Acta Cryst. (1961). **14**, 1080

Structural constants of the mono-halogeno-benzenes.

By D. E. HENSHAW, *Department of Physics, The University of Western Australia, Nedlands, Western Australia**

(*Received* 29 *December* 1960 *and in revised form* 26 *May* 1961)

Single crystals of each of the four mono-halogeno-benzenes were grown by slow crystallization from the liquid phase and were examined at a suitable temperature (T) in a single-crystal X-ray camera (Henshaw, 1957). The lattice parameters and space groups of all four were determined (Table 1) and the structural arrangement of fluorobenzene deduced. The densities (d_T) were calculated and show a reasonable relationship to the densities of the liquids at 20 °C. (d_T/d_{20}); d_T/d_{20} for benzene is 1·21.

The structure of fluorobenzene was solved by the use of the $hk0$ reflections. Since only nineteen independent reflections could be measured and since considerable overlap of atoms was present in the electron-density projection, the X, Y co-ordinates of Table 2 cannot be considered very accurate even though the structure was refined to $R = 0{\cdot}07$. The Z co-ordinates were determined from the X, Y co-ordinates and an assumed C–C bond length, and gave agreement with measured intensities for five hkl reflections.

Table 2. *The fractional co-ordinates of the* C *and* F *atoms of fluoro-benzene in the* $P4_1$ *space group*

	X	Y	Z
F	0·326	0·326	0
C_1	0·485	0·485	0
C_2	0·455	0·674	0·058
C_3	0·627	0·839	0·058
C_4	0·820	0·820	0

* Present address: C.S.I.R.O., Wool Textile Research Laboratories, Geelong, Victoria, Australia.

Reference

HENSHAW, D. E. (1957). *J. Sci. Instrum.* **34**, 270.

Table 1. *Lattice parameters of the mono-halogeno-benzenes*

	C_6H_5F	C_6H_5Cl	C_6H_5Br	C_6H_5I
Melting point	−41·9 °C.	−45 °C.	−30·6 °C.	−31·35 °C.
T	−65 °C.	−65 °C.	−52 °C.	−50 °C.
Cell dimensions (Å) a	5·83 ± 0·05	13·66 ± 0·06	14·19 ± 0·06	15·23 ± 0·06
b		11·14 ± 0·06	11·26 ± 0·06	10·81 ± 0·06
c	14·61 ± 0·07	7·20 ± 0·10	7·35 ± 0·10	7·65 ± 0·06
Unit-cell volume (Å^3)	497	1108	1174	1260
Space group	$P4_12_12$ or $P4_32_12$	*Pbcn*	*Pbcn*	*Pbcn*
No. of molecules/cell	4	8	8	8
Calculated density (d_T)	1·29	1·35	1·78	2·15
d_T/d_{20}	1·26	1·22	1·19	1·18

Acta Cryst. (1961). **14**, 1080

The orientation of single crystals.

By P. P. WILLIAMS,* *Dominion Laboratory, Wellington, New Zealand* and D. HALL, *Chemistry Department, University of Auckland, New Zealand*

(*Received* 7 *June* 1961)

The methods developed by Hendershot (1937) and Weisz & Cole (1948) by which a single crystal may be accurately adjusted to rotate about an axis, consist essentially in analysis of the form of the trace of reflections in the axis zone on stationary or small-angle oscillation photographs. By measuring the displacement of this layer line, Weisz & Cole deduce the angular corrections necessary in the planes parallel to the rotation axis and respectively perpendicular and parallel to the X-ray beam. Accordingly, the setting photograph is taken with the goniometer arc planes thus oriented with respect to the beam.

We have found it more convenient to deduce adjustments in the planes parallel to the rotation axis, but making angles of 45° to the beam, and to set the goniometer accordingly. The displacement of the zero layer line at $\theta = 45°$ on each side of the film is then a consequence of the mis-setting in one arc only. For small angular deviations, the displacement per degree of mis-setting in the arc is 0·0124 times the diameter of the camera, i.e. for the common camera diameter of 57·3 mm., the displacement per degree is 0·71 mm.

This procedure has advantages over that described by Weisz & Cole in that the measured displacement as a function of mis-setting is greater, and the direction of movement for correction of the arcs is more obvious, particularly when the layer line is not very well defined at high θ values. Their use of unfiltered radiation and of a composite photograph (two exposures at 180° interval) is again of great value.

One of us (P. P. W.) wishes to thank the Director of Dominion Laboratory, Wellington, New Zealand, for permission to publish this note.

* At present at Cavendish Laboratory, Cambridge, England.

References

HENDERSHOT, O. P. (1937). *Rev. Sci. Instrum.* 8, 436.
WEISZ, O. & COLE, W. F. (1948). *J. Sci. Instrum.* **25**, 213.