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Stereochemical Studies of Oligomers. XXVI.* Bis[4-(4-aminophenoxy)phenyl]-dimethylmethane

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Abstract. $C_{27}H_{26}N_2O_2$, $M_r = 410.5$, monoclinic, Pc , $a = 15.138$ (2), $b = 7.269$ (3), $c = 10.386$ (2) Å, $\beta = 99.60$ (4)°, $V = 1126.9$ Å³, $Z = 2$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 5.69$ cm⁻¹, $F(000) = 436$, room temperature, final $R = 0.075$ for 1349 observed reflections. As expected the four rings C4...C9 (*A*), C10...C15 (*B*), C16...C21 (*C*) and C22...C27 (*D*) are planar and their reciprocal dihedral angles are $A-B = 103.3$ (3), $A-C = 91.6$ (2), $A-D = 7.6$ (2), $B-C = 11.8$ (3), $B-D = 110.4$ (3) and $C-D = 98.8$ (2)°.

Experimental. Colourless prismatic crystal $0.4 \times 0.5 \times 0.8$ mm, Siemens AED single-crystal diffractom-

eter equipped with an IBM PS2/30 personal computer, nickel-filtered Cu $K\alpha$ radiation, intensities measured with a modified version (Belletti, Cantoni & Pasquinelli, 1988) of the Lehmann & Larsen (1974) procedure, $3 \leq \theta \leq 70^\circ$ range, 26 reflections with $11.37 \leq \theta \leq 44.15^\circ$ employed for refinement of lattice dimensions, index range $-18 \leq h \leq 18$, $0 \leq k \leq 8$, $0 \leq l \leq 12$, significant crystal decomposition revealed by a decrease in intensity of about 17% of one check reflection measured every 50, intensities

* Part XXV: Bocelli & Cantoni (1989).

Table 1. Atomic fractional coordinates ($\times 10^4$) and U_{eq} ($\times 10^4$ Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq} *
O1	8719 (4)	-76 (9)	1648 (6)	727 (22)
O2	2955 (4)	4434 (9)	210 (6)	782 (23)
N1	10257 (7)	-7137 (17)	2296 (14)	1105 (46)
N2	1450 (6)	11469 (12)	228 (13)	953 (43)
C1	5876 (11)	2469 (13)	4217 (11)	625 (29)
C2	6254 (9)	4052 (22)	5078 (12)	1001 (56)
C3	5504 (8)	1013 (19)	5073 (12)	859 (45)
C4	6622 (5)	1659 (12)	3559 (8)	578 (28)
C5	6952 (5)	-99 (13)	3717 (10)	591 (31)
C6	7650 (6)	-771 (14)	3120 (9)	686 (35)
C7	8014 (5)	351 (12)	2303 (8)	580 (27)
C8	7687 (6)	2150 (13)	2096 (10)	692 (34)
C9	7013 (6)	2764 (13)	2722 (9)	654 (33)
C10	9088 (6)	-1855 (13)	1840 (9)	663 (33)
C11	9669 (7)	-2232 (15)	2942 (10)	774 (40)
C12	10086 (7)	-3961 (16)	3103 (13)	819 (43)
C13	9863 (6)	-5266 (13)	2146 (11)	693 (37)
C14	9288 (6)	-4905 (15)	1055 (12)	720 (40)
C15	8886 (7)	-3147 (16)	860 (11)	822 (43)
C16	5102 (5)	3095 (11)	3157 (8)	619 (27)
C17	4700 (6)	1848 (13)	2242 (10)	686 (33)
C18	3990 (6)	2324 (14)	1284 (12)	777 (38)
C19	3673 (5)	4099 (11)	1202 (8)	602 (27)
C20	4060 (6)	5361 (12)	2114 (9)	718 (32)
C21	4779 (4)	4916 (10)	3083 (8)	510 (23)
C22	2576 (5)	6205 (11)	187 (8)	635 (31)
C23	1977 (6)	6545 (12)	1024 (10)	460 (33)
C24	1598 (7)	8274 (12)	964 (11)	773 (36)
C25	1824 (5)	9691 (12)	198 (9)	687 (34)
C26	2380 (6)	9205 (17)	-676 (9)	778 (38)
C27	2779 (5)	7474 (12)	-666 (9)	658 (29)

* Hamilton (1959).

