Instrumental Achievements

Crystal Structure of 3-Methoxy-11-methyldibenz[*b, f*][1,4]oxazepine-8-carboxylic Acid, BY-1949

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The title compound (Fig. 1) increases regional cerebral blood flow.¹ Transparent yellow prisms of BY-1949, grown from a methanol solution², were subjected to the crystal structure analysis. Experimental conditions: crystal size 0.50×0.45×0.15 mm, Cu K α radiation (graphite-monochromated), θ -2 θ scan with variable scan speed 1.03–4.12°/min(θ), scan width (0.50+0.14 tan θ)°, range of indices $-15 \leq h \leq 15$, $0 \leq k \leq 13$, $0 \leq l \leq 12$ ($2\theta < 150^\circ$). Lattice constants were determined based on twenty-five 2θ values ($34 < 2\theta < 67^\circ$). No significant changes in the intensities of three standard reflections were observed during data collection. A total of 2691 unique reflections were collected, of which 2495 observed

reflections with $I > 2\sigma(I)$ were used for refinement of the structure.

The structure was solved by direct methods with MULTAN 11/82, and refined by a full-matrix least squares method. All hydrogen atoms were found on D-Fourier maps. Non-H atoms were refined with anisotropic thermal parameters and H atoms with the isotropic ones (fixed at $B=5.0 \text{ \AA}^2$). The quantity $\sum w(|F_o| - |F_c|)^2$ was minimized; $w=1.0$ for $|F_o| < 78.50$, $w=(78.50/|F_o|)^2$ for $|F_o| \geq 78.50$. The final refinement yielded the values: $R=0.046$, $wR=0.056$, $S=13.9$ for 243 variables, secondary-extinction factor (g) $7.3(1) \times 10^{-6}$ [$|F_o| = |F_c| / (1+gI)$], $(\Delta/\sigma) \leq 0.09$. The highest peak in the final D-Fourier map was $+0.23 \text{ e \AA}^{-3}$.

The crystal and experimental data are given in Table 1, and the final atomic parameters are in Table 2. The molecular structure is shown in Fig. 2, together with an atom-numbering scheme. The bond distances and angles are listed in Table 3. The oxazepine ring of BY-

Fig. 1 Chemical structure of BY-1949.

Table 1 Crystal and experimental data

Formula: C ₁₆ H ₁₃ NO ₄
Formula weight=283.28
Crystal system: monoclinic
Space group: <i>P</i> 2 ₁ / <i>n</i> <i>Z</i> =4
<i>a</i> =12.442(2) Å α —
<i>b</i> =10.449(1) Å β =93.55(1)°
<i>c</i> =10.061(1) Å γ —
<i>V</i> =1305.5(5) Å ³
<i>D_x</i> =1.441 g/cm ³
<i>R</i> =0.046
No. of reflections used=2495
Measurement: Enraf-Nonius CAD4
Program system: Enraf-Nonius SDP
Structure determination: MULTAN 11/82
Refinement: full matrix

Table 2 Final coordinates of non-H atoms

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} /Å ²
O(1)	0.4969(1)	0.0978(2)	0.6265(2)	2.83(3)
C(2)	0.5572(2)	0.0989(2)	0.7469(2)	2.44(4)
C(3)	0.5074(2)	0.1483(2)	0.8553(2)	2.75(4)
C(4)	0.5621(2)	0.1447(2)	0.9788(2)	2.87(5)
O(5)	0.5230(2)	0.1935(2)	1.0914(2)	3.84(4)
C(6)	0.4243(2)	0.2632(3)	1.0774(3)	4.11(6)
C(7)	0.6641(2)	0.0895(3)	0.9938(2)	3.26(5)
C(8)	0.7122(2)	0.0429(3)	0.8843(3)	3.14(5)
C(9)	0.6609(2)	0.0480(2)	0.7560(2)	2.65(4)
C(10)	0.7129(2)	-0.0013(2)	0.6386(2)	2.85(5)
C(11)	0.7911(3)	-0.1091(3)	0.6597(3)	4.48(6)
N(12)	0.6993(2)	0.0420(2)	0.5182(2)	2.79(4)
C(13)	0.6371(2)	0.1507(2)	0.4827(2)	2.45(4)
C(14)	0.6734(2)	0.2314(2)	0.3840(2)	2.71(4)
C(15)	0.6166(2)	0.3403(2)	0.3449(2)	2.70(4)
C(16)	0.6529(2)	0.4292(3)	0.2407(3)	3.27(5)
O(17)	0.6051(2)	0.5257(2)	0.2092(2)	6.00(5)
O(18)	0.7424(2)	0.3927(2)	0.1879(2)	3.97(4)
C(19)	0.5205(2)	0.3682(3)	0.4022(2)	3.17(5)
C(20)	0.4812(2)	0.2873(3)	0.4965(3)	3.18(5)
C(21)	0.5393(2)	0.1807(2)	0.5357(2)	2.56(4)

