Acta Cryst. (1980). B**36**, 1688–1689

2-Methylsparteine Perchlorate

By Andrzej Katrusiak, Andrzej Hoser, Zygmunt Kałuski and Władysław Boczoń

Institute of Chemistry, A. Mickiewicz University, Grunwaldzka 6, 60-780 *Poznań, Poland*

(*Received* 21 *January* 1980; *accepted* 7 *March* 1980)

Abstract. $C_{16}H_{29}N_2^+.ClO_4^-$, monoclinic, $P2_1$, $Z = 2$, $a = 9\cdot618\,(1)$, $b = 10\cdot690\,(1)$, $c = 9\cdot342\,(1)$ Å, $\beta = 113\cdot21\,(1)^\circ$, $D_c = 1\cdot31$ Mg m^{-3}, $\mu = 2\cdot11$ mm^{-1}. The molecule has the all-chair conformation; the sparteine skeleton has the *trans–cis* configuration. An intramolecular hydrogen bond was observed: N(1)···N(16) 2·710 (7) Å. The methyl substituent is in the equatorial position. The final R factor for 1180 reflections was 0·055; $R_w = 0\cdot069$.

Introduction. Colourless crystals of 2-methylsparteine perchlorate were obtained from ethanol solution. That used for data collection had dimensions 0·3 × 0·3 × 0·3 mm. Measurements were made on a Syntex $P2_1$ diffractometer, using graphite-monochromated Cu $K\alpha$ radiation. 1275 reflections were collected, of which 1180 had intensities greater than $1\cdot96\sigma_I$. Only Lorentz and polarization corrections were applied.

The Cl atom was found from the Patterson map. All remaining nonhydrogen atoms were located from difference Fourier maps. After three cycles of anisotropic full-matrix least-squares refinement H atoms, except those of the methyl group and C(9), were located from a difference Fourier map. The position of H(C9) was calculated from the geometry. The final R factor for 1180 reflections was 0·055; $R_w = 0\cdot069$. In the last few cycles of the refinement the following weighting scheme was used: $w = (F_o/2\cdot23)^2$ for $F_o < 2\cdot23$, $w = 1$ for $2\cdot23 \le F_o \le 9\cdot98$, $w = (9\cdot98/F_o)^2$ for $F_o > 9\cdot98$. The final positional parameters are listed in Table 1.*

All the programs were from the *XTL/E-XTL* structure determination system (Syntex, 1976). All calculations were performed on a Nova 1200 computer.

Discussion. The bond distances and valency angles are presented in Fig. 1. All are in good agreement with results obtained for other sparteine derivatives (Kałuski, Skolik & Wiewiórowski, 1979). The methyl substituent is in the equatorial position. This can be seen in Fig. 2, which is a projection of the unit cell along **b**. The H atom of N(16) was located from a difference Fourier map. This protonation site was also confirmed by the N–C distances; those for N(16) are significantly longer than those for N(1).

The spectroscopic results reported by Boczoń (1980) suggest N(1) protonation for the title compound in the solid state. Protonation at N(1) occurs in sparteine perchlorate crystals [X-ray study of Borowiak, Bokii & Struchkov (1980)], so it is evident that the methyl substituent at C(2) changes the site of protonation in the solid state. As in sparteine perchlorate, the title compound has a strong intramolecular hydrogen bond:

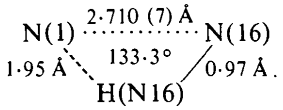

Table 1. *Fractional atomic coordinates* (×10⁴) *and isotropic thermal parameters* (Å²)

	x	y	z	B_i
Cl	6275 (2)	6562	3597 (2)	4·1 (1)
O(1)	7742 (6)	6617 (11)	4814 (11)	12·2 (6)
O(2)	5496 (9)	5745 (7)	4178 (8)	6·8 (4)
O(3)	5611 (12)	7766 (7)	3479 (12)	9·5 (6)
O(4)	6285 (16)	6102 (7)	2225 (10)	11·1 (6)
N(1)	1762 (5)	7241 (5)	4274 (5)	2·9 (3)
C(2)	1055 (8)	7547 (8)	2588 (8)	4·4 (4)
C(3)	1357 (6)	6518 (12)	1600 (7)	5·0 (4)
C(4)	965 (9)	5267 (9)	1946 (9)	5·5 (5)
C(5)	1776 (7)	4977 (8)	3659 (8)	4·5 (4)
C(6)	1383 (6)	5961 (7)	4624 (7)	2·8 (3)
C(7)	2173 (7)	5705 (7)	6380 (7)	3·4 (3)
C(8)	1592 (7)	6631 (10)	7264 (7)	4·8 (4)
C(9)	2032 (8)	7926 (7)	6883 (8)	4·0 (4)
C(10)	1277 (7)	8146 (8)	5162 (9)	4·4 (4)
C(11)	3747 (7)	8090 (6)	7506 (7)	3·5 (3)
C(12)	4541 (9)	8155 (8)	7260 (8)	4·1 (4)
C(13)	6249 (8)	8323 (8)	9786 (8)	5·3 (5)
C(14)	6877 (7)	7279 (8)	9126 (8)	3·7 (4)
C(15)	6096 (6)	7181 (7)	7363 (7)	4·1 (4)
N(16)	4383 (5)	7061 (5)	6326 (5)	2·6 (3)
C(17)	3888 (7)	5768 (7)	7018 (8)	3·8 (4)
C(18)	1602 (11)	8841 (10)	2266 (10)	5·7 (5)

