J. L. BRIANSÓ
J. F. PINIELLA
Dept. de Cristalografía y Mineralogía,
Universidad Autónoma de Barcelona
Bellaterra — Spain

X. SOLANS
Dept. de Cristalografía,
Universidad de Barcelona
Spain

MOLECULAR AND CRYSTAL STRUCTURE OF 4-bromo-5-bromomethyl--3-hydroxyoxolan-2-one *

The molecular and crystal structure of 4-bromo-5-bromomethyl--3-hydroxyoxolan-2-one ($C_5H_6Br_2O_3$) has been determined by X-Ray Diffraction.
This molecule crystallizes in the Orthorhombic space group $Pna2_1$, a = 5.547(1). b = 21.759(5). c = 6.387(1) A .
The structure was solved by direct methods (MULTAN) and refined to R = 0.059.
Results from this X-Ray structure determination show that the title compound has the following configuration: c-4-bromo--c-5-bromomethyl-r-3-hydroxyoxolan-2-one.

(*) Communication presented at VII Congresso Iberoamecano de Cristalografia, Coimbra, September 1981. Manuscript received 29.3.1982.

PRELIMINARY INFORMATION

The reaction between Bromine and (E)-2,4--pentadienoic acid gives a series of 3, 4, 5 trisubstituted brominated γ-lactones [1] which appeared to be of several different configurations. One of them, a solid with melting point 149-151°C was shown to present the constitution 4-bromo-5--bromomethyl-3-hydroxyoxolan-2-one ($C_5H_6Br_2O_3$), but its configuration could not be completely solved by NMR methods. Since the knowledge of its configuration was important for a correct assignment of the configuration of other products formed in this and related reactions an X-ray structural study was felt necessary.

CRYSTAL DATA

From single crystal diffractometry, Mo Kα, λ = 0.71069 Å. Orthorhombic, a = 5.547(1), b = 21.759(5), c = 6.387(1) Å. V = 770.8(3) Å^3. Space group $Pna2_1$ Z = 4.

INTENSITY DATA, STRUCTURE DETERMINATION AND REFINEMENT

Intensity data were collected on an automatic Enraf-Nonius CAD-4 four circle diffractometer using Mo Kα radiation monochromated by a graphite crystal. 631 independent reflections were collected, out of the total; 547 reflections $I \geq 2\sigma(I)$ were accepted for the refinement. After the Lorentz polarization had been applied, normalized structure factors amplitudes were computed and the structure was solved by direct methods (MULTAN program [2] using 158 $|E_{hkl}| \geq 1.205$.

The atomic parameters were refined using the program SHELX-76 [3].

After three isotropic and three anisotropic cycles the R value was 0.059 for all reflections. The Hydrogen positions were not identified. Atomic coordinates and anisotropic thermal factors are listed in Table 1, bond distances and angles in Tables 2 and 3 repectively. They allow the determination of the structure as shown in figs. 1 and 2.

Table 1
Atomic coordinates ($\times 10^4$) and anisotropic thermal factors ($\times 10^4$) (standard deviations in parentheses)

Atom	X	Y	Z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O (1)	2791(23)	902(5)	4110(24)	233(71)	320(63)	381(71)	-7(61)	49(58)	-42(50)
C (2)	1989(32)	555(7)	5680(33)	189(96)	284(82)	402(127)	-10(85)	116(87)	-134(72)
C (3)	-806(29)	554(9)	5655(28)	202(87)	404(96)	256(103)	37(78)	-25(77)	76(68)
C (4)	-1336(34)	1127(10)	4312(32)	281(109)	506(112)	243(100)	88(96)	80(82)	19(86)
C (5)	835(31)	1138(9)	2790(30)	285(91)	402(92)	265(93)	7(96)	31(77)	92(76)
C (6)	1542(39)	1758(9)	1926(141)	645(141)	363(105)	385(138)	8(95)	375(120)	87(93)
Br(7)	-1535(4)	1860(1)	6044(5)	638(14)	416(11)	474(14)	44(12)	322(13)	130(10)
Br(8)	4177(4)	1691(1)	0(0)	465(12)	615(13)	292(11)	15(11)	160(12)	-57(9)
O (9)	-1786(27)	513(7)	7621(26)	330(91)	526(82)	363(96)	141(75)	99(75)	7(65)
O (10)	3291(22)	317(6)	6995(28)	284(68)	479(74)	421(84)	204(74)	62(67)	43(60)

