$NaBH_4$ at 90° K.: body-centred tetragonal, $a = 4{\cdot}353$, $c = 5{\cdot}909$ (Abrahams & Kalnajs (1954), $a = 4{\cdot}354 \pm 0{\cdot}005$, $c = 5{\cdot}907 \pm 0{\cdot}005$).
$KBH_4$ at 293° K.: face-centred cubic, $a = 6{\cdot}722$ (Abrahams & Kalnajs (1954), $a = 6{\cdot}7272 \pm 0{\cdot}0005$).
$KBH_4$ at 90° K.: face-centred cubic, $a = 6{\cdot}636 \pm 0{\cdot}002$.

The results for $NaBH_4$ and $KBH_4$ at room temperature, and for $NaBH_4$ at low temperature, are in good agreement with previous measurements (Soldate, 1947; Abrahams & Kalnajs, 1954). Unlike its sodium analogue, $KBH_4$ at 90° K. shows no change in crystal structure beyond a lattice contraction. Stockmayer & Stephenson (1953) suggested that $NaBH_4$ 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. $KBH_4$, 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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## 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 $\alpha_1$ and $\alpha_2$ reflexions were measured on each film by two independent observers. Calibration was by a platinum substandard against silver ($a = 4{\cdot}0775$ Å) and the radiation employed was Cu $K\alpha$ ($\lambda\alpha_1 = 1{\cdot}5405$ Å, $\lambda\alpha_2 = 1{\cdot}5443$ Å). The planes used, together with their Bragg angles, are given in Table 1.

Table 1. *Planes used*

| *hkl* | $\theta\alpha_1$ | $\theta\alpha_2$ |
|---|---|---|
| 13,5,0 | 75° 20·0′ | 75° 54·8′ |
| 17,$\bar{3}$,0 | 78° 57·3′ | 79° 35·3′ |
| 870 | 83° 24·1′ | 84° 45·3′ |
| 9$\bar{7}$0 | 77° 15·36′ | 77° 56·73′ |
| 12,0,3 | 68° 32′ | 68° 56·9′ |
| 15,0,$\bar{3}$ | 75° 51·1′ | 76° 30·1′ |
| 06$\bar{3}$ | 71° 1·6′ | 71° 29·3′ |

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

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

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 $\lambda\alpha_1$ | | 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 |
| $\alpha^*$ | 88° 28′ | $\alpha$ | 91° 15′ | ±2′ | 91° 38′ |
| $\beta^*$ | 84° 36′ | $\beta$ | 95° 19′ | ±1′ | 95° 18′ |
| $\gamma^*$ | 86° 56′ | $\gamma$ | 92° 56′ | ±1′ | 92° 44′ |

the mean of the $\alpha_1$ and $\alpha_2$ calculations. The third column shows the deviation from their mean of the values calculated from the $\alpha_1$ and $\alpha_2$ observations. The estimated systematic errors are less than these. Toussaint's values are given for comparison.

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