

(ν_1 and ν_3 vibrations). The maximum at 630 cm^{-1} can be attributed to the ν_2 vibration, and the last two modes (525m , 460m) are probably due to the ν_4 vibration. The nondegeneracy of the ν_3 and ν_4 vibrations as well as the existence of a high intensity maximum at 960 cm^{-1} are in agreement with oxygen coordination,^{2,3} although the occurrence of a band at 1020 cm^{-1} (if not an overtone or a combination band) complicates the interpretation.

The ammonium ions are situated at the edges of the unit cell and are possibly connecting the structure in the directions of the *a* and *c* axes *via* hydrogen bonding.

As the superstructure reflections are very weak they may be due to the oxygen atoms only. On the other hand, the temperature factors for the sulphur and nitrogen atoms are high, although not unreasonable (*cf.* Table 1).

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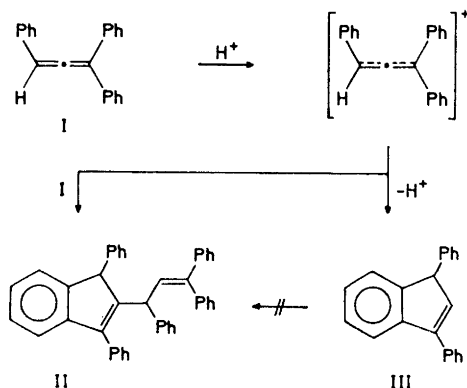
The Acid-catalyzed Reactions of Triphenylallene. A Reexamination

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Acid-catalyzed rearrangement of triphenylallene (I) has been reported to give a dimeric product, $\text{C}_{42}\text{H}_{32}$.¹ To this product, which also was formed by acid-catalyzed dehydration of 1,3,3-triphenylprop-2-en-1-ol and 1,1,3-triphenylprop-2-en-1-ol,^{2,3} Jones⁴ and Rewicki⁵ independently assigned structure II. In analogy with the formation of 1,3,3-triphenylindene from tetraphenylallene,⁶ 1,3-diphenylindene (III) was the expected product; this was isolated in 40% yield from the reaction of the above mentioned allylic alcohols with phosphorus pentoxide.² We want to report that compound III is also formed by treatment of triphenylallene with acid.

The reaction of I with HCl/acetic acid gave besides the dimer a small amount of another component which was isolated. The NMR spectrum showed a vinyl proton at δ 6.6 coupled to a methine proton at δ 4.6 ($J = 2\text{ Hz}$) and aromatic protons at δ 7.0–7.7. The mass spectrum (m/e 268) corresponded to the indene III. The isomeric 3,3-diphenylindene was not present.



The formation of the dimer has been suggested to proceed through the addition of an allylic carbonium ion to another

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molecule of triphenylallene;⁵ however, an electrophilic attack at the 2-position of the indene (III) cannot be excluded. In order to examine this possibility III was reacted with I in HCl/acetic acid. After 4 h at 100°C unreacted 1,3-diphenylindene was recovered quantitatively. Hence, the reaction takes place as previously suggested, which is not unexpected in view of the high reactivity of allenes towards electrophilic reagents.^{7,8}

In an attempt to raise the yield of III compared to the dimer, some experiments were performed. When a 0.1 M solution of triphenylallene in benzene was refluxed with *p*-toluenesulfonic acid, II and III were formed in a ratio of about 2:1. Since III is formed by a monomolecular mechanism, a dilute solution would be expected to prefer this reaction. This indeed was found to be true. With an actual concentration of triphenylallene in benzene less than 0.02 M, the indene (III) was isolated in 73 % yield and the dimer in 11 % yield.

Experimental. Triphenylallene (2.68 g, 0.01 mol) in benzene (500 ml) was added dropwise to a refluxing solution of *p*-toluenesulfonic acid (0.5 g) in benzene (300 ml) over a period of 1.5 h. After heating for another 0.5 hour, the solution was cooled, washed with aqueous Na₂CO₃, dried and evaporated. The residue was treated with hexane whereby most of the dimer precipitated, 0.30 g (11 %), m.p. 213–215°C (lit.⁵ m.p. 214–216°C). The filtered solution was chromatographed on neutral alumina (activity III) and the pure indene was eluted with hexane, 1.95 g (73 %), m.p. 68–69°C (lit.² m.p. 71–72°C).

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Complex Dibenzofurans

XV.* Mass Spectra of the Isomeric Benzobisbenzofurans

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The mass spectra of the five isomeric benzobisbenzofurans have been recorded and are now reported. The relative intensities of the most important peaks in the spectra are given in Table 1.

Table 1. Relative intensities of the main peaks in the mass spectra of the five isomeric benzobisbenzofurans.

<i>m/e</i>	Relative intensity (%)				
	I	II	III	IV	V
260	3.1	3.0	2.8	2.8	2.9
259	20.9	21.3	20.9	20.8	21.0
258	100.0	100.0	100.0	100.0	100.0
230	1.7	2.0	1.9	2.1	1.8
229	7.2	7.5	8.6	8.9	7.4
202	7.2	5.3	4.5	4.7	4.7
201	6.0	4.3	3.6	3.8	4.3
200	9.6	7.7	7.1	7.6	7.4
176	4.6	2.3	1.8	1.9	1.9
175	2.3	2.2	1.4	1.6	1.6
174	2.4	2.3	1.5	1.7	1.7
129.5	3.1	4.1	3.6	3.8	4.2
129	12.9	17.6	16.7	17.6	17.5
114.5	2.3	2.7	2.9	3.2	2.5
101.5	2.2	1.6	1.4	1.4	1.1
101	7.9	4.9	3.9	4.2	3.7
100.5	3.1	2.7	1.9	1.9	2.2
100	8.2	5.8	5.5	6.0	5.5
88.5	1.7	1.5	1.1	1.2	1.1
88	6.5	5.8	5.5	5.8	5.7
87.5	1.7	1.5	1.1	1.1	1.6
87	3.4	2.7	2.2	2.4	2.6
86.33	0.15	0.16	0.15	0.13	0.16
86	1.7	1.6	1.4	1.4	1.7
75	3.1	2.9	2.9	3.0	2.8
74	2.3	2.0	1.6	2.1	1.8
63	1.7	1.9	1.6	1.6	1.8
51	1.4	1.4	1.4	1.6	1.0
39	1.8	1.6	1.4	1.1	1.5

I, Benzobis[1,2-b:4,3-b']benzofuran; II, benzobis[1,2-b:3,4-b']benzofuran; III, benzobis[1,2-b:5,4-b']benzofuran; IV, benzobis[1,2-b:4,5-b']benzofuran; V, benzobis[1,2-b:6,5-b']benzofuran.

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