Table 2
Bond distances (Å) (standard deviations in parentheses)

O(1)-C(2)	1.33(2)	C(3)-O (9)	1.37(2)
O(1)-C(5)	1.47(2)	C(4)-C (5)	1.55(2)
C(2)-C(3)	1.55(2)	C(4)-Br(7)	1.95(2)
C(2)-O(10)	1.22(2)	C(5)-C (6)	1.51(3)
C(3)-C(4)	1.54(3)	C(6)-Br(8)	1.92(2)

Table 3
Bond angles (degrees) (standard deviations in parentheses)

C(2)-O(1)-C(5)	113(1)	C(3)-C(4)-C (5)	102(1)
C(1)-C(2)-C(3)	109(2)	C(3)-C(4)-C (7)	111(1)
O(1)-C(2)-O(10)	124(1)	C(5)-C(4)-Br(7)	113(1)
C(37-C(2)-O(10)	127(2)	O(1)-C(5)-C (4)	102(1)
C(2)-C(3)-C(4)	101(1)	O(1)-C(5)-C (6)	109(2)
C(2)-C(3)-O(9)	113(1)	C(4)-C(5)-C (6)	116(1)
C(4)-C(3)-O(9)	119(2)	C(5)-C(6)-Br(8)	111(1)

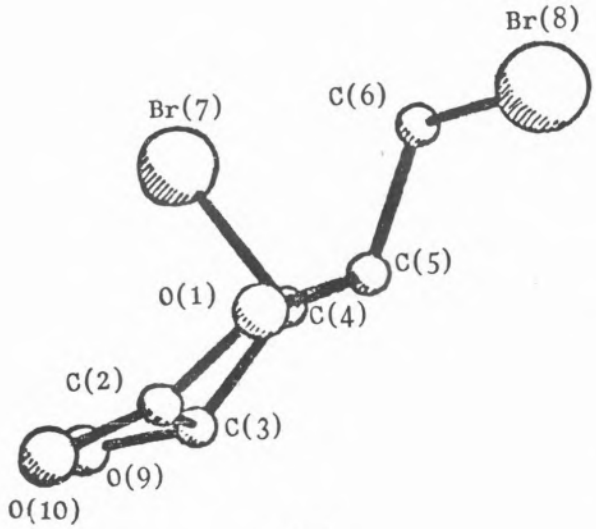

Fig. 1

COMMENTS

The results from this study demonstrated that in the cristalline state the title compound possesses the following configuration: *c*-4-bromo-*c*-5--bromomethyl-*r*-3-hydroxyoxolan-2-one.

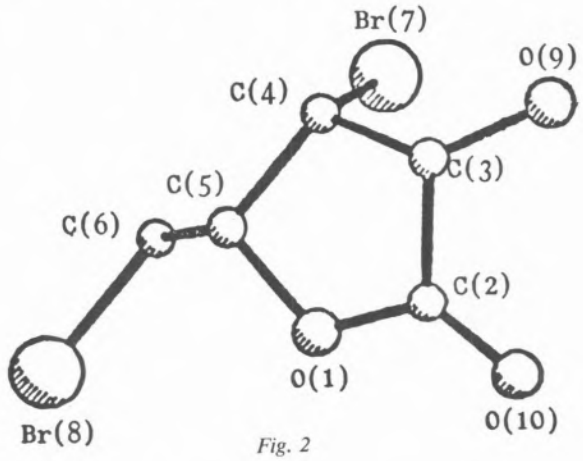

Fig. 2

ACKNOWLEDGEMENTS

The authors are indebted to Prof. J. Font, Prof. F. Sánchez--Ferrando and Dr. L. Vilamajó (Universidad Autónoma de Barcelona — Bellaterra — Spain) for supplying the sample.

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