131. *The Relation between Configuration and Conjugation in Diphenyl Derivatives. Part VIII.** *Some Compounds derived from 2 : 2′-Diacetyldiphenyl.*

By G. H. Beaven and E. A. Johnson.

The ultraviolet absorption spectra of some substituted diphenyls derived from the reduction of 2 : 2′-diacetyldiphenyl [1] have been determined. The spectra of the 2 : 2′-di-(α-hydroxyalkyl)diphenyls are similar to those of 2 : 2′-dialkyldiphenyls, and show the same trend of decreasing intensity and short-wave shift of the conjugation band with increasing size of *ortho*-substituent. The spectra of the 2 : 2′-bridged diphenyls with saturated bridges are consistent with the extent of diphenyl-type conjugation expected for such non-coplanar structures. The introduction of a double bond into a C_3 bridge alters the spectrum very markedly, in a manner consistent with increased overall conjugation.

In diphenyls with *ortho*-substituents which are also capable of conjugation with the phenyl groups, *e.g.*, olefinic or carbonyl groups, the steric interactions prevent the coplanarity condition from being satisfied for either the diphenyl-type or the alternative mode of conjugation. Their spectra can be most simply accounted for on the basis of overlapping contributions from both the two potential types of conjugation which are possible in such compounds.

The reduction products of 2 : 2′-diacetyldiphenyl, and the dehydration products of 2 : 2′-di-(1-hydroxy-1-methylethyl)diphenyl derived from it,[1] form a series of 2 : 2′-disubstituted diphenyls, some of which are bridged. As in the case of the compounds discussed in

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* Part VII, *J.*, 1956, 4637.

[1] Hall, Ladbury, Lesslie, and Turner, *J.*, 1956, 3475.

Part V,[2] the contrast between the ultraviolet absorption characteristics of the bridged diphenyls and those with non-bridging 2 : 2′-substituents is striking, but in the present series the position is complicated by the presence in some compounds of additional ethylenic or ketonic double bonds conjugated with the diphenyl system. The degree of conjugation is subject to steric restrictions.

The compounds considered here can be divided into four groups (see p. 657).

(*a*) Unbridged compounds without additional conjugation : 2 : 2′-di-(1-hydroxyethyl)-diphenyl (I; R = Me, R′ = H); 2 : 2′-di-(1-hydroxy-1-methylethyl)diphenyl (I; R = R′ =Me) [see Fig. 1, which includes 2 : 2′-di(hydroxymethyl)diphenyl (I; R = R′ = H) for comparison].

(*b*) Bridged compounds without additional conjugation : 2 : 7-dihydro-2 : 7-dimethyl-3 : 4-5 : 6-dibenzoxepin (II; R = Me, R′ = H); 2 : 7-dihydro-2 : 2 : 7 : 7-tetramethyl-3 : 4-5 : 6-dibenzoxepin (II; R = R′ = Me) [see Fig. 2, which includes 2 : 7-dihydro-3 : 4-5 : 6-dibenzoxepin (II; R = R′ = H) for comparison]; 2-methyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-3 : 5-diene (III; R = H); 2 : 2 : 7-trimethyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-3 : 5-diene (III; R = Me) (for spectra, see ref. 1).

(*c*) Unbridged compounds with additional conjugation : 2 : 2′-divinyldiphenyl (IV; R = H); 2 : 2-di-(1-methylvinyl)diphenyl (IV; R = Me) (Fig. 3); 2 : 2′-diacetyldiphenyl (V) (Fig. 5).

(*d*) Bridged compounds with additional conjugation : 2-methyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-1 : 3 : 5-triene (VI; R = H); 2 : 7 : 7-trimethyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-1 : 3 : 5-triene (VI; R = Me); 3 : 8-dimethyl-4 : 5-6 : 7-dibenzo-1 : 2-diazocine (VIII) (Fig. 6).

The first two groups may be considered in the same way as corresponding compounds in Parts I [3] and V.[2] The increase in the size of substituent groups in the unbridged compounds is accompanied by an increase in the minimum angle possible between the ring planes. This results in a short-wave shift and decrease in intensity of the diphenyl-type conjugation band. The resolved long-wave fine-structure from the individual phenyl chromophores becomes correspondingly more prominent although its total intensity tends also to decrease owing to progressive reduction in overlapping by the conjugation band.

