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THE STRUCTURE OF ARSANTHRENE (9,10-DIARSAANTHRACENE)

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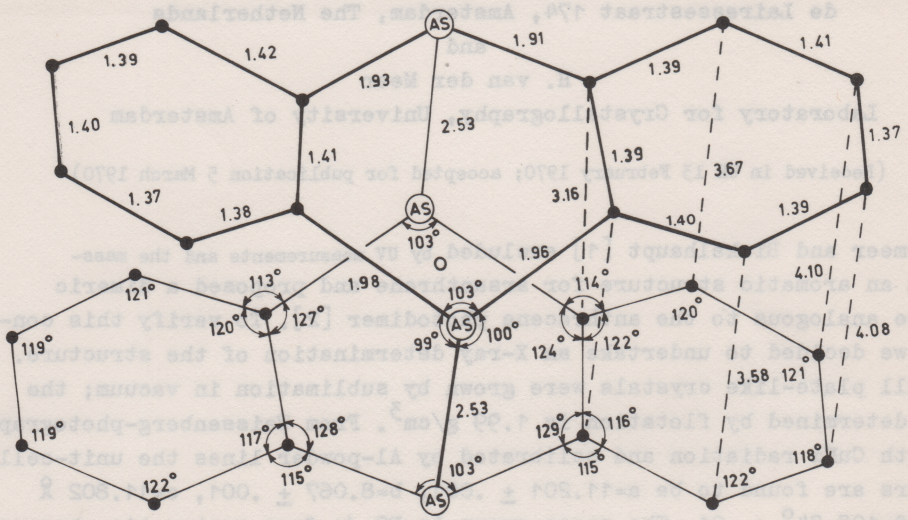
Vermeer and Bickelhaupt [1] excluded by UV measurements and the mass-spectrum an aromatic structure for arsanthrene and proposed a dimeric structure analogous to the anthracene photodimer [2]. To verify this conclusion we decided to undertake an X-ray determination of the structure.

Small plate-like crystals were grown by sublimation in vacuum; the density determined by flotation is 1.99 g/cm^3. From Weissenberg-photographs taken with CuKα radiation and calibrated by Al-powder lines the unit-cell parameters are found to be a=11.201 ± .001, b=8.067 ± .001, c=11.802 Å ± .001, β=108.84° ± .01. The space group is $P2_1/c$ from systematic absences. There are 4 $C_{12}H_8As_2$-units in the cell. With an automatic Nonius diffractometer 989 independent intensities were collected; CuKα radiation was used. The data were corrected with Lorentz-polarization factors, but not for absorption. The positions of the two independent As atoms were found from a threedimensional Patterson synthesis. A subsequent electron density map with phases based on the As contributions revealed the positions of the carbon atoms. The structure was refined by a block-diagonal least-squares program with anisotropic temperature parameters for the As atoms and isotropic ones for the carbon atoms to an R index of 6.5%. From a final difference electron density map an appreciable influence of absorption was found. The standard deviations in distances and angles are therefore estimated as .02Å and 1.5°, respectively. Only vague indications for the hydrogen positions could be obtained.

The result of the structure determination is presented in the figure. Arsanthrene is indeed a dimer, as predicted [1]. In the crystal these dimers are situated around centres of symmetry (small circle in the middle of the figure). The four As atoms form an exact rectangle, the angles being equal to 90° within the standard deviation. The two As atoms of one

arsanthrene unit lie in planes with each of the neighbouring benzene rings; these two planes include an angle of 23°. The central part of the unit has the boat conformation; the tips of the boat, i.e. the two C-As-C planes, make angles of 15° with the plane of the four central



carbon atoms. The C-As-C-angle is 103°, about the same value as found in $AsCl_3$, $As(CF_3)_3$, etc. [3]. A list of atomic coordinates can be obtained from the first author.

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[1] H. Vermeer and F. Bickelhaupt, Tetrahedron Letters, in Press.
[2] J. Hengstenberg and J. Palacios, An. Soc, Esp. de Fys. Y. Quim 30, 5 (1932).
[3] Tables of interatomic distances and configuration in molecules and ions, L.E. Sutton editor The Chemical Society, London (1965).

Final coordinates and thermal parameters

	x	y	z	B(iso)
As-1	0.03683	0.03647	0.69866	——
As-2	0.03093	0.23225	0.05265	——
C-1	0.2191	0.0235	0.3398	2.79
C-2	0.1210	0.0677	0.3813	2.25
C-3	0.3428	0.0853	0.3904	4.04
C-4	0.7158	0.0892	0.2642	3.59
C-5	0.8427	0.1063	0.3395	2.95
C-6	0.1485	0.1795	0.4759	2.01
C-7	0.3686	0.1925	0.4848	3.20
C-8	0.6182	0.1831	0.2797	3.97
C-9	0.8641	0.2214	0.4338	2.25
C-10	0.2714	0.2618	0.0281	2.56
C-11	0.6465	0.2943	0.3754	4.03
C-12	0.7678	0.3153	0.4497	3.16

	B-11	B-22	B-33	B-12	B-23	B-13
As-1	0.0069	0.0097	0.0037	-0.0017	-0.0012	0.0017
As-2	0.0066	0.0076	0.0045	0.0003	0.0013	0.0027