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

O1—C7	1.393 (11)	C10—C15	1.381 (15)
O1—C10	1.410 (11)	C11—C12	1.403 (16)
O2—C19	1.389 (9)	C12—C13	1.375 (16)
O2—C22	1.408 (10)	C13—C14	1.335 (14)
N1—C13	1.482 (15)	C14—C15	1.415 (16)
N2—C25	1.413 (12)	C16—C17	1.379 (12)
C1—C2	1.510 (16)	C16—C21	1.409 (11)
C1—C3	1.548 (15)	C17—C18	1.381 (13)
C1—C4	1.532 (9)	C18—C19	1.374 (13)
C1—C16	1.537 (8)	C19—C20	1.377 (12)
C4—C5	1.372 (13)	C20—C21	1.392 (11)
C4—C9	1.387 (13)	C22—C23	1.379 (14)
C5—C6	1.399 (14)	C22—C27	1.349 (12)
C6—C7	1.358 (13)	C23—C24	1.379 (13)
C7—C8	1.402 (13)	C24—C25	1.379 (14)
C8—C9	1.373 (14)	C25—C26	1.383 (14)
C10—C11	1.350 (13)	C26—C27	1.395 (15)
C7—O1—C10	116.9 (6)	C12—C13—C14	121.6 (9)
C19—O2—C22	116.1 (6)	N1—C13—C14	117.6 (9)
C4—C1—C16	109.0 (4)	C13—C14—C15	120.2 (10)
C3—C1—C16	107.9 (5)	C10—C15—C14	118.1 (9)
C3—C1—C4	111.1 (6)	C1—C16—C21	122.2 (6)
C2—C1—C16	111.5 (6)	C1—C16—C17	119.7 (6)
C2—C1—C4	108.8 (6)	C17—C16—C21	118.1 (7)
C2—C1—C3	108.6 (6)	C16—C17—C18	122.2 (8)
C1—C4—C9	118.7 (6)	C17—C18—C19	120.1 (9)
C1—C4—C5	125.8 (6)	O2—C19—C18	115.6 (7)
C5—C4—C9	115.4 (7)	C18—C19—C20	118.6 (8)
C4—C5—C6	124.0 (8)	O2—C19—C20	125.7 (7)
C5—C6—C7	118.8 (8)	C19—C20—C21	122.3 (7)
O1—C7—C6	126.5 (7)	C16—C21—C20	118.7 (7)
C6—C7—C8	119.1 (8)	O2—C22—C27	120.0 (7)
O1—C7—C8	114.4 (7)	O2—C22—C23	117.5 (7)
C7—C8—C9	120.1 (8)	C23—C22—C27	122.5 (7)
C4—C9—C8	122.5 (8)	C22—C23—C24	116.6 (8)
O1—C10—C15	119.1 (8)	C23—C24—C25	124.4 (8)
O1—C10—C11	119.5 (8)	N2—C25—C24	122.1 (8)
C11—C10—C15	121.3 (9)	C24—C25—C26	115.1 (8)
C10—C11—C12	119.9 (9)	N2—C25—C26	122.6 (8)
C11—C12—C13	118.8 (10)	C25—C26—C27	122.4 (8)
N1—C13—C12	120.8 (10)	C22—C27—C26	118.4 (8)

corrected for this decay and for Lorentz and polarization effects, absorption ignored. 2437 reflections collected, 2338 unique, 1817 considered observed [$I \geq 3\sigma(I)$] and used in the refinement. Direct methods (*MULTAN80*, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), block-matrix anisotropic least squares (*SHELX76*, Sheldrick, 1976), H atoms located in a ΔF map, those of N atoms not found, in the last cycles of refinement 12 reflections probably affected by extinction were excluded, $\sum w\Delta F^2$ minimized, unit weights, $R = 0.075$, $(\Delta/\sigma)_{\max} = 0.09$, the final difference electron density map shows peaks from 0.28 to -0.23 e \AA^{-3} , 167 and 216 parameters refined in two blocks. Atomic scattering factors were from *SHELX76*.

All the calculations were performed on an IBM PS2/80 personal computer with the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreotti, 1987). The final atomic parameters are in Table 1, Table 2 reports bond distances and angles and the molecule is illustrated in Fig. 1.*

Related literature. This paper is strictly related to other systematic analyses of monomers of epoxy resins (Bel'skii, Chernikova, Rotaru & Kruchinin, 1983; Grenier-Loustalot & Bocelli, 1983).

* Lists of structure factors, anisotropic thermal parameters and H coordinates with isotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51933 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Crystalline Modification of Spironolactone

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Abstract. 7α -Acetylthio-3-oxo- 17α -pregn-4-ene-21,17 β -carbolactone, $\text{C}_{24}\text{H}_{32}\text{O}_4\text{S}$, $M_r = 416.6$, orthorhombic, $P2_12_12_1$, $a = 10.584(4)$, $b = 11.005(2)$, $c = 18.996(3) \text{ \AA}$, $V = 2213(2) \text{ \AA}^3$, $Z = 4$, $D_x = 1.25 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.7107 \text{ \AA}$, $\mu = 0.16 \text{ mm}^{-1}$, $F(000) = 896$, $T = 294 \text{ K}$, final $R = 0.047$ for 1237 reflections. The *A*-ring conformation is near that of a sofa, the *B* and *C* rings are chair shaped. The *D* ring is a distorted 13β envelope ($\Delta = 32.5^\circ$, $\varphi_m = 45.9^\circ$) and the *E* ring is almost a plane. All the molecules are held together by van der Waals forces.

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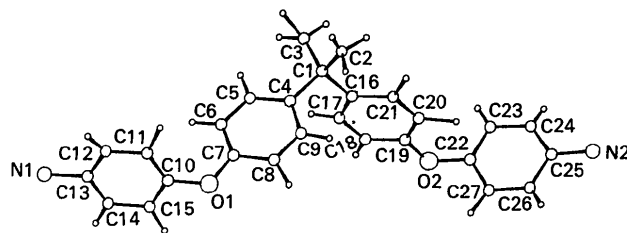


Fig. 1. A perspective view of the molecule with the atomic numbering scheme.

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The bioavailability of spironolactone (a useful diuretic, poorly soluble) depends on its allotropic form. Therefore, this crystalline modification, which is the more thermodynamically stable, has been studied.

Experimental. Single crystals prepared by cooling a supersaturated solution of spironolactone in acetone. Prismatic crystal: $0.22 \times 0.37 \times 0.40 \text{ mm}$. Enraf-Nonius CAD-4 diffractometer; lattice parameters determined from 25 reflections having $5.04 \leq \theta \leq$

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