The steric effect of an aliphatic hydroxyl group in (I) appears to be almost the same as that of a methyl group similarly located, which might be expected (Fig. 1). The more polar nature of the hydroxyl group leads, however, to decreased resolution of vibrational fine-structure.

Connection of the 2 : 2′-positions by a saturated bridge, linking the benzene rings by a chain of three atoms, permits a closer approach to coplanarity and, in accordance with expectation for a purely steric effect, little difference is observed between the oxepins (II; see Fig. 2) and the bridged hydrocarbons (III). In these compounds the conjugation band is of the same order of intensity as in diphenyl itself (cf. Braude and Forbes [4]). The substitution respectively of single (II; R = Me, R′ = H; III, R = H) and paired (II; R = R′ = Me; III; R = Me) methyl groups on the bridge atoms adjacent to the benzene rings in both classes of compound alters the wavelength of the conjugation band in opposite directions; single methyl groups give a short-wave shift and paired methyl groups a long-wave shift, accompanied in the latter case by a perceptible reduction in intensity (Table 1; Fig. 2). These changes, although small, form a consistent pattern, and suggest that the bridge atoms and their substituent groups may be directly involved, through hyperconjugation, in the interaction between the phenyl groups, in addition to their influence on the angle between the benzene ring planes.

With the space-filling type of model [5] available to us, the angle between the phenyl

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[2] Beaven, Bird, Hall, Johnson, Ladbury, Lesslie, and Turner, *J.*, 1955, 2708.

[3] Beaven, Hall, Lesslie, and Turner, *J.*, 1952, 854.

[4] Braude and Forbes, *J.*, 1955, 3776.

[5] Settatree, Thomas, and Yardley, *Nature*, 1950, **166**, 59.

ring planes in the rigid oxepin-type structure is large (*ca.* 45°) and allows one methyl group to be substituted on each of the 2- and 7-carbon atoms, with a choice of approximately axial or equatorial conformations and no further departure from coplanarity. The tetramethyl derivative can also be built but there is, of course, no choice of conformation, and rotation of the methyl groups is restricted. In this connection, it may be noted that the model used by Braude and Forbes[4] to depict the oxepin structure with an interplanar angle of *ca.* 20° implies a completely unsymmetrical bridge, in which equality of the C–C–O bond angles cannot possibly be maintained.

It is also noteworthy that the intensities of the short-wave bands of both the oxepins and the dibenzo*cyclo*heptadienes are almost completely insensitive to the introduction of methyl groups on the bridge carbon atoms, although the tri-and tetra-methyl derivatives show an inflection in the 210—215 mμ region. Increasing substitution tends to result in the disappearance of the rudimentary long-wave inflection at 276—278 mμ, though this may be due to increasing overlapping as the conjugation band shifts to longer wavelengths.

FIG. 1.

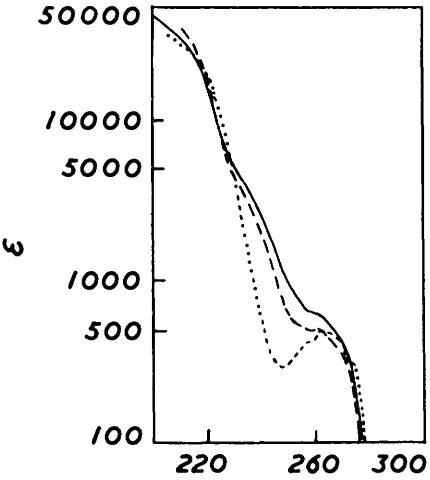

FIG. 2.

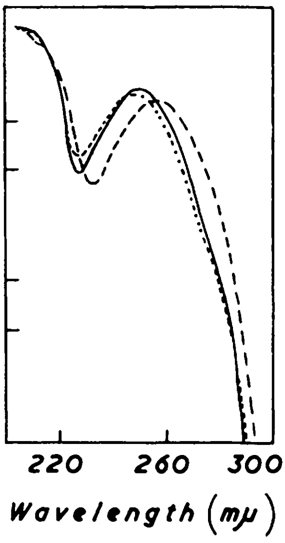

FIG. 3.

