

which were later shown to have resulted from the assumption of incorrect structures. Unfortunately, it is impossible to assess how many additional false Fouriers are at present lying undetected there. In the case of 2-phenylazulene, the Fourier evidence for the ordered structure is apparently strengthened by the least squares results, but in view of the experiences reported for azulene itself* it would be rash indeed to conclude that the structure of the former has been established. The principal conclusion which should be drawn, we feel, is that structures based on incomplete data, in this case a single, albeit well-resolved, projection, should be accepted with extreme caution, whether or not unusual structural features turn up, regardless of how free the electron density distribution is from false detail, and corroboration by least squares notwithstanding.

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J. DONOHUE
B. D. SHARMA

Department of Chemistry,
University of Southern California,
Los Angeles.

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Crystal Structure of a Nitrogen Isostere of Pentacyclo-Octacosadodecaene

A NITROGEN isostere of pentacyclo-octacosadodecaene, namely, 2,8,14,20 tetraazapentacyclo [19,3,1,1³,7,1⁹,13,1¹⁵,19] octacosadodecaene, recently prepared in these laboratories, has a structural formula as shown in Fig. 1 and has been examined by optical and X-ray diffraction methods. Crystals were grown from the mother liquid in bunches of long, thin needles, up to 1 cm in length and with a cross-section of 0.1–0.3 mm. The prism faces showed well-developed forms of {100} and {110} with all interfacial angles, constant within experimental error, of 45°. Frequently the form {110} showed the greater development, but pinacoid and pyramidal faces were not observed.

In polarized light, straight extinction was observed on all prism faces. No cleavage was found perpendicular to the needle axis and only with difficulty could a near basal section be cut which gave an interference figure of a uniaxial crystal of negative optic sign. The birefringence was high and the refractive indices were measured as $\omega > 1.844$ and $\epsilon = 1.594$.

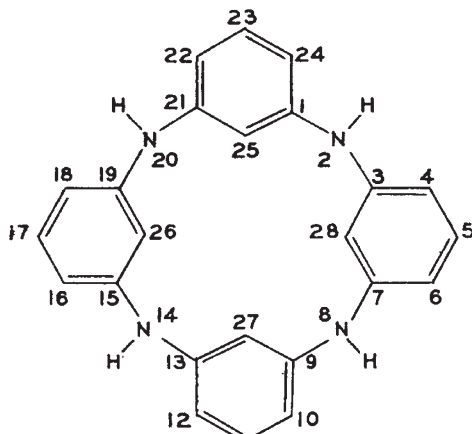


Fig. 1

Single-crystal zero and upper layer equi-inclination Weissenberg photographs about the *c* axis showed diffraction symmetry $4/mmm$, and from zero layer Weissenberg photographs about the *a* and *c* axes the following unit cell parameters were obtained: $a = 20.69 \pm 0.02$ Å, $c = 8.69 \pm 0.02$ Å; density measured by flotation = 1.297 ± 0.006 g/c.c.; No. of molecules/cell = 8; theoretical density = 1.301 g/c.c.

No systematic absences were observed for the hkl reflexions but systematic absences were observed for $0kl$ with *l* odd and hhl with *l* odd. The space-group is therefore either $P4cc$ or $P4/mcc$. A crude test for pyroelectricity was negative and the distribution of the $0kl$ intensities favoured the centric curve. The distribution of the $hk0$ intensities lay between the centric and hypercentric distributions.

In its unstrained state the molecule is not flat and from general packing considerations it seems very unlikely that the eight molecules could be sited in the centrosymmetric space group. The Patterson projection $P(v,w)$ calculated using 114 independent $0kl$ reflexions showed features that were not consistent with the presence of a mirror plane. The Patterson projection down the *c* axis was also calculated using 443 $hk0$ reflexions. This map was too difficult to interpret, though the distribution of the peaks indicated that the molecules were lying in general (*x,y*) positions and that many vectors, obviously of intermolecular origin, were approximately of the type $(\frac{1}{2},x)$, $(x,\frac{1}{2})$ and $(\frac{1}{2},\frac{1}{2})$. This is not surprising as the diameter of the molecule, excluding the hydrogen atoms, is about 10 Å or roughly half the *a* axis.

In view of this evidence and the absence of any cleavage it would appear that the space group is probably $P4cc$. The first ten orders of the $h00$ and $0k0$ reflexions showed odd orders were very weak and even orders very strong and, from packing models, the most likely sites for the molecules which allow sufficient Van der Waals separation is for the centres of the molecules, projected down the *c* axis, to be close to the positions $(\frac{1}{2},\frac{1}{2})$, $(\frac{3}{4},\frac{1}{4})$, $(\frac{1}{4},\frac{3}{4})$ and $(\frac{3}{4},\frac{3}{4})$ with an approximate orientation as shown in Fig. 2. The positions of the other four molecules are obtained by the *c* glide operation so that the benzene rings of the upper molecule lie between the rings of the lower molecule. It would seem that the high symmetry of the molecule itself is not used within the crystal.

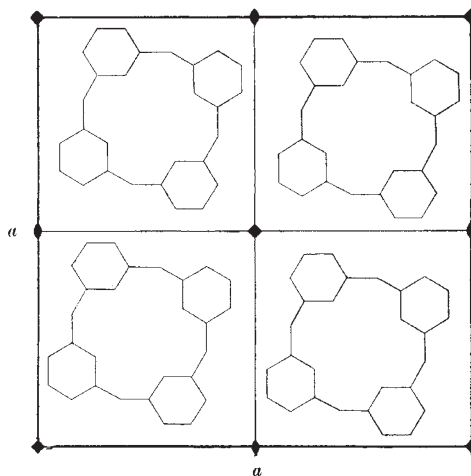


Fig. 2

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G. W. SMITH

British Petroleum Co., Ltd.,
Petroleum Division,
BP Research Centre,
Sunbury-on-Thames.