

Acta Cryst. (1965). **19**, 681

The unit cell and space group of benzaldehyde phenylhydrazone, $C_6H_5.CH:N.NH.C_6H_5$. By R. H. DE VÈRE,
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(Received 16 February 1965)

Benzaldehyde phenylhydrazone was first prepared by Fischer (1877) who described the crystals as pale yellow needles which turned pink on exposure to air. From the measurement of interfacial angles Fischer deduced that the crystals were monoclinic, elongated along the c axis, had axial ratios $a:b:c=0.853:1:0.670$, and $\beta=87^\circ 40.5'$. The melting point was $151^\circ C$.

In the present work crystals were prepared by condensation in alcohol (Fischer) and acetic acid (Mann & Saunders 1954). In both cases the crystals were pale yellow needles, elongated along the a axis, which turned a deep red on exposure to sunlight. After several days in the dark the crystals faded to a pale pink. Examination of the crystals in polarized light revealed that they were pleochroic and strongly birefringent.

Chemical analysis gave the following values for the percentage composition by weight:

C 79.3, H 5.74, N 14.65%. These agree reasonably with the theoretical values for benzaldehyde phenylhydrazone: C 79.6, H 6.13, N 14.26%. The melting point was $151^\circ C$.

A Weissenberg camera was used to record the $hk0$, $h0l$, $0kl$, $1kl$ and $2kl$ reflexions with Cu $K\alpha$ radiation. The unit cell was found to be monoclinic with

$$a = 5.95 \pm 0.05 \text{ \AA} \quad a:b:c = 0.32:1:0.86$$

$$\begin{aligned} b &= 17.76 \pm 0.05 & U &= 1609 \text{ \AA}^3 \\ c &= 15.15 \pm 0.05 & D_m &= 1.16 \text{ g.cm}^{-3} \\ \beta &= 92.5^\circ & Z &= 5.7 \approx 6 \end{aligned}$$

It will be seen that the axial ratios differ from those found by Fischer.

Systematic absences were

$$\begin{aligned} hkl & \text{ none} \\ h0l & \quad l = 2n \\ 0k0 & \quad k = 2n \end{aligned}$$

indicating the space group $P2_1/c$ with four asymmetric units per unit cell.

No further work on this compound is contemplated.

The author wishes to thank the Governors and Principal of the College for providing the facilities for this work, and Mr R. E. Reed for his advice on the chemistry of benzaldehyde phenylhydrazone.

References

- FISCHER, E. (1877). *Liebigs Ann.* **190**, 135.
MANN, F. G. & SAUNDERS, B. C. (1954). *Practical Organic Chemistry*. 3rd Edition. p. 177. London: Longmans.

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Crystal data for monorden (radicol). By F. W. COMER and J. TROTTER. *Department of Chemistry, University of British Columbia, Vancouver 8, B.C., Canada*

(Received 1 March 1965)

The antibiotic monorden (radicol) has been isolated from *Monosporium bonorden* (Delmotte & Delmotte-Plaquée, 1953). Recrystallization from chloroform gave colourless irregular crystals; both X-ray data and elemental analyses confirmed the presence of one-half mole of chloroform of crystallization. Crystal data were determined from various rotation, Weissenberg and precession films (λ , Cu $K\alpha=1.5418 \text{ \AA}$; λ , Mo $K\alpha=0.7107 \text{ \AA}$):

$C_{18}H_{17}O_6Cl \cdot \frac{1}{2}CHCl_3$; M.W. 424.5; m.p. $190-193^\circ C$
(Found: C, 52.72; H, 4.23; Cl, 19.5%. Required: C, 52.34; H, 4.16, Cl, 20.88%).

Monoclinic

$a = 9.12 \pm 0.02$, $b = 23.18 \pm 0.04$,
 $c = 8.95 \pm 0.02 \text{ \AA}$, $\beta = 99.7^\circ \pm 0.2^\circ$.
 $U = 1865 \text{ \AA}^3$, D_m (floatation in aqueous KI) $= 1.49 \pm 0.02$,
 $Z = 4$, $D_x = 1.51 \text{ g.cm}^{-3}$.

No systematic absences; space group probably $P2$.

In an effort to obtain a more suitable derivative, monorden was recrystallized from dibromomethane; this gave colourless prisms elongated along b with (100) and (001) developed. The crystals were, however, not solvated:

$C_{18}H_{17}O_6Cl$; M.W. 364.8; m.p. $190-194^\circ C$
Monoclinic

$a = 9.16$, $b = 15.01$, $c = 12.35$ (all $\pm 0.03 \text{ \AA}$).

$\beta = 100.2^\circ \pm 0.2^\circ$.
 $U = 1671 \text{ \AA}^3$, D_m (floatation in aqueous KI) $= 1.42 \pm 0.02$,
 $Z = 4$,
 $D_x = 1.45 \text{ g.cm}^{-3}$.
Space group probably $P2$ or $P2_1$ (the $0k0$ reflexions were not recorded).

At this stage the structure was deduced from other chemical and physical methods (Mirrington, Ritchie, Shoppee, Taylor & Sternhell, 1964; McCapra, Scott, Delmotte & Delmotte-Plaquée, 1964), and no further X-ray work is planned.

The authors are indebted to Dr F. McCapra and Dr A. I. Scott for suggesting the problem and for helpful discussion, and to the National Research Council of Canada for financial support and for the award of a research studentship (to F.C.).

References

- DELMOTTE, P. & DELMOTTE-PLAQUÉE, J. (1953). *Nature, Lond.* **171**, 344.
MCCAPRA, F., SCOTT, A. I., DELMOTTE, P. & DELMOTTE-PLAQUÉE, J. (1964). *Tetrahedron Letters*, 869.
MIRRINGTON, R. N., RITCHIE, E., SHOPPEE, C. W., TAYLOR, W. C. & STERNHELL, S. (1964). *Tetrahedron Letters*, 365.