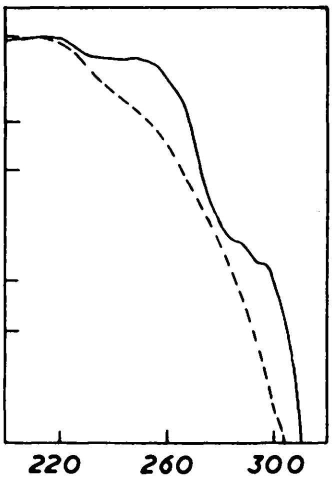

FIG. 1. ——, 2 : 2′-Di(hydroxymethyl)diphenyl (I; R = R′ = H); — — —, 2 : 2′-di-(1-hydroxyethyl)-diphenyl (I; R = Me, R′ = H); - - -, 2 : 2′-di-(1-hydroxy-1-methylethyl)diphenyl (I; R = R′ = Me). Solvent, 96% ethanol.

FIG. 2. ——, 2 : 7-Dihydro-3 : 4-5 : 6-dibenzoxepin (II; R = R′ = H); - - - -, 2 : 7-dihydro-2 : 7-dimethyl-3 : 4-5 : 6-dibenzoxepin (II; R = Me, R′ = H); — — —, 2 : 7-dihydro-2 : 2 : 7 : 7-tetramethyl-3 : 4-5 : 6-dibenzoxepin (II; R = R′ = Me). Solvent, 96% ethanol.

FIG. 3. ——, 2 : 2′-Divinyldiphenyl (IV; R = H); — — —, 2 : 2′-di-(1-methylvinyl)diphenyl (IV; R = Me). Solvent, light petroleum (b. p. 100—120°).

In Group (*c*) the steric conformations of two conjugated systems must be considered in each compound, the diphenyl system itself and the individual substituted benzene (styrene or acetophenone) systems. In this group the coplanarity condition cannot be approached for the diphenyl system in any of the three compounds, and for the substituted benzene systems departures from coplanarity will be considerable in the 1-methylvinyl derivative (IV; R = Me) and probably significant in the others.

The spectrum of 2 : 2′-divinyldiphenyl (IV; R = H; Fig. 3) indicates that bands due to styrene-like conjugation occur at *ca.* 290 mμ and at *ca.* 250 mμ, but the intensity is

TABLE 1.*

Compound (solvent in parentheses)		Short-wave band $\lambda_{\text{max.}}$	Short-wave band $\varepsilon_{\text{max.}}$	Minimum $\lambda_{\text{min.}}$	Minimum $\varepsilon_{\text{min.}}$	Conjugation band $\lambda_{\text{max.}}$	Conjugation band $\varepsilon_{\text{max.}}$	Minimum $\lambda_{\text{min.}}$	Minimum $\varepsilon_{\text{min.}}$	Long-wave features $\lambda_{\text{max.}}$	Long-wave features $\varepsilon_{\text{max.}}$
2 : 2′-Di(hydroxymethyl)diphenyl (EtOH)	I; R = R′ = H	(207)	35,500	—	—	(232)	4,500	—	—	(260) (268)	625 460
2 : 2′-Di-(1-hydroxyethyl)diphenyl (EtOH)	I; R = H, R = Me	(210)	37,000	—	—	(230)	4,450	258·5	495	260·5 (266)	512 420
2 : 2′-Di-(1-hydroxy-1-methylethyl)diphenyl (EtOH)	I; R = R′ = Me	(210)	29,500	—	—	—	—	247·5	295	(257) 262·5 (267) 273	425 500 455 320
· 2 : 7-Dihydro-3 : 4-5 : 6-dibenzoxepin (EtOH)	II; R = R′ = H	206·5	40,000	227	4,920	250·5	16,500	—	—	(*ca.* 277)	1,600
2 : 7-Dihydro-2 : 7-dimethyl-3 : 4-5 : 6-dibenzoxepin (EtOH)	II; R = H, R′ = Me	206·5	39,500	227	6,150	248·5	15,300	—	—	(*ca.* 276)	1,500
2 : 2 : 7 : 7-Tetramethyl-3 : 4-5 : 6-dibenzoxepin (EtOH)	II; R = R′ = Me	204·5 (213)	41,000 32,000	232	4,200	256·5	14,000	—	—	—	—
2-Methyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-3 : 5-diene (L.P.)	III; R = H	205·5	44,000	227	5,830	248	15,300	—	—	(*ca.* 276)	1,400
2 : 2 : 7-Trimethyl-3 : 4-5 : 6-dibenzo-*cyclo*hepta-3 : 5-diene (L.P.)	III; R = Me	205 (211)	45,000 37,500	229	5,350	250	14,200	—	—	(*ca.* 278)	1,500
2-Methyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-1 : 3 : 5-triene (L.P.)	VI; R = H	—	—	217	19,500	233·5 (238)	36,000 34,000	—	—	(255) (*ca.* 290)	11,000 800
2 : 7 : 7-Trimethyl-3 : 4-5 : 6-dibenzo-*cyclo*hepta-1 : 3 : 5-triene (L.P.)	VI; R = Me	(214)	16,700	219	15,700	236·5 241·5	37,500 37,000	—	—	(258) (*ca.* 295)	10,500 540
3 : 8-Dimethyl-4 : 5-6 : 7-dibenzo-1 : 2-diazocine (EtOH)	VIII	200—216	35,000	—	—	(*ca.* 238)	13,000	—	—	(*ca.* 268)	1,500

