

X-ray powder photographs of the other preparations showed no change in the lattice constants throughout.

The magnetic moments listed in Table 1 were obtained with a vibrating sample magnetometer by P. K. Baltzer and S. Kasdan. These results are difficult to interpret since it is not obvious what the type and strength of the interaction of magnetic ions in this structure might be. Moments of this magnitude could conceivably be accounted for by the presence of small amounts of ferrimagnetic impurities (*e.g.*  $\text{Fe}_3\text{O}_4$ ), although no such phases have been observed in the X-ray patterns.

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### References

- DAUBEN, C. H. and TEMPLETON, D. H. (1955). *Acta Cryst.* **8**, 841.  
 GOLDSCHMIDT, V. M. & HAUPTMANN, H. (1932). *Nachr. Ges. Wiss. Göttingen*, p. 53.  
 KEITH, M. L. & ROY, R. (1954). *Amer. Min.* **39**, 1.  
*Internationale Tabellen*. (1935). Vol. II. Berlin: Borntraeger.  
*Strukturbericht* (1931). **1**, 292.

*Acta Cryst.* (1963). **16**, 850

### Crystallographic data for the $\alpha$ and $\beta$ isomers of 4-hydroxy-2,2,6-trimethylpiperidine hydrochloride and the $\beta$ -O-benzyl hydrochloride. By J. G. SCANE, *Physics Department, College of Technology, Portsmouth, England*

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Data for the  $\alpha$  and  $\beta$  isomers of the free base have already been obtained (Scane, 1961), and it was intended to use the hydrochlorides to establish the configuration and conformation of each isomer. Owing to the small number of reflexions observed for the hydrochlorides they have been dropped in favour of the non-isomorphous hydrobromides.

Doubt exists as to the position of the methyl groups, for although infrared studies have shown the existence of an N-H stretch in piperidine and the ketone from which both isomers are prepared, both the  $\alpha$  and  $\beta$  bases show no sign of the N-H stretch.

#### $\alpha$ -B.HCl (m. p. over 300 °C)

The hydrochloride of the  $\alpha$  isomer was prepared from the ( $\pm$ )base by precipitation from solution by HCl gas and recrystallization from ethanol. Very good colourless crystals were obtained elongated along [100] with (011) and (001) prominent. A full survey of the reciprocal lattice was carried out twice on two separate crystals with a stationary-film single-crystal camera and Cu  $K\alpha$  radiation. The density was measured by flotation in a benzene-bromoform mixture, and analysis confirmed the formula  $\text{C}_8\text{H}_{18}\text{ONCl}$ .

#### $\beta$ -B.HCl (m. p. 191-193 °C)

Crystals of the hydrochloride of the  $\beta$  isomer were prepared and studied in the same way as for the  $\alpha$ -B.HCl. They are colourless tablets with (010) prominent. Analysis confirmed the formula  $\text{C}_8\text{H}_{18}\text{ONCl}\cdot\text{H}_2\text{O}$ , and vacuum desiccation at 80 °C removed the water of crystallization and caused the crystals to disintegrate.

#### $\beta$ -O-benzyl HCl (m. p. 269-271 °C)

The hydrochloride of the  $\beta$  isomer of the O-benzyl

derivative crystallized in well formed plates with (001) prominent. The X-ray investigation was carried out in the same way.

Table 1. *Crystallographic data*

	$\alpha$ -B.HCl	$\beta$ -B.HCl	$\beta$ -O-Benzyl HCl
<i>a</i>	8.7 <sub>0</sub> Å	7.7 <sub>3</sub> Å	10.2 Å
<i>b</i>	9.4 <sub>5</sub> Å	19.6 Å	12.3 Å
<i>c</i>	12.5 Å	8.6° Å	48.4 Å
$\beta$		122°	
<i>D</i> <sub>o</sub>	1.16 g.cm <sup>-3</sup>	1.17 g.cm <sup>-3</sup>	1.23 g.cm <sup>-3</sup>
<i>D</i> <sub>c</sub>	1.16 g.cm <sup>-3</sup>	1.18 g.cm <sup>-3</sup>	1.24 g.cm <sup>-3</sup>
<i>Z</i>	4	4	16
Space group	$P2_12_12_1$	$P2_1/c$	$Fd2d$

Crystallographic data for the three hydrochlorides are given in Table 1. The space groups were determined uniquely from the systematic absences in all cases.

Since  $P2_12_12_1$  requires four identical asymmetric units to the unit cell, the unit cell of the  $\alpha$ -B.HCl cannot contain both + and - molecules. Hence the  $\alpha$ -racemate must separate into the + and - forms on crystallization. This is consistent with the observation that the  $\alpha$  hydrochlorides all melt above 300 °C whether prepared from D, L or DL base (King, 1924). The other two hydrochlorides obviously crystallize as racemates.

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### References

- KING, H. (1924). *J. Chem. Soc.* p. 41.  
 SCANE, J. G. (1961). *Acta Cryst.* **14**, 1005.