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The Structure of a Condensation Product of Isobutylidenecyanoacetic Acid Ester with Cyclohexanone

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Abstract. 4-Ethyl-2-oxo-1-oxaspiro[5.5]undec-3-ene-3-carbonitrile, $C_{13}H_{17}NO_2$, $M_r = 219.3$, orthorhombic, *Pnca*, $a = 17.454$ (3), $b = 17.650$ (4), $c = 7.882$ (1) Å, $V = 2428.1$ (7) Å^3, $Z = 8$, $D_m = 1.20$, $D_x = 1.18$ g cm^{-3}, λ(Mo $K\alpha$, graphite monochromated) = 0.71069 Å, $\mu = 0.871$ cm^{-1}, $F(000) = 944$, room temperature, final $R(F) = 0.065$ for 971 reflections. The molecular structure of a condensation product of isobutylidenecyanoacetic acid ester with cyclohexanone was determined by the X-ray diffraction method and the product confirmed as a spiro compound. There are no unusual bond angles or distances.

Experimental. The title compound was prepared as described earlier (Igarashi, Nakano & Yatsu, 1984). D_m was measured by flotation in aqueous potassium iodide. The intensity data were collected on an automated Rigaku AFC-3 four-circle diffractometer at the Institute for Solid State Physics. A specimen with approximate dimensions $0.1 \times 0.2 \times 0.2$ mm (from ethanolic solution) was used. Reflections within the range $2\theta < 55°$ were collected by the θ–2θ scan technique with a scan speed of 4° min^{-1} in 2θ and scan width $\Delta\omega$ (°) = $1.1 + 0.5\tan\theta$. $h0\rightarrow22$, $k0\rightarrow22$, $l0\rightarrow10$. In total 1833 unique reflections were collected, 971 reflections with $|F_o| > 2.5\sigma(|F_o|)$ considered as observed. No corrections for absorption or extinction. Three standard reflections showed $<1.3\%$ variation.

Cell dimensions determined by a least-squares method from setting angles of 38 accurately centered reflections ($13 < \theta < 20°$). The structure was determined by direct-methods *MULTAN* (Main, Woolfson & Germain, 1971). An E map computed from the phase set
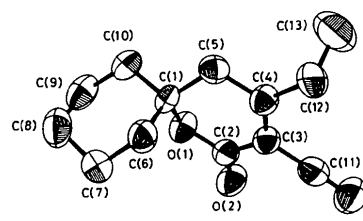

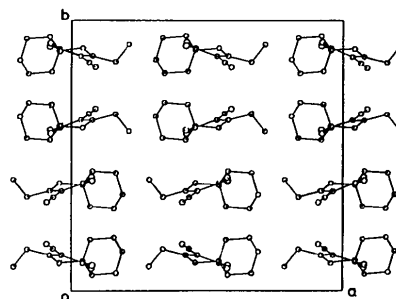Fig. 1. Perspective view and numbering scheme of the spiro compound.

Fig. 2. The arrangement of the compound in the unit cell viewed along c.

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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 $\frac{4}{3}(\sum_i \sum_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j)$.

	x	y	z	$B_{eq}(\text{Å}^2)$
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)

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 *UNICS*III 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 e Å^{-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 $w = [\sigma^2(|F_o|) + 0·042(|F_o|)^2]^{-1}$.

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.*

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Structures of 3-Cyanohexahydronaphth[2,3-e][1,2]oxazines. 2

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Abstract. 9-Chloro-2-cyclohexyl-4β-methyl-3,4,4aα,5,-10,10aα-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)°, $V = 922·3$ Å^3, $Z = 2$, $D_x = 1·242$ 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