* Solvent 96% ethanol (EtOH) or light petroleum (b. p. 100—120°) (L.P.); wavelengths in $m\mu$; values in parentheses denote inflections.

TABLE 2.†

Compound (solvent in parentheses)		Short-wave band $\lambda_{\text{max.}}$	Short-wave band $\varepsilon_{\text{max.}}$	Minimum $\lambda_{\text{min.}}$	Minimum $\varepsilon_{\text{min.}}$	Conjugation band $\lambda_{\text{max.}}$	Conjugation band $\varepsilon_{\text{max.}}$	Long-wave features $\lambda_{\text{max.}}$	Long-wave features $\varepsilon_{\text{max.}}$
Styrene [6] (2 : 2 : 4-trimethylpentane)	—	—	—	219·5	3,900	(243) 248 (256·5)	15,500 15,800 9,800	273·5 281·5 290·5	780 790 615
2 : 2′-Divinyldiphenyl (L.P.)	IV; R = H	215 (235)	34,000 25,300	243	24,800	248 (253) (262)	25,000 24,000 17,000	(286) (296)	1870 1340
α-Methylstyrene (L.P.)	—	203·5	22,000	220·5	4,350	242·5	10,900	(277) (285) (289)	320 147 100
2 : 2′-Di-(1-methylvinyl)diphenyl (L.P.)	IV; R = Me	(213)	33,000	—	—	(245)	13,300	(301·5)	150
Acetophenone (EtOH)	—	—	—	216·5	1,760	242·5	12,700	279·5 (310)	1050 67
2 : 2′-Diacetyldiphenyl (EtOH)	V	(220)	27,000	—	—	(244)	13,800	291 (*ca.* 325)	3100 410
1 : 3-Diphenylpropene (L.P.)	VII	(203)	30,000	226·5	5,750	251 (263)	17,500 11,500	(276) 284·5 293	2350 1500 1020

† See footnote to Table 1.

significantly less than twice the value found for styrene itself.[6] Similarly, the inflection at 235 mμ may be ascribed to a severely hindered di-*ortho*-substituted diphenyl (cf. 2 : 2'-dialkyldiphenyls, Part VI [7]); its apparent high intensity must be partly determined by the overlapping bands at both longer and shorter wavelengths.

The spectrum of 2 : 2'-di-(1-methylvinyl)diphenyl (IV; R = Me) (Fig. 3) may be interpreted in a precisely similar manner. The additional steric requirements of the two methyl groups would be expected to result in a reduction of intensity of the band systems due to both types of conjugation. The difference between the spectrum of styrene and of α-methylstyrene (Fig. 4) is, in fact, accurately reflected in the spectra of the corresponding diphenyls, though the changes in diphenyl-type conjugation below 230 mμ are masked by heavy overlapping.

FIG. 4.

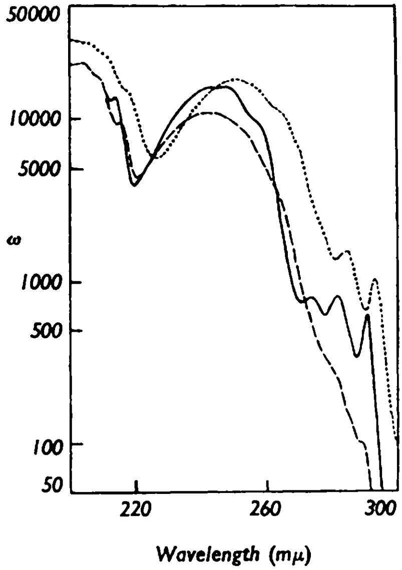

FIG. 5.

