

& Nereson, 1968). The tetragonal structure, however, is different from that of  $UC_2$ ,  $LaC_2$  and other known tetragonal dicarbides (Atoji, 1961) where the  $C_2$  groups are aligned parallel to the  $c$  axis. The monoclinic structure is also unique among dicarbide structures.

The actual C–C distances in the high temperature phases are probably larger than the observed values, owing to the effect of thermal motion of the carbon atoms. Thus it would appear that the C–C distance is essentially constant at about 1.30–1.35 Å in all three phases, in good agreement with the other group 3A dicarbides.

#### References

- ATOJI, M. (1961). *J. Chem. Phys.* **35**, 1950.  
 BACON, G. E. (1962). *Neutron Diffraction*. Oxford: Clarendon Press.  
 BOWMAN, A. L., ARNOLD, G. P., WITTEMAN, W. G., WALLACE, T. C. & NERESON, N. G. (1966). *Acta Cryst.* **21**, 670.  
 BOWMAN, A. L., KRİKORIAN, N. H., ARNOLD, G. P., WALLACE, T. C. & NERESON, N. G. (1968). *Acta Cryst.* **B24**, 459.  
 BOWMAN, A. L., WALLACE, T. C., YARNELL, J. L., WENZEL, R. G. & STORMS, E. K. (1965). *Acta Cryst.* **19**, 6.  
 BOWMAN, M. G., HULL, D. E., WITTEMAN, W. G., ARNOLD, G. P. & BOWMAN, A. L. (1966). *Rev. Sci. Instrum.* **37**, 1543.  
 CHIOTTI, P., KORBITZ, F. W. & DOOLEY, G. J., III (1967). *J. Nucl. Mat.* **23**, 55.  
 ELLIOTT, N. & HASTINGS, J. (1961). *Acta Cryst.* **14**, 1018.  
 HILL, N. A. & CAVIN, O. B. (1964). *J. Amer. Ceram. Soc.* **47**, 360.  
 HUNT, E. G. & RUNDLE, R. E. (1951). *J. Amer. Chem. Soc.* **73**, 4777.  
 LANGER, S., BALDWIN, N., GANTZEL, P., KESTER, F. & HANCOCK, C. (1964). *Nuclear Metallurgy, Vol. X. Compounds of Interest in Nuclear Reactor Technology*, p. 359. The Metallurgical Society of American Inst. of Mining, Metallurgical and Petroleum Engineers.  
 RUPERT, G. N. (1965). *Rev. Sci. Instrum.* **36**, 1629.  
 SATOW, T. (1967). *J. Nucl. Mat.* **21**, 249.  
 SEQUEIRA, A. (1965). *Acta Cryst.* **18**, 291.  
 SIDHU, S. S., HEATON, L., ZAUBERIS, D. D. & CAMPOS, F. P. (1956). *J. Appl. Phys.* **27**, 1040.  
 WILHELM, H. A. & CHIOTTI, P. (1950). *Trans. Amer. Soc. Metals*, **42**, 1295.

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## The Crystal Structure of the Dimer of 9-Anthraldehyde

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The structure of the dimer of 9-anthraldehyde has been determined by means of Patterson projections and three-dimensional least-squares refinements. The aldehyde groups are in the *trans* configuration.

Crystals of the dimer of 9-anthraldehyde,  $[(C_{14}H_9)CHO]_2$ , were prepared by P. Sarti-Fantoni, the dimerization taking place in the solid state by the action of ultraviolet radiation on the monomer. Studies of the infrared spectra (Craig & Sarti-Fantoni, 1966) indicated the *trans* configuration of the CHO groups with respect to the anthracene skeleton, although from the monomer structure a *cis* dimer formation might be expected. The structure determination was undertaken in order to confirm the *trans* configuration of the CHO groups with respect to the anthracene.

