

The corresponding dimer, $C_{24}H_{16}$, of acenaphthylene, $C_{12}H_8$, (I) has now been examined. The X-ray data for the two isomers obtained by irradiation of acenaphthylene (Dziewonski & Rapalski, 1912) are as follows:

Melting point ($^{\circ}$ C.)	307	234
Crystal system	Monoclinic	Monoclinic
a (A.)	7.81 ± 0.02	9.91 ± 0.05
b (A.)	4.86 ± 0.01	13.81 ± 0.05
c (A.)	20.16 ± 0.05	12.14 ± 0.06
β ($^{\circ}$)	92 ± 0.5	106 ± 1.0
Space group	$P2_1/n$	$P2_1/n$
Density at 18° C. (g.cm. $^{-3}$)	1.31	1.25

The observed densities are consistent with the unit cells containing two and four molecules of dimer respectively, and it follows that the high-melting crystals

plane, with the condensed planar systems of five- and six-membered rings inclined at about 45° to (010). The sides of the four-membered ring, even in projection, appear to be somewhat longer than 1.54 A., but accurate values are not yet available. Certain distortions from

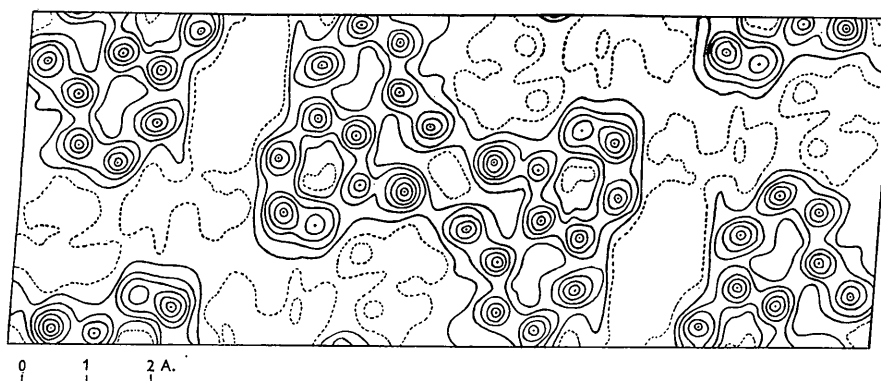
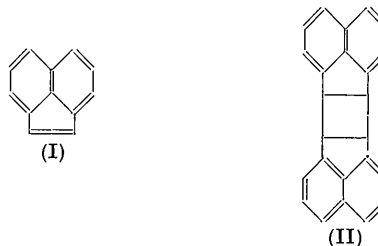


Fig. 1. Acenaphthylene dimer: Fourier projection on (010), showing one unit cell.

contain centrosymmetric molecules of the dimer. No molecular symmetry is imposed crystallographically on the low-melting isomer.

The structure determination of the centrosymmetric isomer has been commenced. Trial-and-error methods have been employed to fix the signs of the ($h0l$) reflexions and a Fourier projection on (010) has been calculated and is shown in Fig. 1. There is no doubt that the molecule is dinaphthylene cyclobutane (II), in confirmation of the conclusions, based on chemical evidence, of Dziewonski & Rapalski (1912).

The atomic positions cannot yet be fixed with any precision, but certain features of the structure seem clear. The four-membered ring must lie almost in the (010)

regularity also appear to exist in the naphthylene groups.

Work is being continued with a view to obtaining more accurate values for the dimensions of the molecule.

We wish to thank Mrs D. Hodgkin (Dorothy Crowfoot) for her continued interest.

References

- DUNITZ, J. D. (1949). *Acta Cryst.* **2**, 1.
 DZIEWONSKI, K. & RAPALSKI, R. (1912). *Ber. dtsh. chem. Ges.* **45**, 2491.
 FULTON, J. D. & DUNITZ, J. D. (1947). *Nature, Lond.*, **160**, 161.

Acta Cryst. (1949). **2**, 63

A preliminary X-ray investigation of some pyridindene derivatives. By C. H. CARLISLE and S. FURBERG.
Biomolecular Research Laboratory, Birkbeck College, London E.C. 4, England

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2-Methyl-9-phenyl-2, 3, 4, 9-tetrahydro-1-pyridindene (I), or thephorin (T.M. Reg. U.S. Pat. Off.), is reported to be a useful drug in hay fever and other allergic disorders. Closely related compounds (e.g. II), apparently only differing from thephorin in the position of a double bond, show very low biological activity. Samples of these interesting compounds were kindly sent us from the research laboratories of Hoffmann La Roche, Inc., Nutley,

N.J., where they were synthesized by Dr J. Plati and Dr W. Wenner.