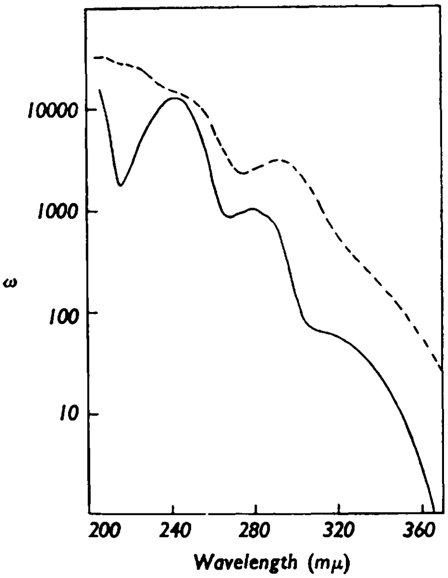

FIG. 4. ——, Styrene (solvent, 2 : 2 : 4-trimethylpentane; redrawn from ref. 6); — — —, α-methylstyrene; - - - -, 1 : 3-diphenylpropene (VII). Solvent, light petroleum (b. p. 100—120°).

FIG. 5. ——, Acetophenone; — — —, 2 : 2'-diacetyldiphenyl (V). Solvent, 96% ethanol.

The relationship of the spectrum of 2 : 2'-diacetyldiphenyl (V) to that of acetophenone (Fig. 5) is again very similar. The band systems of acetophenone are all represented, and the deep short-wave minimum at 216·5 mμ in acetophenone has evidently been obscured by the overlapping diphenyl-type conjugation band. It may be observed that the intensity of absorption of diacetyldiphenyl at 242·5 mμ, the wavelength of the conjugation band of acetophenone, is very much less than twice that of acetophenone. Since at this wavelength the diphenyl conjugation band probably contributes significantly to the total absorption, the diacetyldiphenyl may be regarded as an acetophenone showing a strong *ortho*-effect, to which its conjugation band is especially sensitive.[8]

A discussion of the spectra of compounds in this group on the basis of two separate types of conjugation appears to account for all the main features and to a large extent for

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[6] American Petroleum Institute, Research Project 44, Catalogue of Ultraviolet Spectral Data; serial no. 168, contributed by Shell Development Co.

[7] Everitt, Hall, and Turner, *J.*, 1956, 2286.

[8] Braude and Sondheimer, *J.*, 1955, 3754.

their intensities, although in 2 : 2′-diacetyldiphenyl the intensity of long-wave absorption above about 300 mμ is too great to be interpreted as that of two substituted acetophenones. Apart from this feature, however, it does not appear necessary to consider large contributions from new or totally conjugated systems. It is significant that both the types of conjugation are structurally possible and that both make appreciable contributions to the spectra, even though the coplanarity condition is not satisfied in either case (cf. Dewar [9]).

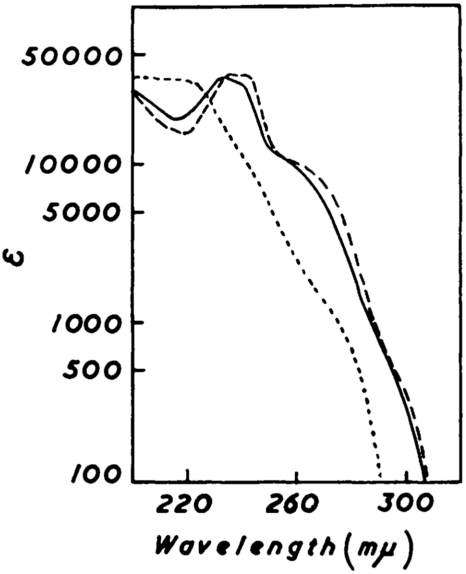
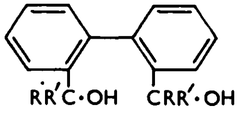


FIG. 6. ———, 2-Methyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-1 : 3 : 5-triene (VI; R = H); — — —, 2 : 7 : 7-trimethyl-3 : 4-5 : 6-dibenzo*cyclo*hepta-1 : 3 : 5-triene (VI; R = Me). Solvent, light petroleum (b. p. 100—120°). - - - -, 3 : 8-Dimethyl-4 : 5-6 : 7-dibenzo-1 : 2-diazocine (VIII). Solvent, 96% ethanol.

