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# 7,11-Bis(5-methyl-2-furyl)spiro[5.5]undecane-1,5,9-trione

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Abstract. $C_{21}H_{22}O_5$, $M_r = 354.43$, orthorhombic, $P2_12_12_1$, $a = 9.052$ (4), $b = 10.439$ (6), $c = 19.561$ (2) Å, $V = 1848.5$ Å^3, $Z = 4$, $D_m = 1.25$, $D_x = 1.273$ Mg m^{-3}, $R_F = 0.041$ for 1736 reflections. The cyclohexanone ring has the twist-boat configuration.

Introduction. Crystals of the title compound (I) were provided, as a racemic mixture, by Professor H. Wynberg and Dr W. ten Hoeve, University of Groningen. The structure determination was undertaken to confirm the proposed twisted conformation of the cyclohexanone ring. A colourless transparent block-shaped crystal 0.20 × 0.33 × 0.61 mm was investigated on an Enraf–Nonius CAD-4 diffractometer to obtain cell dimensions. The space group is

(I)
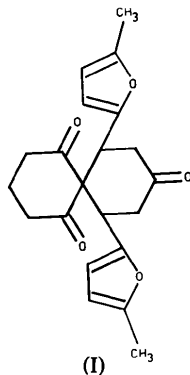
uniquely determined by the observed extinctions $h00$ for $h = 2n + 1$, $0k0$ for $k = 2n + 1$, $00l$ for $l = 2n + 1$ to be $P2_12_12_1$. Diffraction intensities were collected up to $\theta = 27.5°$ with Zr-filtered Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). Lorentz and polarization corrections but none for absorption were applied ($\mu = 0.098$ mm^{-1}). 2415 intensities were measured; 1736 with $I > 2\sigma(I)$ were included in the refinement.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and by a subsequent cycle of structure factor and Fourier calculations. The structure was refined by a block-diagonal least-squares procedure. 15 H atoms were found in the difference map, the other 7 were placed at calculated positions, and refined with isotropic temperature factors fixed at $U_{iso} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$, with U's of bonded C atoms. The final R_F and R_{wF} were 0.041 and 0.055 respectively. Cromer & Mann's (1968) analytical scattering factors for O and C were used. Those for H were taken from Stewart, Davidson & Simpson (1965). A Dutch version of the XRAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) was used for most calculations.

The positional parameters are listed in Tables 1 and 2.*

Table 1. *Fractional atomic coordinates* ($\times 10^4$)

	x	y	z
O(1)	7353 (3)	6456 (3)	1559 (1)
O(2)	5831 (3)	4130 (2)	4152 (1)
O(3)	2108 (3)	8256 (2)	3094 (1)
O(4)	5480 (3)	8155 (2)	3886 (1)
O(5)	3122 (3)	4566 (2)	2900 (1)
C(1)	7133 (4)	6289 (3)	2769 (2)
C(2)	5987 (3)	5565 (3)	3194 (1)
C(3)	6603 (4)	5108 (3)	3856 (2)
C(4)	6517 (5)	3853 (4)	4766 (2)
C(5)	5842 (7)	2800 (5)	5164 (2)
C(6)	7656 (5)	4632 (4)	4851 (2)
C(7)	7729 (4)	5443 (4)	4262 (2)
C(8)	4521 (4)	6356 (3)	3283 (1)
C(9)	4692 (4)	7229 (3)	3915 (2)
C(10)	3868 (7)	6902 (5)	4549 (2)
C(11)	2609 (7)	6139 (10)	4494 (3)
C(12)	2176 (5)	5466 (4)	3920 (2)
C(13)	3240 (4)	5385 (3)	3332 (2)
C(14)	4291 (4)	7225 (3)	2640 (1)
C(15)	2720 (4)	7623 (3)	2547 (2)
C(16)	655 (4)	8543 (3)	2914 (2)
C(17)	−220 (5)	9225 (5)	3433 (2)
C(18)	394 (4)	8116 (4)	2284 (2)
C(19)	1725 (4)	7529 (4)	2048 (2)
C(20)	4914 (4)	6627 (3)	1993 (2)
C(21)	6565 (4)	6461 (3)	2050 (2)

Table 2. *Fractional coordinates* ($\times 10^3$) *and bond distances* (Å) *for the* H *atoms*

