341 (2)	1·339 (2)	C(6)–C(7)	1·543 (2)	1·535 (4)	C(8)–C(2)	1·499 (2)	1·499 (3)
O(3)–C(4)	1·450 (2)	1·455 (3)	C(7)–C(71)	1·455 (2)	1·452 (3)	C(8)–C(5)	1·530 (2)	1·527 (3)
C(4)–C(5)	1·506 (2)	1·496 (3)	C(71)–N(72)	1·137 (2)	1·132 (3)			
O(1)–C(2)–O(3)	120·90 (14)	121·3 (2)	C(4)–C(5)–C(8)	105·26 (11)	104·9 (2)	C(8)–C(7)–C(71)	118·03 (12)	113·9 (2)
O(1)–C(2)–C(8)	128·15 (15)	128·3 (2)	C(6)–C(5)–C(8)	89·72 (11)	90·3 (2)	C(7)–C(71)–N(72)	178·28 (16)	178·8 (2)
O(3)–C(2)–C(8)	110·94 (12)	110·5 (2)	C(5)–C(6)–C(7)	89·69 (11)	90·8 (2)	C(7)–C(8)–C(2)	114·36 (11)	113·2 (2)
C(2)–O(3)–C(4)	111·62 (11)	111·4 (2)	C(6)–C(7)–C(8)	88·78 (11)	88·8 (2)	C(7)–C(8)–C(5)	89·73 (11)	89·9 (2)
O(3)–C(4)–C(5)	106·10 (12)	107·3 (2)	C(6)–C(7)–C(71)	117·88 (13)	115·6 (2)	C(2)–C(8)–C(5)	103·86 (12)	105·1 (2)
C(4)–C(5)–C(6)	116·49 (12)	116·9 (2)						

The cyclobutane rings are also non-planar in both compounds. The puckering angle (Bucourt, 1974) for *endo* is 15·4° and for *exo* 4·6°. The increased puckering of *endo* probably arises from steric repulsion between the atoms of the lactone and the carbonitrile group.

Hydrogen bonding is not a major consideration in either structure. The shortest O···H contacts in *endo* are O(1)···H(5)($x-\frac{1}{2}$, $\frac{1}{2}-y$, $z+\frac{1}{2}$) = 2·57 (2) and O(3)···H(6B)(1−x, 1−y, 1−z) = 2·63 (2) Å, and in *exo* O(1)···H(6B)($\frac{1}{2}-x$, $\frac{1}{2}+y$, z) = 2·65 (3) and O(1)···H(8) = 2·69 (2) Å.

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2-(2-Quinolyl)cyclohexanone Phenylhydrazone, C$_{21}$H$_{21}$N$_3$

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Abstract. $M_r = 315·4$, monoclinic, $P2_1/n$, $a = 16·249$ (2), $b = 9·058$ (1), $c = 11·846$ (2) Å, $\beta = 102·42$ (1)°, $V = 1702·7$ (4) Å^3, $Z = 4$, $D_x = 1·23$ g cm^{-3}, $F(000) = 672$, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 5·35$ cm^{-1}, $R = 0·041$ for 1698 observed reflections. The quinoline moiety is not planar and its two component rings make an angle of 1·7 (1)°. The cyclohexane ring shows partial disorder, and three of