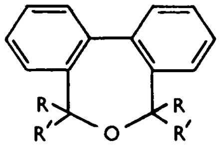
For the compounds of Group (*d*) it is less easy to find structural analogies. The introduction of a double bond into the C_3 bridge to give the dibenzo*cyclo*hepta-1 : 3 : 5-triene system (VI) results in a striking alteration in the spectrum when compared with the related dienes (III), notably the appearance of an intense band (with some fine-structure at the maximum) at *ca.* 235 mμ, accompanied by a prominent inflection at *ca.* 260 mμ and a much weaker long-wave inflection at *ca.* 290 mμ. The comparison strongly suggests that the band at *ca.* 235 mμ in the spectra of the trienes must be due to additional overall conjugation, resulting from the double bond in the bridge, and also the closer approach to coplanarity of the diphenyl structure. Molecular models indicate that the angle between the benzene ring planes is somewhat smaller in (III) than in (VI). Hyperconjugation *via* the bridge methylene group ($C_{(7)}$) does not appear to be an important factor, as the introduction of methyl groups at this position (VI; R = Me) only results in a small long-wave shift, with no serious intensity changes. In terms of the simple partial chromophore concept used above to discuss the spectra of the compounds of types (IV) and (V), the spectra of the dibenzo*cyclo*heptatrienes may be regarded as composed of overlapping contributions from the diphenyl-type conjugation through the collinear bond ($C_{(4)}$—$C_{(5)}$) and from a styrene-like chromophore involving the double bond of the bridge. The total absorption intensity in the *ca.* 240 mμ region is certainly far too high to be accounted for solely on the basis of styrene-like conjugation. The weak long-wave inflections are also consistent in location and intensity with contributions from a styrene system, though lacking the characteristic resolved fine-structure of the latter, which is known to be very sensitive to steric effects (Table 2, Fig. 4). Contributions from the individual phenyl groups acting as partial chromophores might be expected in the *ca.* 260—270 mμ region,[7] but because of their low intensity will be obscured by the intense contributions from the conjugated chromophores at this wavelength.

[9] Dewar, *J. Amer. Chem. Soc.*, 1952, **74**, 3345.

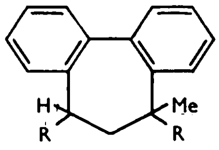
A partial structural analogue to (VI) is 1 : 3-diphenylpropene (VII). Its ultraviolet spectrum, which does not appear to have been previously reported, is shown in Fig. 4 (cf. Table 2); the possible configuration of this material is discussed in the Experimental section. Apart from a long-wave shift, its spectrum is very similar indeed, both qualitatively and quantitatively, to that of β-methylstyrene;[10] when compared with the spectra of the cyclic trienes (VI), the considerable differences emphasise the importance of the diphenyl-type conjugation through the collinear bond ($C_{(4)}$—$C_{(5)}$) in the latter group of compounds, despite the deviation from coplanarity of the diphenyl system.



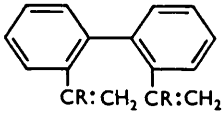
(I)



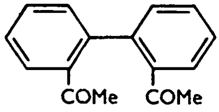
(II)



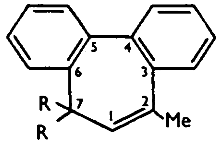
(III)



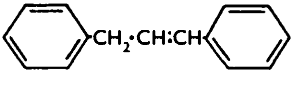
(IV)



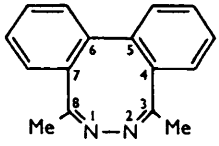
(V)



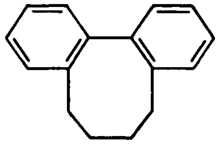
(VI)



(VII)



(VIII)



(IX)

(X) Ph·CH=N·N=CHPh

Ph·CH=NMe (XI)

The spectrum (Fig. 6, Table 2) of the cyclic azine (VIII) has few distinctive features. If the postulated structure is correct this particular type of eight-membered ring must be considerably puckered (cf. *cyclo*octatetraene[11]), and, to judge from the lack of a discrete conjugation band, probably more so than in the dibenzo*cyclo*octadiene derivatives (IX) described in Part V.[2] The result of introducing the additional C=N double bonds into this structure is difficult to predict, but in the *cyclo*heptatrienes (VI), which will be more nearly coplanar than the azine, the effect of a single C=C double bond in the bridge on the spectrum is very marked, as discussed above.

