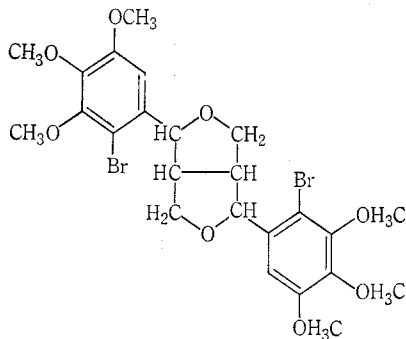


THE STEREOCHEMISTRY OF LIRIORESINOL-B*

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The gross structure of Lirioresinol-B dimethyl ether has been established by Dickey (1958) and independently by Jefferies, Knox, and White (1961). In addition evidence for the *cis*-fusion of the tetrahydrofuran rings was presented. Dickey, by consideration of the molecular rotation differences concluded that the stereochemical arrangement was probably identical with that in (+)-pinioresinol (Erdtman and Gripenberg 1947).



(I)

It was thought worth while investigating the molecular conformation by an independent means to confirm the probable structure. Accordingly an X-ray diffraction structure investigation was made of the 2,2'-dibromodimethyl ether derivative (I), kindly provided by Knox for this purpose. Assuming the *cis*-fusion of the tetrahydrofuran rings only three distinct stereochemical arrangements of the molecule are possible (II, III, IV).

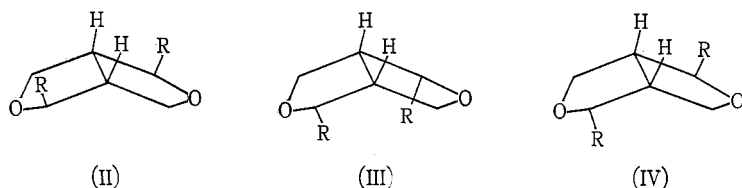
Crystals of the material prepared by slow recrystallization from ethanol were acicular in shape, and conformed to the orthorhombic system. A series

* Manuscript received October 4, 1961.

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of oscillation and zero and higher level Weissenberg photographs were taken. These showed that the unit cell belonged to space group $P_{2,2,2}$ and from them the cell dimensions were found to be: $a=16.7 \text{ \AA}$; $b=15.8 \text{ \AA}$; $c=4.70 \text{ \AA}$.

An approximate density determination showed that there were two molecules in the unit cell, and hence half a molecule in the asymmetric unit. This is possible only if the molecule contains a twofold rotation axis of symmetry,



which enables structure (IV) to be eliminated. Moreover the cell dimension in the direction of the rotation axis, which sets a limit to the thickness of the molecule in that direction, is 4.70 \AA . This, as may readily be shown from a model, is incompatible with structure (III). Structure (II) must therefore be the correct molecular configuration.

As this is in full agreement with the stereochemical arrangement postulated by Dickey from the molecular rotation differences, no further work on the structure is contemplated.

References

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