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THE X-RAY INVESTIGATION OF EUPTELEOGENIN IODOACETATE

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IN connection with the preceding paper[1] of the chemical study on the structure of eupteleogenin carried out by Murata et al., it is reported in this preliminary note that we have so far confirmed their proposed structure by means of the X-ray analysis of its iodoacetate.

Eupteleogenin iodoacetate, $C_{31}H_{43}O_5I$, M = 622.56, was crystallized from a mixture of methanol and methylenechloride to afford colorless monoclinic plates, m.p. 225-228° (decomp.). The unit cell containing two molecules is of the dimensions, a = 17.72, b = 6.76 and c = 12.88 Å, β = 108°, and the space group is $P2_1$. The density measured by the floatation method gave 1.400 (calcd. 1.409) g/cm^3

Since the temperature factor of these crystals was found too large to obtain enough reflections at room temperature, we adopted a technique of cooling the specimen with the cold nitrogen gas from its liquid. Thus the 3695 independent three dimensional data at -140°C were collected from the integrating Weissenberg photographs (7 layers about b and 10 layers about c respectively).

The iodine atom co-ordinates were determined without ambiguity from a three dimensional Patterson synthesis. Further, a minimum

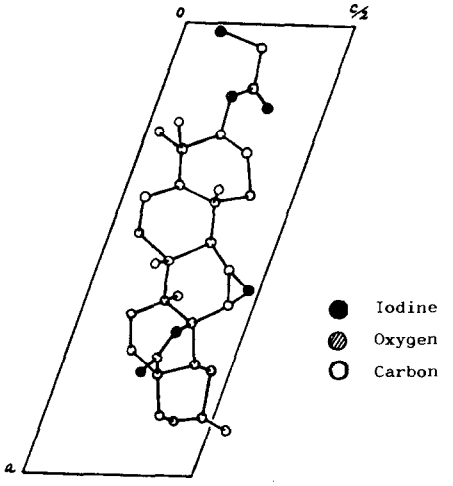
o
c/2
a
Iodine
Oxygen
Carbon

Fig. 1

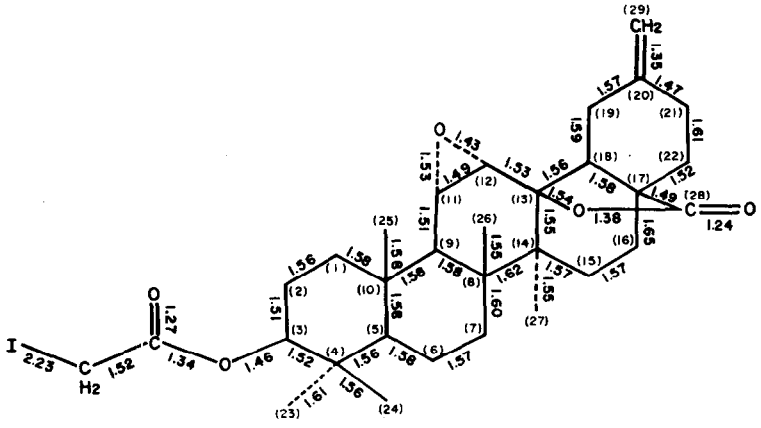
(29)
CH2
1.35
1.57
1.47
(20)
(19)
(21)
1.59
1.61
O
1.43
1.53
1.53
1.49
1.56
(12)
(18)
(22)
(17)
1.52
1.49
(28)
(11)
(13)
1.54
1.38
O
1.38
C
O
1.24
(25)
(26)
1.51
1.55
1.55
(9)
(14)
(16)
1.56
1.58
1.58
1.58
1.62
1.57
1.57
(1)
(15)
(2)
(10)
(8)
1.60
1.55
(27)
O
1.27
1.51
(3)
(5)
(7)
I
2.23
C
H2
1.52
C
1.34
O
1.46
1.52
(4)
1.56
1.58
(6)
1.57
(23)
1.61
1.56
(24)

Fig. 2

function synthesis and a three dimensional Fourier synthesis of the electron density distribution on NEAC 2206 computer visualized a whole structure of eupteleogenin iodoacetate as given in Fig. 1. Further refinement is now in progress, the R-value being 0.22 at the present stage.

The bond lengths in the molecule of the iodoacetate are shown in Fig. 2.

The proposed structure of eupteleogenin is thus confirmed.

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REFERENCE

1. T. Murata, S. Imai, M. Imanishi, M. Goto and K. Morita, Tetrahedron Letters this issue.