Comparison of the spectra[12] of benzylideneazine (X) and *N*-methylbenzylideneimine (XI) with that of the cyclic azine shows that conjugation of the C=N groups with the phenyl groups, either singly or as a pair, must be seriously inhibited, confirming the indications from a molecular model in which the two benzene rings are at an angle of *ca.* 60° with each other, and also at a considerable angle with the planes of the C=N bonds. The spectrum of the cyclic azine is therefore best accounted for on the basis that diphenyl-type conjugation through the collinear bond (VIII; $C_{(5)}$—$C_{(6)}$), conjugation through the azine bridge, and interaction of the C=N bonds with the adjacent phenyl groups, are all operative

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[10] Ref. no. 6, serial no. 121, contributed by Dow Chemical Co.

[11] Dunitz and Robertson, *Ann. Reports*, 1952, **49**, 372; McEwen and Longuet-Higgins, *J. Chem. Phys.*, 1956, **24**, 771.

[12] Cf. Braude, *Ann. Reports*, 1945, **42**, 126.

to some extent, and all affected by the general departure from coplanarity. The short-wave shift of the most intense region of absorption, relative to the *ca.* 240 mμ band of the cyclic trienes, may possibly be due to the methyl substituents on $C_{(3)}$ and $C_{(8)}$, as observed by Barany *et al.*[13] for linear diphenylazines. In the latter compounds the shift is accompanied by a marked reduction in intensity (which was not commented on by these authors), an effect which may also be operative in the cyclic azine. These two effects of alkyl substitution on the spectra of diphenylazines are in contrast with the general spectral similarities between diphenylpolyenes and the corresponding diphenylazines.

EXPERIMENTAL

The majority of the compounds examined were those prepared by Hall, Ladbury, Lesslie, and Turner.[1] The best sample of 2 : 2′-di-(1-methylvinyl)diphenyl (IV; R = Me) was found, by vapour-phase chromatography on a stationary phase of Apiezon M at 197°, to contain 7·5% by wt. of the tetramethyloxepin (II; R = R′ = Me); appropriate corrections were applied to the spectroscopic data. Acetophenone was purified by crystallisation and distillation (n_D^{20} 1·5332); α-methylstyrene was fractionated (b. p. 53·5°/14 mm., n_D^{20} 1·4391).

1 : 3-Diphenylpropene (VII) was prepared from phenylacetaldehyde, as described by Stoermer *et al.*[14] The product was recrystallised twice from ethanol at low temperature, followed by distillation (m. p. 14·5—15°, n_D^{20} 1·6015); it gave a single peak in vapour-phase chromatography under conditions (see above) which easily resolve the *cis-* and the *trans*-form of stilbene. The literature on this compound is confused. Tuot and Guyard [15] reported two isomers: (*a*) m. p. 51°, b. p. 159°/25 mm.; and (*b*) b. p. 178°/18 mm., n_D^{20} 1·5807, which were described as the *trans-* and the *cis*-form respectively. From its behaviour in vapour-phase chromatography, when compared with compounds of related structure, there is support for the conclusion that the present low-m. p. preparation is the *trans*-isomer. Serijan and Wise [16] obtained a fraction with m. p. 11·8°, n_D^{20} 1·6000, from the mixture of isomers formed by dehydration of 1 : 3-diphenylpropan-2-ol over alumina.

Absorption spectra were measured in a Unicam S.P.500 photoelectric spectrophotometer; observations at wavelengths shorter than 220 mμ were made in 1 mm. cells and at low optical densities to minimize errors due to stray radiation. The locations of fine-structure bands and inflections were confirmed photographically, using the logarithmic-cam, moving-plate method,[17] but the wavelengths listed in the Tables for inflections are the values obtained by direct examination of large-scale plots of the spectra.

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