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Crystallographic data for triclinic 5-formylvanillic acid.*

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The unit cell and space group of monoclinic 5-formylvanillic acid, $C_9H_8O_5$, were reported from this laboratory (Morita & Kodama, 1964). A triclinic isomer melting at 227–228 °C has been isolated by slow crystallization of the compound from a solution in a mixture of dioxane and petroleum ether (b.p. 60–80 °C).

Crystal data

The crystal data were determined from rotation, Weissenberg, and precession photographs with Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å).

$a_0 = 6{\cdot}91$, $b_0 = 7{\cdot}93$, $c_0 = 9{\cdot}36$ Å.

$\alpha = 125°22'$, $\beta = 124°58'$, $\gamma = 58°40'$.

$V = 331{\cdot}2$ Å^3.

$D_m = 2{\cdot}0$, $D_x = 1{\cdot}9647$ g.cm^{-3}, $Z = 2$

Of the two possible space groups $P\bar{1}$ and $P1$, $P\bar{1}$ is preferred because the compound is optically inactive.

The Debye–Scherrer powder reflexions were indexed with the use of the unit-cell dimensions obtained from the single-crystal study and their intensities estimated visually. The powder data have been submitted for inclusion in the ASTM X-ray Powder Data File.

No further detailed analysis is contemplated.

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The crystal structure of $YbZn_2$.*

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The determination of the crystal structure of $YbZn_2$ by single-crystal techniques is the subject of this communication.

Stoichiometric amounts of 99·5 % ytterbium and 99·999 % zinc were sealed under helium in a tantalum crucible, melted, and allowed to furnace-cool. Oscillation and Weissenberg photographs of a single crystal chosen from this alloy indicated the resulting compound to be orthorhombic, with $a = 4{\cdot}60$, $b = 7{\cdot}319$, $c = 7{\cdot}561$ Å. The values for b and c were obtained from an $0kl$ backreflection Weissenberg photograph. A Debye–Scherrer photograph, taken with Fe $K\alpha$ ($\lambda = 1{\cdot}93728$ Å) radiation, was subsequently indexed, and the final lattice parameters were found from a least-squares fit to the data to be:

$$a = 4{\cdot}573 \pm 0{\cdot}002, \quad b = 7{\cdot}325 \pm 0{\cdot}005, \quad c = 7{\cdot}569 \pm 0{\cdot}004 \text{ Å}.$$

All hkl reflections with $h+k+l = 2n+1$ and $hk0$ reflections with $h = 2n+1$ are absent on the Weissenberg photographs, indicating that the space group is *Im*2*a* (or *I*2*ma*) or *Imma*.

The intensities of 277 hkl reflections were measured on an equi-inclination Weissenberg counter diffractometer using an ω scan. Mo $K\alpha$ radiation was used, and data were recorded out to $\sin\theta = 0{\cdot}5$. Although the crystal was quite irregular in shape, it was approximated by a cylinder and absorption corrections were calculated according to Bond (1959).

The lattice parameters and possible space group for $YbZn_2$ indicated that it is isostructural with $CeCu_2$ (Larson & Cromer, 1961), and also YZn_2 (Sree Harsha & Ryba, 1964). Thus, there are four ytterbium atoms and eight zinc atoms in the equipoints 4(*e*) and 8(*h*), respectively, of the space group *Imma*. Using the positional parameters for YZn_2, a trial structure was refined with the full-matrix least-squares program of Busing, Martin & Levy (1962). The atomic scattering factors for ytterbium and zinc were taken from *International Tables for X-ray Crystallography* (1962), and were corrected for anomalous dispersion according to Dauben & Templeton (1955). Unit weights were assigned to all reflections. The results of this refinement are given in Table 1. The final residual for all observed reflections is 15 %. Observed and calculated $0kl$ structure factors are given in Table 2. An $0kl$ electron density projection confirmed the correctness of the structure. The high residual and the lack of better agreement between observed and calculated structure factors can probably be attributed to the irregular shape of the crystal and the high absorption of the ytterbium atom. Interatomic distances are given in Table 3; the standard deviations given were calculated by taking into account the standard deviations in both the positional parameters and the lattice parameters.

Metallographic examination of the alloy revealed the presence of extensive twinning. A thermal arrest, indicating a possible phase transition in $YbZn_2$, was observed at 683 ± 3 °C. This transition may be the cause of the twinning.

We would like to acknowledge the assistance of Mr Prabhat K. Kejriwal in the preparation of the alloy sample.