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} (a_i \cdot a_j).$$

Table 3 Bond distances (Å) and angles (°)

O(1)	C(2)	1.385(3)		C(7)	C(8)	1.375(4)		C(14)	C(15)	1.384(3)	
O(1)	C(21)	1.386(3)		C(8)	C(9)	1.405(3)		C(15)	C(16)	1.491(4)	
C(2)	C(3)	1.386(3)		C(9)	C(10)	1.474(3)		C(15)	C(19)	1.390(4)	
C(2)	C(9)	1.393(3)		C(10)	C(11)	1.495(4)		C(16)	O(17)	1.204(3)	
C(3)	C(4)	1.379(3)		C(10)	N(12)	1.294(3)		C(16)	O(18)	1.319(3)	
C(4)	O(5)	1.359(3)		N(12)	C(13)	1.408(3)		C(19)	C(20)	1.382(4)	
C(4)	C(7)	1.394(4)		C(13)	C(14)	1.398(3)		C(20)	C(21)	1.373(4)	
O(5)	C(6)	1.428(4)		C(13)	C(21)	1.394(3)					
C(2)	O(1)	C(21)	111.3(2)	C(14)	C(15)	C(16)	122.7(2)	C(2)	C(9)	C(8)	116.1(2)
O(1)	C(2)	C(3)	116.5(2)	C(14)	C(15)	C(19)	119.4(2)	C(2)	C(9)	C(10)	122.1(2)
O(1)	C(2)	C(9)	120.2(2)	C(16)	C(15)	C(19)	117.8(2)	C(8)	C(9)	C(10)	121.8(2)
C(3)	C(2)	C(9)	123.2(2)	C(15)	C(16)	O(17)	122.7(3)	C(9)	C(10)	C(11)	117.6(2)
C(2)	C(3)	C(4)	118.6(2)	C(15)	C(16)	O(18)	113.8(2)	C(9)	C(10)	N(12)	126.1(2)
C(3)	C(4)	O(5)	124.0(2)	O(17)	C(16)	O(18)	123.5(3)	C(11)	C(10)	N(12)	116.3(2)
C(3)	C(4)	C(7)	120.2(2)	C(15)	C(19)	C(20)	120.4(2)	C(10)	N(12)	C(13)	123.9(2)
O(5)	C(4)	C(7)	115.8(2)	C(19)	C(20)	C(21)	119.4(2)	N(12)	C(13)	C(14)	118.0(2)
C(4)	O(5)	C(6)	117.3(2)	O(1)	C(21)	C(13)	119.3(2)	N(12)	C(13)	C(21)	124.3(2)
C(4)	C(7)	C(8)	119.8(2)	O(1)	C(21)	C(20)	118.8(2)	C(14)	C(13)	C(21)	117.7(2)
C(7)	C(8)	C(9)	121.9(2)	C(13)	C(21)	C(20)	121.9(2)	C(13)	C(14)	C(15)	121.1(2)

Fig. 2 A perspective view with atom-numbering.

and N(12) lie near each mean plane of the aromatic rings to which they are bonded [deviations: 0.034(2) and 0.007(2) Å]. The O(1) atom deviates by -0.111(2) and 0.076(2) Å from these planes. An intermolecular hydrogen bond is observed between carboxy OH group and oxazepine N atom: O(18)···N(12)=2.728(3) Å, H(18)···N(12)=1.75(3) Å.

References

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2. K. Sasahara, N. Honda, M. Koizumi, Y. Murakami, T. Neichi, H. Nakakimura, Y. Noda, H. Matsushita and S. Hata, Tokkyo Koho (Japanese Patent) 2-023550 (1990).

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1949 is in a boat conformation. The dihedral angle between the two aromatic rings is 123.4°. Both C(10)