#### Crystal data

$a = 9.62$ ,  $b = 7.86$ ,  $c = 8.81$  Å  $\alpha = 59^\circ 40'$ ,  $\beta = 90^\circ 0'$ ,  
 $\gamma = 65^\circ 56'$

Density, measured =  $1.346$  g.cm<sup>-3</sup>

Density, calculated =  $1.34$  g.cm<sup>-3</sup>

Systematic absences: none

Space group  $P\bar{1}$

Limited three-dimensional data were collected ( $0kl \rightarrow 7kl, hk0$ ), the integrated intensities being read

on a Joyce–Loebl flying-spot microdensitometer and corrected by the appropriate geometrical correction factors. The structure was solved from  $h0l$  and  $hk0$  Patterson projections and refined with the use of block-diagonal least-squares calculations on 640 observed reflexions. With isotropic temperature factors a discrepancy index of 10.9% was obtained; by using anisotropic temperature factors for the carbon and oxygen atoms, it was reduced to 8.8%. Table 1 gives the final positional and thermal parameters. The anisotropic temperature factors are not regarded as significant because of the paucity of the data.

The dimer molecules lie on centres of symmetry and hence the CHO groups must be in the *trans* configuration with respect to the anthracene skeleton. Bond lengths and angles have normal values within the accuracy of the structure determination. The bond length between C(9) and C(10') connecting the two half-molecules is 1.61 Å, the same as found in dipara-anthracene (Ehrenberg, 1966a). Each half-molecule is bent through an angle of  $2 \times 23^\circ$ , again similar to dipara-anthracene and the photo-isomer of bi(anthracene-9,10-dimethylene) (Ehrenberg, 1966b).

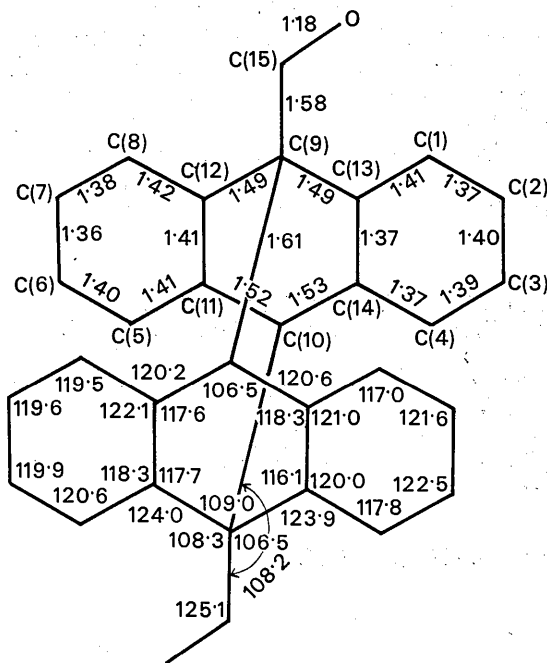
Fig. 1 is a schematic drawing of the molecule showing bond lengths and angles. The standard de-

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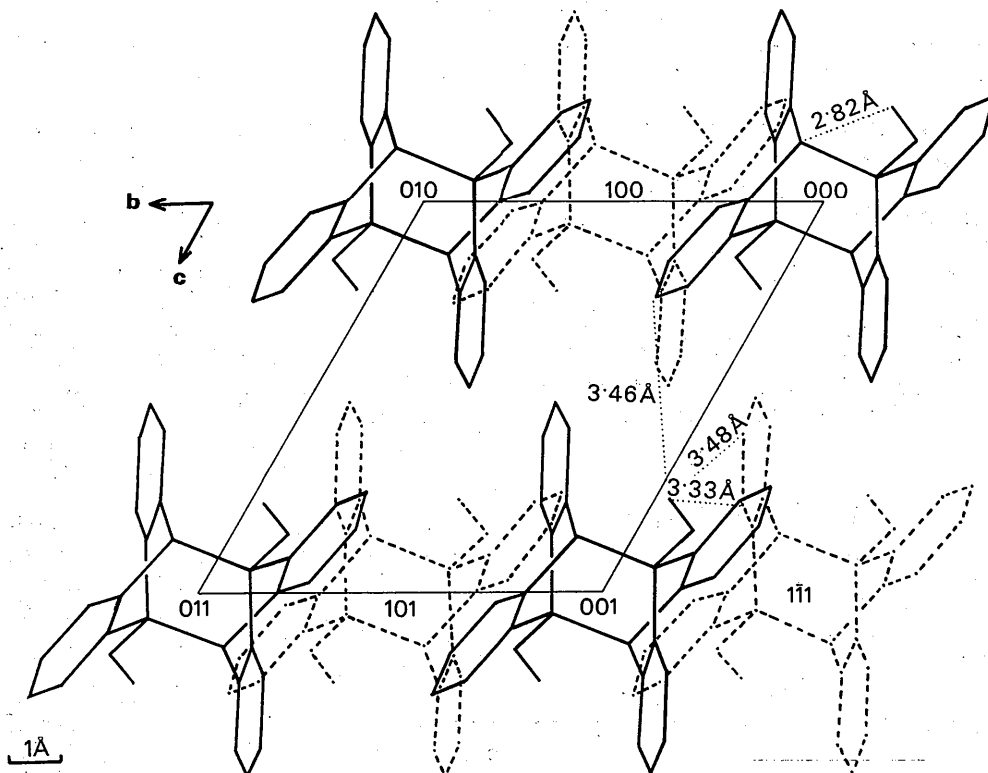
Table 1. Positional and thermal parameters

