

NaBH₄ at 90° K.: body-centred tetragonal, $a = 4.353$,
 $c = 5.909$ (Abrahams & Kalnajs (1954), $a = 4.354$
 ± 0.005 , $c = 5.907 \pm 0.005$).

KBH₄ at 293° K.: face-centred cubic, $a = 6.722$
(Abrahams & Kalnajs (1954), $a = 6.7272 \pm 0.0005$).

KBH₄ at 90° K.: face-centred cubic, $a = 6.636 \pm 0.002$.

The results for NaBH₄ and KBH₄ at room temperature, and for NaBH₄ at low temperature, are in good agreement with previous measurements (Soldate, 1947; Abrahams & Kalnajs, 1954). Unlike its sodium analogue, KBH₄ at 90° K. shows no change in crystal structure beyond a lattice contraction. Stockmayer & Stephenson (1953) suggested that NaBH₄ may change from the cubic form at temperatures below the specific-heat anomaly (Johnston & Hallet, 1953) in order to reduce the repulsive energy between the hydrogen atoms. KBH₄, however, has a more open structure, owing to the larger size of the potassium ion, and remains cubic down to 90° K.

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References

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The unit-cell dimensions of *p*-chlorobenzoic acid. By J. McC. POLLOCK and (Miss) I. WOODWARD, Department of Chemistry, Queen's University, Belfast, Northern Ireland

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In the course of some X-ray investigations on *p*-chlorobenzoic acid, values of the unit-cell dimensions were found differing appreciably from those given by Toussaint (1951).

Seven reflexions chosen for their high Bragg angles from the three principal zones were recorded on a multiple-exposure camera of 14 cm. diameter (Ubbelohde, 1939). Film measurements were made to 0.002 cm. with a travelling microscope, and both α_1 and α_2 reflexions were measured on each film by two independent observers. Calibration was by a platinum substandard against silver ($a = 4.0775$ Å) and the radiation employed was Cu $K\alpha$ ($\lambda_{\alpha_1} = 1.5405$ Å, $\lambda_{\alpha_2} = 1.5443$ Å). The planes used, together with their Bragg angles, are given in Table 1.

Table 1. Planes used

hkl	θ_{α_1}	θ_{α_2}
13,5,0	75° 20.0'	75° 54.8'
17,3,0	78° 57.3'	79° 35.3'
870	83° 24.1'	84° 45.3'
970	77° 15.36'	77° 56.73'
12,0,3	68° 32'	68° 56.9'
15,0,3	75° 51.1'	76° 30.1'
063	71° 1.6'	71° 29.3'

The method of least squares was used to find a^* , b^* and γ^* from the ($hk0$) zone, and the remaining reciprocal-lattice parameters were then determined by solving the general equation for the triclinic system:

$$(2 \sin \theta)^2 = h^2 a^{*2} + k^2 b^{*2} + l^2 c^{*2} + 2klb^*c^* \cos \alpha^* \\ + 2lhc^*a^* \cos \beta^* + 2hka^*b^* \cos \gamma^*.$$

These parameters are given in Table 2, together with the unit-cell dimensions derived from them, the figures being

Table 2. Lattice parameters of *p*-chlorobenzoic acid at 18° C.

Reciprocal parameters for λ_{α_1}	Present work	Deviation from mean	Toussaint
a^* 0.10916 Å ⁻¹	a 14.190 Å	± 0.004 Å	14.39 Å
b^* 0.24835 Å ⁻¹	b 6.213 Å	± 0.001 Å	6.29 Å
c^* 0.40158 Å ⁻¹	c 3.852 Å	± 0.002 Å	3.86 Å
α^* 88° 28'	α 91° 15'	$\pm 2'$	91° 38'
β^* 84° 36'	β 95° 19'	$\pm 1'$	95° 18'
γ^* 86° 56'	γ 92° 56'	$\pm 1'$	92° 44'

the mean of the α_1 and α_2 calculations. The third column shows the deviation from their mean of the values calculated from the α_1 and α_2 observations. The estimated systematic errors are less than these. Toussaint's values are given for comparison.

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