X-Ray Determination of the Structure of a New Insecticide, 2,2-Di-(*p*-ethoxyphenyl)-3,3-dimethyloxetan

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Summary The crystal structure of the title compound has been determined and its stereochemistry defined.

THIS determination was required for the refinement of the theoretical model of DDT type insecticides.[1] The compound synthesised, *a priori* from atomic model projections, was subsequently found to be 25 times as active as DDT as an insecticide.[2] The oxetan ring dimensions of the constructed model were approximate as no accurate parameters were available from previous structure determinations.

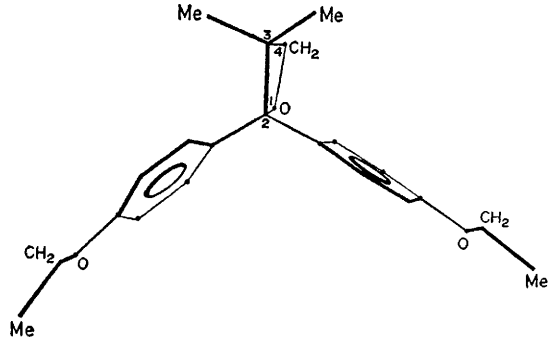

FIGURE. *Perspective view of the molecule as taken from an ORTEP plot.*[7]

The compound was crystallized (n-hexane) in the form of colourless flakes, m.p. 74°.

Crystal data: $C_{21}N_{26}O_3$; $M = 326$, monoclinic; $a = 9{\cdot}276(5)$, $b = 17{\cdot}766(10)$, $c = 11.690(7)$ Å, $\beta = 100°\ 57'(7)$; $U = 1786$ Å; $D_m = 1{\cdot}99(2)$ (by flotation); $Z = 4$; $D_c = 1{\cdot}214$; $\mu = 6{\cdot}39$ cm^{-1}; space group, $P2_1/c$. Intensity data for 2036 independent reflexions ($2\theta < 100°$) were measured with a Siemens diffractometer using filtered Cu-K radiation. As the crystals deteriorated rapidly in the X-ray beam, three were required to collect all the data. The structure was solved by direct methods using 312 reflexions with $|E| > 1{\cdot}4$. Least-squares refinement has lowered the conventional R-factor to 0·13.

Each ethoxy-group is approximately coplanar with its attached phenyl ring, a feature occurring in the insecticidal agent 1,1-di-(*p*-ethoxyphenyl)-2,2-dimethylpropane.[3] As was found for DDT[4] but not for the propane, the two planar substituted aromatic groups are slightly rotated with respect to each other.

The oxetan ring is puckered with a dihedral angle of 16·0° between the planes formed by O(1), C(2), and C(3) and by O(1), C(4) and C(3). This value is only half of the dihedral angles found in puckered cyclobutane rings.[5] The oxetan C–O bond lengths of 1·47 and 1·48 Å are significantly longer than the normal value of 1·43 Å found in paraffins and saturated heterocycles.[6] All other bond distances and angles lie within two standard deviations from expected values.[6] The shape of the dimethyloxetan ring, as viewed down the C(2)–C(3) bond and assuming van der Waals radii of 1·4 and 1·2 Å for O and H respectively, is roughly that of a cylinder of diameter 6·5 Å, a value somewhat greater than that derived from models.[2]

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