

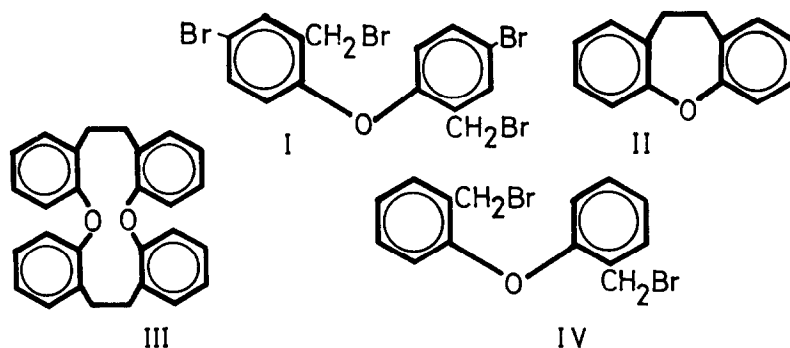
A NEW APPROACH TO THE DIBENZ [b, f] AZEPINE AND [b, f] OXEPINE SYSTEM. — A CORRECTION

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In the paper quoted in the Title (1), we reported that the reaction of 4,4'-dibromo-2,2'-bis (bromomethyl) diphenyl ether (I) with an added excess of phenyl lithium in ether as solvent leads, with a 67% yield, to a solid compound of m.p. 154°, accompanied by a liquid product which we had not yet investigated. We ascribed to the solid product formula II of 10,11-dihydrodibenz [b, f] oxepine on the basis of the analysis, the N.M.R. and the mass spectrum.



Professor Boekelheide has kindly drawn our attention to the fact that he had described, a few months before (2), compound II as an oil (b.p. 100°/1 mm); he also suggested for our solid product a dimeric formula. Osmometric determination of the molecular weight showed that this suggestion is correct. As both the NMR spectrum of the solid compound and the effect of the temperature on the spectrum are identical with those observed for II (3), we suggest for the solid compound formula III; under electron impact it "depolymerizes" to II. Both II and III show at room temperature a singlet, indicating a rapid inversion of the non-planar heterocyclic ring systems. This singlet broadens at lower temperatures and reaches coalescence at about -44°. The energy barrier to inversion is thus about 10^{kcal}/mole.

The oil observed in our previous study (1) has now been purified; its yield is 20% and its boiling point and physical properties, including the behaviour on gas chromatography, are identical with those of a sample of II kindly put at our disposal by Professor Boekelheide.

We report now an even simpler route to II than that described before. 2,2'-Dimethyldiphenyl ether is brominated by 2.5 mole of NBS in boiling carbon tetrachloride and in the presence of benzoyl peroxide in 85% yield to 2,2'-bis(bromomethyl) diphenyl ether (IV, m.p. 95° (from heptane). The latter gives with 1 mole of phenyl lithium in tetrahydrofuran compound II in excellent yield.

REFERENCES

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- 3) Z. Aizenshtat and E.D. Bergmann, *Israel J. Chem.*, 6, 7 p (1968) (abstract).