Table 1. *Final parameters for* $YbZn_2$ *from the least-squares refinement*

Atom	Position	x	y	z	B(Å^2)
Yb	4e	0	$\frac{1}{4}$	0·5503 ± 0·0005	0·91 ± 0·04
Zn	8h	0	0·0587 ± 0·0009	0·1641 ± 0·0009	1·23 ± 0·08

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Table 2. Observed and calculated structure factors for $YbZn_2$

hkl	F_o	F_c	hkl	F_o	F_c
040	113	104	015	100	107
060	114	-107	035	31	-32
080	27	19	055	96	106
0·10·0	62	-64	075	37	-36
0·12·0	27	25	095	38	36
011	10	6	0·11·5	42	-42
031	109	-99	006	33	31
051	32	-26	026	61	63
071	46	-41	046	17	-16
091	21	20	066	16	-4
0·11·1	11	4	086	38	-40
002	56	51	0·10·6	7	-6
022	111	-122	017	35	40
042	82	71	037	70	-80
062	51	-43	057	13	15
082	69	61	077	42	-47
0·10·2	25	-22	097	32	30
0·12·2	26	26	008	63	-63
013	88	87	028	20	35
033	79	-80	048	36	-43
053	67	60	068	39	42
073	59	-50	088	22	-20
093	41	37	019	12	13
0·11·3	30	-26	039	14	-18
004	17	-13	059	9	9
024	60	-57	079	11	-12
044	23	21	00·10	66	-62
064	7	-4	02·10	24	30
084	35	33	04·10	34	-40
0·10·4	4	-1			

Table 3. *Interatomic distances in* $YbZn_2$

Yb–2Yb	3·790 ± 0·006 Å	Zn–2Yb	3·329 ± 0·005 Å
Yb–2Yb	3·741 ± 0·003	Zn–1Yb	3·241 ± 0·008
Yb–4Zn	3·329 ± 0·005	Zn–2Yb	3·134 ± 0·005
Yb–2Zn	3·241 ± 0·008	Zn–1Yb	3·128 ± 0·007
Yb–4Zn	3·134 ± 0·005	Zn–1Zn	2·802 ± 0·013
Yb–2Zn	3·128 ± 0·007	Zn–2Zn	2·630 ± 0·007
		Zn–1Zn	2·629 ± 0·014

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Crystal data on *o*-phenanthroline hydrate.

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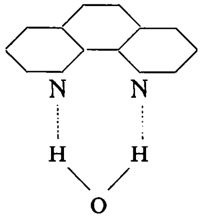
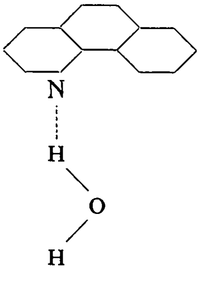
The bidentate ligand *o*-phenanthroline crystallizes from hot water as a monohydrate. The water molecules may be non-bonded, or hydrogen-bonded to the nitrogen atoms of the phenanthroline molecule. It is unlikely that the water is entirely in the non-bonded form, since the nitrogen atoms of the ligand are good proton acceptors; one molecule of water may be readily accommodated as shown in configuration *A*.

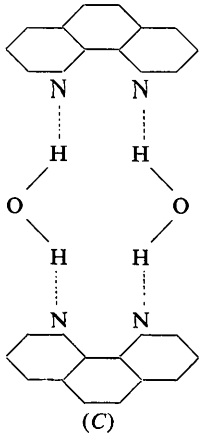
Configuration *B* is less likely because only one hydrogen donor and one hydrogen acceptor are used and because the dihydrate, which should exist if the configuration were *B*, has not been reported in the literature. Configuration *C* may be regarded as a special case of $(C_{12}H_8N_2 . H_2O)_n$ with $n=2$; n-membered rings of any size, as well as infinite chains, are consistent with this formulation. The predicted dimensions of a planar phenanthroline molecule are 8·98 Å in length and 5·51 Å in width, using accepted bond lengths (Sutton, 1958).



(*A*)



(*B*)



(*C*)

Infrared spectroscopy should indicate a single hydrogen-bonded OH band for structures *A* and *C*, or two OH bands (hydrogen-bonded OH and free OH) for structure *B*. However, solution spectra in dry carbon tetrachloride indicate displacement of the water molecule by the solvent, and solid state spectra (KBr) are unsatisfactory.

A sample obtained from the G. Frederick Smith Chemical Company in Columbus, Ohio, was recrystallized twice from hot water. Its melting point was determined as 93–94 °C. Elementary analyses are consistent with the formula $C_{12}H_8N_2 . H_2O$ with F.W. 198·226. The crystals, a few millimeters long, are colorless and fragile. They belong to the hexagonal system. In habit they are acicular **c** and singly terminated. Recrystallized on a glass slip from a drop of solution, they show plumose habit and subspherulitic aggregates, with bright interference colors between crossed nicols.

Optically the crystals are positive. The indices of refraction are roughly estimated as $nO \sim 1{\cdot}55$, $nE \sim 1{\cdot}77$, with very large birefringence $B \sim 0{\cdot}22$. As a consequence of the positive optical character, the molecules can be predicted to lie parallel to **c**, an arrangement that will also agree with the prismatic cleavage.