	x	y	z	$U_{iso}$
	(Å)	(Å)	(Å)	
C(1)	0.773	1.319	2.172	0.0372
C(2)	2.149	1.252	2.460	0.0348
C(3)	3.134	1.053	1.500	0.0330
C(4)	2.705	0.930	0.263	0.0285
C(5)	-1.349	3.137	-3.842	0.0284
C(6)	-2.904	4.349	-4.513	0.0348
C(7)	-3.860	4.517	-3.576	0.0349
C(8)	-3.366	3.539	-1.926	0.0290
C(9)	-1.099	1.168	0.574	0.0257
C(10)	0.854	0.800	-1.329	0.0259
C(11)	-0.806	2.155	-2.157	0.0207
C(12)	-1.798	2.327	-1.200	0.0197
C(13)	0.337	1.190	0.919	0.0219
C(14)	1.326	0.989	0.002	0.0159
C(15)	-2.254	2.014	1.339	0.0351
O(16)	-2.907	1.356	2.213	0.0628
H(17)	0.023	1.314	2.979	0.0461
H(18)	2.487	1.368	3.437	
H(19)	4.223	0.992	1.738	
H(20)	3.475	0.784	-0.507	
H(21)	-0.591	2.937	-4.585	
H(22)	-3.319	5.201	-5.831	
H(23)	-5.054	5.475	-4.185	
H(24)	-4.144	3.682	-1.164	
H(25)	-2.548	3.331	1.013	
H(26)	1.563	1.021	-2.154	

In the monomer of 9-anthraldehyde the angle between the anthracene and the aldehyde planes is  $27^\circ$  (Trotter, 1959) and a close contact of  $3.06 \text{ \AA}$  is ob-

Fig. 1. Bond distances (Å) and bond angles ( $^\circ$ ).

viations of the bond lengths vary between  $0.011$  and  $0.014 \text{ \AA}$  and for the angles between  $39'$  and  $56'$ . Fig. 2 shows the projection of the structure on to the  $bc$  plane.

Fig. 2. Projection of the structure on the  $bc$  plane.

served between the oxygen atom and C(1). In the dimer, however, the oxygen of the aldehyde group is closest to the central carbon in the second half of the dimer molecule, C(10'), and the angle defined above is  $85^{\circ}30'$  and the distance is  $2.82 \text{ \AA}$  (Fig. 2).

The closest contacts to the oxygen atom at the equivalent point (000) are:

- 3.33  $\text{\AA}$  to C(8) at the equivalent point (1 $\bar{1}$ 0),
- 3.46  $\text{\AA}$  to C(2) at the equivalent point (001),
- 3.48  $\text{\AA}$  to C(7) at the equivalent point (1 $\bar{1}$ 0),

indicating that the molecules are held together by van der Waals forces only.