We have carried out a preliminary X-ray investigation of four of the substances. They are: (1) thephorin (I), (2) the thiocyanate salt of thephorin, (3) 4a-9a-dihydroxy-dihydro-thephorin, and (4) the nitrate salt of a base, which is isomeric with thephorin and probably has formula (II).

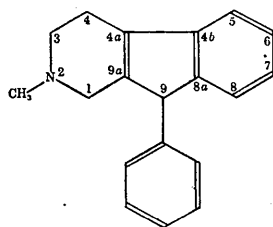
(1) and (2) and other derivatives with the ring system (I) are biologically active, whilst (4) is nearly inactive.

X-ray data

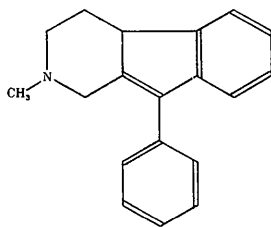
X-ray single-crystal photographs were taken, and the density was determined in a mixture of CCl_4 and C_6H_6 . The results of the measurements are:

Compound	Space group	<i>a</i> (Å.)	<i>b</i> (Å.)	<i>c</i> (Å.)	β (°)	Density (g.cm. ⁻³)
(1) Thephorin	$P2_1/c$	5.53	9.37	31.2	115	1.17
(2) Thiocyanate	$P2_1/n$	10.68	10.74	14.80	100	1.27
(3) Dihydroxy	$P2_1/c$	12.85	8.30	14.64	99	1.26
(4) Nitrate	Aa	8.24	10.94	18.58	92	1.28

The compounds all have four molecules per unit cell (calculated 3.96–3.99).



I
Thephorin



II

Morphology and optics

Each compound was examined under the polarizing microscope with the following result:

(1) Thephorin

Small, monoclinic needles elongated along [100], bounded by {001} and {011}.

$\beta \parallel b$; γ about 5° from *c*. Negative. Optic axial angle medium.

(2) Thephorin thiocyanate

Monoclinic prisms elongated parallel to [010]. Forms {001}, {101} and {10 $\bar{1}$ } developed, the last one predominant.

$\beta \parallel b$; α and γ approximately parallel to *a* and *c* respectively. Positive. $2E \simeq 80^\circ$.

(3) Dihydroxy-dihydro-thephorin

Well-developed monoclinic prisms, elongated along [010], bounded by {100}, {001} and {10 $\bar{2}$ }.

$\alpha \parallel b$; γ lies about 25° from *c* in the acute angle. Negative. $2V \simeq 70^\circ$.

(4) Nitrate of isomeric thephorin

The crystals occur either as nearly hexagonal prisms elongated along *a*, or in a scalenohedral-like habit.

$\gamma \parallel b$; α lies 20° from *a* in the obtuse angle. Negative. Optic axial angle very small.

Discussion

In thephorin the birefringence is negative, the α -vibration direction is nearly perpendicular to the *a* plane, the *a* axis is short, and the 100 reflexion is strong. This suggests that the molecules are lying approximately parallel to the *a* plane.

The nitrate derivative is interesting crystallographically, as it has strongly pseudohexagonal symmetry. This is shown by the X-ray diffraction pattern on the $0kl$ zone, as well as by the optical and morphological properties, which are nearly those of a uniaxial crystal.

A model of the main ring-systems (I and II) based on the chemically established formulae and accepted bond distances can be represented roughly by a very flat trigonal prism with sides about 9Å. This general shape and size of the molecules is confirmed by the investigation. Thephorin shows that the three six-membered rings in the molecule cannot be much tilted in relation to each other, and the hexagonal character of the nitrate derivative is probably related to the apparent threefold symmetry of the molecules.

However, the differences in biological activity between these closely related compounds appear to be linked with differences in stereochemical detail. A complete X-ray analysis of one or more of the substances is therefore needed before an attempt can be made to correlate physiological activity with stereochemical configuration. Crystallographically thephorin suggests itself for this purpose.

Reference

LEHMANN, G. (1948). *J. Pharmacol.* **92**, 249.

Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. Copy should be sent direct to the British Co-editor (R. C. Evans, Crystallographic Laboratory, Cavendish Laboratory, Cambridge, England).

International Union of Crystallography

Further formal notification of adhesion has been given, as follows:

On 14 December 1948 by the Netherlands.

On 15 December 1948 by Australia through the Australian National Research Council.

On 23 December 1948 by France through the Académie des Sciences de l'Institut de France.