	x	y	z	C–H
H'(C1)	269 (3)	216 (3)	204 (1)	0·99
H"(C1)	194 (3)	78 (3)	224 (1)	0·99
H'(C2)	428 (3)	977 (2)	206 (1)	1·00
H'(C5)	612 (4)	278 (4)	565 (2)	0·99
H"(C5)	475 (4)	285 (4)	519 (2)	1·00
H'''(C5)	595 (4)	197 (4)	492 (2)	0·99
H'(C6)	330 (4)	39 (3)	474 (2)	0·98
H'(C7)	155 (3)	109 (3)	82 (1)	0·96
H'(C10)	458 (4)	631 (3)	480 (2)	1·02
H"(C10)	372 (4)	762 (4)	477 (2)	0·88
H'(C11)	191 (5)	677 (5)	456 (2)	0·92
H"(C11)	228 (6)	565 (5)	487 (2)	0·94
H'(C12)	131 (4)	600 (4)	363 (2)	1·11
H"(C12)	168 (4)	465 (4)	397 (2)	0·97
H'(C14)	486 (3)	801 (3)	273 (1)	0·98
H'(C17)	126 (4)	464 (3)	184 (2)	1·17
H"(C17)	69 (4)	363 (3)	127 (2)	0·95
H'''(C17)	17 (4)	997 (3)	366 (2)	0·96
H'(C18)	62 (4)	328 (3)	297 (2)	1·06
H'(C19)	195 (3)	712 (3)	158 (1)	1·03
H'(C20)	451 (3)	577 (3)	191 (1)	0·98
H"(C20)	531 (3)	227 (3)	341 (1)	1·06

Discussion. The structure is shown in Fig. 1 with the atom numbering. The bond lengths and angles are given in Tables 3 and 4 respectively. C(10)—C(11) [1·395 (10) Å] and C(12)—C(11) [1·383 (8) Å] are much shorter than the value usually found for a single C—C bond; the angles C(13)—C(12)—C(11) [118·0 (4)°], C(12)—C(11)—C(10) [125·8 (5)°] and
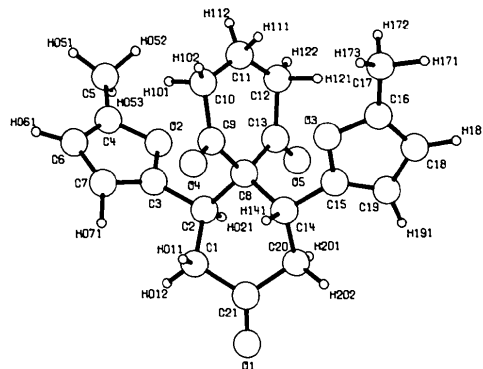

Fig. 1. Projection of the structure and atom numbering.

Table 3. *Bond distances* (Å) *involving non-hydrogen atoms*

O(1)—C(21)	1·196 (4)		
C(21)—C(1)	1·508 (5)	C(21)—C(20)	1·509 (5)
C(1)—C(2)	1·529 (5)	C(20)—C(14)	1·520 (4)
C(2)—C(8)	1·573 (4)	C(14)—C(8)	1·565 (4)
C(8)—C(9)	1·544 (4)	C(8)—C(13)	1·543 (5)
C(9)—C(10)	1·487 (6)	C(13)—C(12)	1·502 (6)
C(10)—C(11)	1·395 (10)	C(12)—C(11)	1·383 (8)
C(9)—O(4)	1·202 (4)	C(13)—O(5)	1·207 (4)
C(2)—C(3)	1·488 (4)	C(14)—C(15)	1·492 (5)
C(3)—O(2)	1·366 (4)	C(15)—O(3)	1·375 (4)
O(2)—C(4)	1·384 (4)	O(3)—C(16)	1·394 (4)
C(4)—C(5)	1·479 (6)	C(16)—C(17)	1·472 (6)
C(4)—C(6)	1·323 (6)	C(16)—C(18)	1·332 (6)
C(6)—C(7)	1·431 (5)	C(18)—C(19)	1·429 (5)
C(7)—C(3)	1·338 (5)	C(19)—C(15)	1·332 (5)

Table 4. *Bond angles* (°) *involving non-hydrogen atoms*

C(1)—C(21)—C(20)	114·8 (3)		
O(1)—C(21)—C(1)	123·0 (3)	O(1)—C(21)—C(20)	122·1 (3)
C(21)—C(1)—C(2)	109·6 (3)	C(21)—C(20)—C(14)	110·7 (3)
C(1)—C(2)—C(8)	111·9 (2)	C(20)—C(14)—C(8)	112·4 (2)
C(8)—C(2)—C(3)	112·8 (2)	C(8)—C(14)—C(15)	112·8 (3)
C(1)—C(2)—C(3)	112·2 (3)	C(20)—C(14)—C(15)	111·5 (3)
C(14)—C(8)—C(13)	109·3 (3)		
C(2)—C(8)—C(9)	108·3 (3)		
C(2)—C(8)—C(14)	109·1 (2)		
C(13)—C(8)—C(9)	114·5 (3)		
C(9)—C(8)—C(14)	108·3 (2)		
C(2)—C(8)—C(13)	107·2 (2)		
C(8)—C(9)—C(10)	118·8 (3)	C(8)—C(13)—C(12)	119·5 (3)
C(8)—C(9)—O(4)	119·7 (3)	C(8)—C(13)—O(5)	119·3 (3)
C(10)—C(9)—O(4)	121·5 (3)	C(12)—C(13)—O(5)	121·3 (3)
C(9)—C(10)—C(11)	118·5 (4)	C(13)—C(12)—C(11)	118·0 (4)
C(12)—C(11)—C(10)	125·8 (5)		
C(2)—C(3)—C(7)	135·9 (3)	C(14)—C(15)—C(19)	135·6 (3)
C(2)—C(3)—O(2)	114·6 (3)	C(14)—C(15)—O(3)	114·9 (3)
O(2)—C(3)—C(7)	109·5 (3)	O(3)—C(15)—C(19)	109·5 (3)
C(3)—O(2)—C(4)	107·1 (3)	C(15)—O(3)—C(16)	106·6 (3)
O(2)—C(4)—C(6)	109·3 (3)	O(3)—C(16)—C(18)	109·2 (3)
O(2)—C(4)—C(5)	115·3 (4)	O(3)—C(16)—C(17)	116·0 (3)
C(6)—C(4)—C(5)	135·4 (4)	C(18)—C(16)—C(17)	134·8 (4)
C(4)—C(6)—C(7)	107·4 (4)	C(16)—C(18)—C(19)	107·0 (3)
C(6)—C(7)—C(3)	106·7 (3)	C(18)—C(19)—C(15)	107·6 (3)