### References

- CRAIG, D. P. & SARTI-FANTONI, P. (1966). *Chem. Comm.* **20**, 742.  
 EHRENBERG, M. (1966*a*). *Acta Cryst.* **20**, 177.  
 EHRENBERG, M. (1966*b*). *Acta Cryst.* **20**, 183.  
 TROTTER, J. (1959). *Acta Cryst.* **12**, 922.

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## A Refinement of the Crystal Structure of KSCN

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The crystal structure of KSCN has been redetermined in order to obtain more reliable bond distances for the thiocyanate ion. The C-S bond length was found to be  $1.689 \pm 0.013 \text{ \AA}$ , the C-N bond length  $1.149 \pm 0.014 \text{ \AA}$ , and the S-C-N angle  $178.3 \pm 1.2^{\circ}$ .

Two previous investigations of the crystal structure of potassium thiocyanate have been made (Klug, 1933; Büsser, Günther & Tubin, 1934). Both studies made use of zonal data and differed substantially in only the position of the carbon atom in the thiocyanate ion. Because of the inherent errors in results from projection data and the growing need for more accurate interatomic distances for theoretical purposes, it was decided to reinvestigate this structure.

Crystals of analytical grade were recrystallized from an alcohol-water solution. Two separate crystals, whose dimensions varied from 0.07 to 0.10 mm, were used to collect the data because the first began to decompose before the investigation was complete. Systematic extinctions ( $0kl$  for  $k=2n+1$  and  $h0l$  for  $l=2n+1$ ) agree with the previous space group assignments of *Pbcm*. Lattice constants, determined from precession photographs, are  $a=6.673 \pm 0.003$ ,  $b=6.715 \pm 0.003$ ,  $c=7.543 \pm 0.005 \text{ \AA}$ . Intensity data were collected on a Picker diffractometer equipped with a General Electric single-crystal orienter using Mo  $K\alpha$

radiation. Angle settings were calculated with an existing Fortran II program (Shoemaker, 1962). The Lorentz-polarization corrections were made but absorption effects were neglected. A three-dimensional Fourier synthesis (Sly, Shoemaker & Van den Hende, 1962), based on signs calculated from the positions of one of the previous investigations (Klug, 1933), gave the correct position for the carbon atom and improved the other positions. Anisotropic least-square refinement was carried out with the Busing, Martin & Levy (1962) program, and final values of  $R_1=0.080$  and  $R_3=0.067$  were obtained. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1962). Final parameters are listed in Table 1, with observed and calculated structure factors given in Table 2. The weighting scheme used is as follows:

$$\text{Weight} = 1/\sigma^2$$

$$\sigma^2 = F^2/4I^2[E + 2B + (0.1I)^2]$$

where  $F$  is the structure factor,  $I$  is the intensity,  $E$  is the total count and  $B$  is the background. Bond dis-

Table 1. *Final parameters and R values for KSCN*

Standard deviations are given in parentheses.

The  $\beta_{ij}$  are defined by:  $T = \exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl)$ .

$\beta_{13}$  and  $\beta_{23}=0$  by symmetry.

	$x$	$y$	$z$	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$
K	0.2059 (5)	0.25	0.0	0.0128 (8)	0.0089 (7)	0.0063 (5)	0.0
S	0.6033 (5)	0.1074 (5)	0.25	0.0085 (8)	0.0068 (9)	0.0068 (7)	-0.0008 (8)
C	0.7737 (19)	0.2919 (18)	0.25	0.0077 (32)	0.0132 (38)	0.0014 (21)	-0.0013 (31)
N	0.8934 (15)	0.4139 (16)	0.25	0.0094 (27)	0.0133 (31)	0.0061 (20)	0.0003 (30)

$$R_1 = \frac{\sum_{hkl} ||F_{\text{obs}}| - |F_{\text{calc}}||}{\sum_{hkl} |F_{\text{obs}}|} = 0.080$$

$$R_3 = \sqrt{\frac{\sum_{hkl} w(|F_{\text{obs}}| - |F_{\text{calc}}|)^2}{\sum_{hkl} w(F_{\text{obs}})^2}} = 0.067$$