---

* Lists of structure factors, anisotropic thermal parameters, and hydrogen parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35194 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/80/071688-02$01.00

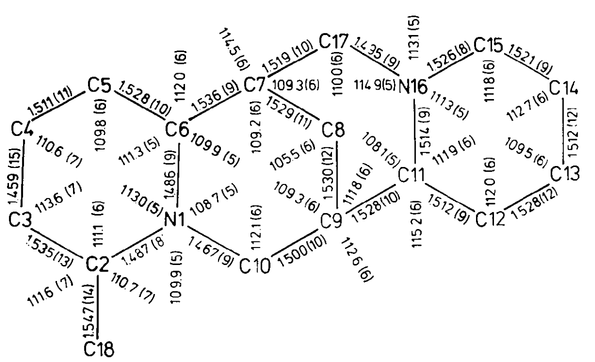

Fig. 1. Bond distances (Å) and valency angles (°) with their e.s.d.'s.

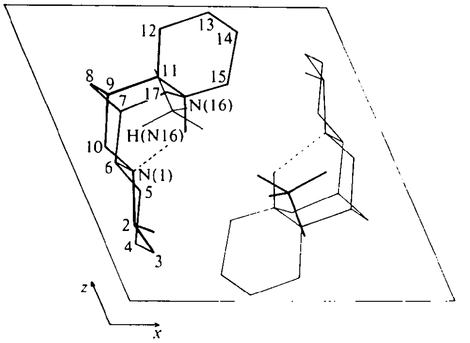

Fig. 2. Projection of the unit cell along the *b* axis.

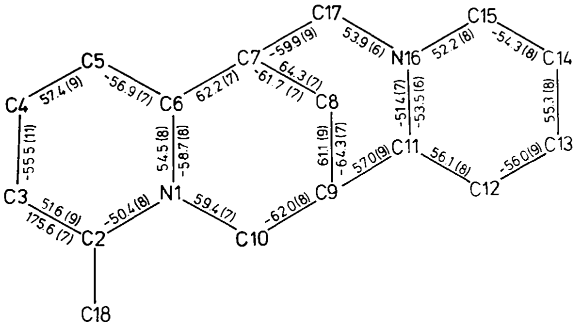

Fig. 3. Torsion angles (°) with their e.s.d.'s.

The intramolecular interaction in sparteine perchlorate is insignificantly weaker [N(1)···N(16) 2·752 Å], and the angles describing the inclination of two quinolizidine skeletons *A*/*B* and *C*/*D* are almost the same.

The molecule has the all-chair conformation. The torsion angles with their e.s.d.'s are shown in Fig. 3. The asymmetry parameters (Duax & Norton, 1975) for the piperidine rings are: $\Delta C_s^{2} = 0\cdot95$, $\Delta C_2^{3,4} = 6\cdot16$, $\Delta C_2^{1,2} = 2\cdot3^\circ$ for ring *A*; $\Delta C_s^{1} = 0\cdot64$, $\Delta C_2^{6,7} = 2\cdot44$, $\Delta C_s^{7} = 2\cdot1^\circ$ for ring *B*; $\Delta C_s^{8} = 2\cdot21$, $\Delta C_2^{7,17} = 11\cdot7$, $\Delta C_2^{8,9} = 5\cdot3^\circ$ for ring *C*; and $\Delta C_2^{12,13} = 0\cdot8$, $\Delta C_s^{11} = 2\cdot9$, $\Delta C_s^{12} = 1\cdot6^\circ$ for ring *D*. There are no significant changes of conformation between 2-methylsparteine perchlorate and sparteine perchlorate.

There are only van der Waals interactions between the molecules in the crystal (Fig. 2).

We thank Professor M. Wiewiórowski for suggesting the determination of this structure.

This study was supported by the Polish Academy of Sciences as part of the problem MR I-9.

References

Boczoń, W. (1980). *Rocz. Chem.* In the press.

Borowiak, T., Bokii, N. G. & Struchkov, Yu. T. (1980). In preparation.

Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structure*. New York: Plenum.

Kałuski, Z., Skolik, J. & Wiewiórowski, M. (1979). Proc. Pre-Congress Symposium on Organic Crystal Chemistry, Poznań–Dymaczewo, Poland, 30 July–2 August 1978.

Syntex (1976). *XTL/E-XTL Structure Determination System Operation Manual*. Syntex Analytical Instruments Inc., 10040 Bubb Road, Cupertino, California 95014, USA.