Table 5. *Some relevant torsion angles* (°)

Cyclohexanone ring		Cyclohexanedione ring		Exocyclic	
C(8)—C(2)—C(1)—C(21)	−64·8 (3)	C(8)—C(13)—C(12)—C(11)	−18·8 (7)	C(8)—C(14)—C(15)—O(3)	56·9 (3)
C(2)—C(1)—C(21)—C(20)	33·1 (4)	C(13)—C(12)—C(11)—C(10)	9·2 (11)	C(8)—C(14)—C(15)—C(19)	−123·9 (4)
C(1)—C(21)—C(20)—C(14)	28·0 (4)	C(12)—C(11)—C(10)—C(9)	11·8 (11)	C(20)—C(14)—C(15)—C(19)	3·6 (5)
C(21)—C(20)—C(14)—C(8)	−63·0 (3)	C(11)—C(10)—C(9)—C(8)	−22·7 (7)	C(8)—C(2)—C(3)—O(2)	72·8 (3)
C(20)—C(14)—C(8)—C(2)	31·3 (3)	C(10)—C(9)—C(8)—C(13)	12·5 (5)	C(8)—C(2)—C(3)—C(7)	−104·6 (4)
C(14)—C(8)—C(2)—C(1)	30·5 (3)	C(9)—C(8)—C(13)—C(12)	7·6 (4)	C(1)—C(2)—C(3)—C(7)	22·9 (5)
				C(1)—C(2)—C(3)—O(2)	−159·7 (3)

C(11)—C(10)—C(9) [118·5 (4)°] are larger than expected for tetrahedral angles. These observations may be related to the large thermal parameters of C(10), C(11) and C(12), possibly indicating some disorder in the cyclohexanedione ring, the nature of which has not been analysed further.

Some endocyclic and exocyclic torsion angles are listed in Table 5. The torsion angles in the cyclohexanone ring (−64·8, 33·1, 28·0, −63·0, 31·3, 30·5°) are near to the ideal twist-boat torsion angles −70·6, 33·2, 33·2, −70·6, 33·2, 33·2° (Pazdernik, Brisse & Rivest, 1977); the cyclohexanedione ring also has the twist-boat configuration, but less pronounced.

The methylfuryl rings are flat; the maximum deviation of the atoms from the least-squares plane is 0·004 Å in both rings. Intermolecular distances correspond to van der Waals contacts and no strong intermolecular interactions are apparent.

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cis-Bicyclo[3.2.0]hept-2-en-6-one p-Nitrophenylhydrazone

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Abstract. $C_{13}H_{13}N_3O_2$, $M_r = 243 \cdot 26$, monoclinic, $C2/c$, $a = 33 \cdot 55$ (5), $b = 4 \cdot 72$ (5), $c = 31 \cdot 11$ (5) Å, $\beta = 97 \cdot 83$ (2)° from diffractometer measurements (Mo $K\overline{\alpha}$ radiation), $V = 4876$ Å^3, $Z = 16$, $D_c = 1 \cdot 32$ Mg m^{-3}, $F(000) = 2048$, $\mu = 0 \cdot 055$ mm^{-1}, approximate crystal dimensions $0 \cdot 4 \times 0 \cdot 2 \times 0 \cdot 1$ mm. The molecules are linked into hydrogen-bonded tetramers.

Introduction. Systematic absences (from precession photographs) *hkl*: *h* + *k* odd and *h0l*: *l* odd indicated space group *C2/c*. Data were collected for *h*0–3*l* with $\theta_{max} = 23 \cdot 75°$ on a Stoe STADI-2 two-circle

diffractometer (graphite-monochromated Mo $K\overline{\alpha}$ radiation). This gave 2968 data of which 1301 unique reflexions with $I > 3\sigma(I)$ were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were applied, and the data scaled by a Wilson plot. The structure was solved by direct phasing methods with *SHELX* 76 (Sheldrick, 1976), which was used for all calculations. Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement (including isotropic H atoms) converged at $R = 0 \cdot 066$ for