(ν₁ and ν₂ vibrations). The maximum at 630 cm⁻¹ can be attributed to the ν₂ vibration, and the last two modes (525m, 460m) are probably due to the ν₁ vibration. The nondegeneracy of the ν₂ and ν₁ vibrations as well as the existence of a high intensity maximum at 960 cm⁻¹ are in agreement with oxygen coordination,⁵,⁶ 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).

Acknowledgements. We are indebted to Professor Peder Kierkegaard and Professor Arne Magnus for their encouraging interest and many valuable discussions. We also wish to thank Dr. Sven Westman for his correction of the English of this paper. This investigation has been performed with financial support from the Tri-Centennial Fund of the Bank of Sweden and from the Swedish Natural Science Research Council. Financial aid from the Neste Oy Foundation for one of us (L. N.) is gratefully acknowledged.


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The Acid-catalyzed Reactions of Triphenyllallene. A Reexamination

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Acid-catalyzed rearrangement of triphenyllallene (I) has been reported to give a dimeric product, C₆H₁₂.¹ 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,¹,² Jones ¹ and Rowicki ³ independently assigned structure II. In analogy with the formation of 1,3,3-triphenyllindene from tetrathenyllallene,⁴ 1,3-diphenyllindene (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 triphenyllallene 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 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-diphenyllindene was not present.

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molecule of triphenyllene; 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 reac-
tion takes place as previously suggested,
which is not unexpected in view of the high
reactivity of allenes towards electrophilic re-
agents.5,6

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

Experimental. Triphenyllene (2.68 g, 0.01
mol) in benzene (500 ml) was added dropwise
to a refluxing solution of p-toluenesulphonic acid
(0.5 g) in benzene (300 ml) over a period of 1.5
h. After heating for another 0.5 hour, the solu-
tion 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.6 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.6 m.p. 71–72°C).

1. Jacobs, T. L., Dankner, D. and Singer, S. Tetra-
hedron 20 (1964) 2177.
57 (1924) 1083.
Rend. 238 (1954) 861.
6. Vorländer, D. and Siebert, S. Ber. 39 (1906)
1024.

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Complex Dibenzofurans

XV.* Mass Spectra of the Isomeric
Benzobisbenzofurans

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Sweden

The mass spectra of the five isomeric
benzobisbenzofurans have been recorded
and are now reported. The relative intensi-
ties 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 benzo-
abisbenzofurans.

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