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Space group and cell constants of 3, 4, 5-tricarbethoxy pyrazole. By JONATHAN PARSONS, The Edsel B. Ford Institute for Medical Research, Henry Ford Hospital, Detroit 2, Michigan, U.S.A.

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symmetrical.

A space-group determination has been made for a crystalline derivative of pyrazole tricarboxylic acid, using the Weissenberg X-ray method. This compound, 3,4,5-tricarbethoxy pyrazole, was isolated in the Biochemistry Department of the Institute, using the method of Curtius (1915). The structural formula can be given as



Rotation and Weissenberg photographs were prepared for the *b* and *c* axes of a single crystal of this compound. The only systematic absences found were for 0k0 when *k* is odd. This indicates that the space group is either $P2_1-C_2^2$ or $P2_1/m-C_{2h}^2$. Measurement of the unit-cell dimensions from the central lattice line spacings on the zero-layer photographs led to the following lattice constants:

a = 9.60, b = 7.33, c = 10.38 Å, and $\beta = 97^{\circ} 35' \pm 15'$.

The inclination angle β was measured using the method of angular lag (Buerger, 1942).

The approximate density of the crystals was found to be 1.3 g.cm.^{-3} by flotation in mixtures of benzene and carbon tetrachloride. The density computed from X-ray data on the assumption of two molecules per unit cell is 1.304 g.cm.^{-3} . The presence of two rather than four molecules per unit cell is rather strong evidence for the space group $P2_1$ rather than $P2_1/m$ as, for the latter

Table	1.	Powder	pattern	d	values	and	relative	intensities		
for 3.4.5-tricarbethoxy pyrazole										

space group, the molecule would have to be centro-

d (Å)	Ι	d (Å)	I	d (Å)	I
10.28	2	3.58	7	$2 \cdot 25$	1
8.12	1	3.44	5	$2 \cdot 20$	1
7.50	10	3.31	5	$2 \cdot 17$	2
6.53	6	3.20	1	2.13	1
5.96	3	3.10	5	2.09	4
5.71	2	2.98	3	2.05	3
5.20	7	2.89	4	1.99	2
4.87	3	2.84	4	1.92	3
4.73	5	2.73	2	1.88	1
4.51	1	2.65	3	1.85	2
4.21	5	2.60	1	1.80	3
4.08	1	2.47	3	1.70	1
3.97	7	2.41	ī	1.65	2
3.84	9	2.36	5	1.58	1
3.66	6	2.29	ī	1.49	2

Table 1 gives the powder pattern d values and visual estimates of their relative intensities. This data was obtained using Ni-filtered Cu $K\alpha$ radiation. The X-ray tube was operated at 35 kV. and 20 mA.

References

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The unit cell and space group of tetrapyridinecopper (II) fluoborate.* By JAMES A. IBERS[†], Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena, U.S.A.

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Tetrapyridinecopper (II) fluoborate, $Cu(C_5H_5N)_4(BF_4)_2$, was kindly supplied to us by Prof. James C. Warf (1952) of the University of Southern California. The deep-blue crystals exhibit primarily a columnar habit. Upon exposure to air for a few days the crystals turn light green in color, presumably owing to the loss of pyridine.

With Laue photographs prepared with a continuous spectrum of minimum wavelength 0.3 Å, and with rotation photographs prepared with copper K radiation filtered through nickel foil, we have found the crystal to be orthorhombic with

$$a = 10.22 \pm 0.01, \ b = 13.87 \pm 0.01, \ c = 16.56 \pm 0.01 \text{ Å}$$
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\ast\, Contribution No. 1765 from the Gates and Crellin Laboratories.
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On Weissenberg photographs made with filtered copper radiation even orders from the respective pinacoids were observed up to h = 12, k = 16, and l = 18, but no odd orders were observed. Reflections of all other types were present. Hence the space group is probably $D_2^4 - P2_12_12_1$. No piezoelectric effect, however, could be observed. The density measured pycnometrically was 1.55 g.cm.⁻³, whereas a density of 1.565 g.cm.⁻³ corresponds to four molecules in the unit cell. The observed density is probably low because the substance is very slightly soluble in water, the pycnometric liquid.

Because of the size and the complexity of the unit cell, no further work is contemplated on this structure.

Reference

WARF, J. C. (1952). J. Amer. Chem. Soc. 74, 3702.

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