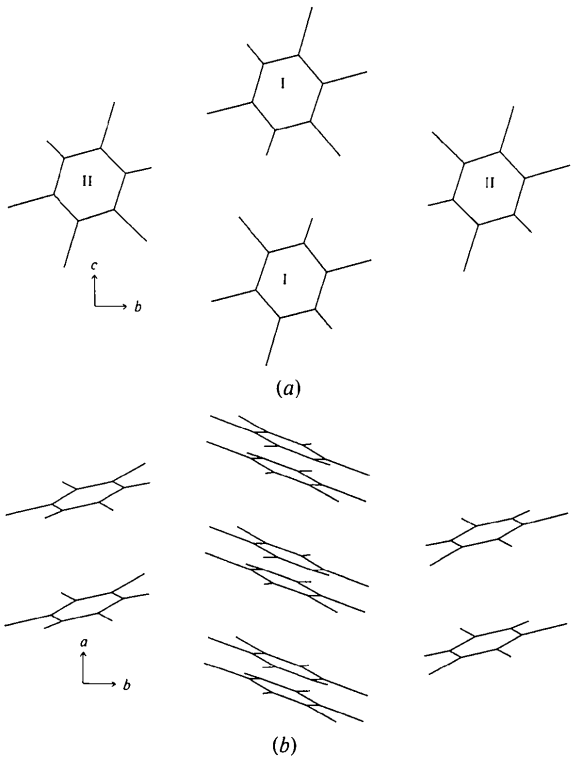

Fig. 2. The packing of the molecules around an inversion center. (*a*) A view along the short *a* lattice direction, looking down superimposed molecules arranged in stacks of type I or type II molecules. (*b*) A view along the *c* lattice direction showing the herringbone packing along *b*.

Intramolecular Cl···Cl repulsions produce minor distortions in the benzene ring. The bond angles C(1)–C(2)–C(3), C(3)–C(4)–C(5) and C(5)–C(6)–C(1) are less than 120° (118·1, 118·6, and 118·0°, e.s.d.'s 0·3°), while C(2)–C(3)–C(4), C(4)–C(5)–C(6) and C(6)–C(1)–C(2) are greater than 120° (121·3, 122·1, and 121·8°, e.s.d.'s 0·3°). Both molecules display similar trends.

Fig. 2 illustrates the molecular packing. Type I and type II molecules stack in separate columns along the short-lattice-constant direction. The molecules, when viewed along *c*, show a herringbone type of packing. Along *b* the stacks are staggered. The intermolecular distances are in the normally expected range for nonbonded contacts; the shortest intermolecular distance is an H···Cl contact, at 3·025 Å. The shortest intermolecular Cl···Cl distance is 3·563 Å.

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15-Oxosparteine Perchlorate Hemihydrate

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Abstract. $C_{15}H_{25}N_2O^+.ClO_4^-.\frac{1}{2}H_2O$, $P22_12_1$, a = 9·071 (2), b = 11·096 (2), c = 16·793 (2) Å, Z = 4, D_x = 1·44 Mg m^{-3}. The final R = 0·052, R_w = 0·062 for 1217 reflections. Two types of hydrogen bonds were observed: O(C15)···O(W) and N(1)···O(C1) of 2·854 (5) and 2·912 (6) Å respectively. The piperidine

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rings *A*, *B*, *C* and *D* have chair, chair, boat and half-chair conformations respectively. The two quinolizidine skeletons have the *trans–trans* configuration.

Introduction. The present work is part of an X-ray study on the sparteine lactams (Kałuski, Skolik & Wiewiórowski, 1978; Skrzypczak-Jankun & Kałuski, 1978; Doucerain, Chiaroni & Riche, 1976; Małuszyńska, Hoser & Kałuski, 1979; Skrzypczak-Jankun, Hoser, Kałuski & Perkowska, 1980; Katrusiak, Hoser, Grzesiak & Kałuski, 1980). 15-Oxosparteine is one of four isomeric lactams, and does not occur naturally. It was obtained recently during the oxidation of sparteine by [Hg(edta)] (Wiewiórowski, Legocki & Bratek-Wiewiórowska, 1967). Chemical and spectroscopic studies have shown that the carbonyl group of this lactam is situated in the 15 position in the external ring of the bisquinolizidine skeleton (Wiewiórowski, Legocki & Bratek-Wiewiórowska, 1967; Wiewiórowski, Edwards & Bratek-Wiewiórowska, 1967).

In the case of the 15-oxosparteine salt, the amine N(1) atom in the rigid* *trans*-quinolizidine fragment (rings *A* and *B*) is protonated; therefore conformational changes may occur only in the *cis*-quinolizidine fragment of the molecule (rings *C* and *D*).

On the basis of NMR spectra (Wiewiórowski, Edwards & Bratek-Wiewiórowska, 1967) it was assumed that this latter fragment of the 15-oxosparteine, which occurs as a free base in $CDCl_3$ solution, exists in the boat/sofa conformation, and that the same conformation may be retained in its salt (Perkowska & Wiewiórowski, 1980).

Colourless crystals of the 15-oxosparteine perchlorate salt were obtained from ethanol solution. The dimensions of the crystal used for data collection were 0·6 × 0·6 × 0·4 mm. The measurements were carried out at room temperature using Cu $K\alpha$ radiation and a graphite monochromator. The θ–2θ scan method was applied with a scan speed depending on the intensity of the reflection (range 2–29·3° min^{-1}). Two control reflections were monitored after each 49 reflections; 1335 reflections were collected, including systematic absences, 1217 of which with intensities greater than $1{\cdot}96\sigma_I$ were included in the refinement. The background and integrated intensity for each reflection were obtained by the profile-analysis method of Lehmann & Larsen (1974), using the program *PRAN* (Jaskólski, 1979). The structure was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). The *E* map based on a correct phase set contained the positions of all nonhydrogen atoms. As a result of isotropic full-matrix least-squares refinement, an *R* of 0·112 was obtained. All H atoms except two [H(4) and H(13)] were located from a difference Fourier map. They were used in the structure-factor calculations, but were not included in the refinement. The function $\sum w(F_o - F_c)^2$ was minimized, where $w = \sigma^{-2}$. In the last few cycles of anisotropic refinement the following weighting scheme was applied: $w = (F_o/$

Table 1. *Fractional atomic coordinates* (×10⁴; ×10³ *for* H) *and isotropic thermal parameters* (Å²)

The isotropic thermal parameters for the H atoms are 6·0 Å². Those for the nonhydrogen atoms are derived from the anisotropic parameters by $B_{eq} = \frac{1}{3}(B_{11} + B_{22} + B_{33})$.

	x	*y*	*z*	B_{eq}
Cl	3835 (2)	2745 (1)	1549 (1)	3·86 (7)
O(1Cl)	4972 (7)	2089 (6)	1155 (1)	8·7 (3)
O(2Cl)	3010 (5)	3421 (4)	985 (3)	5·2 (2)
O(3Cl)	2881 (7)	1924 (5)	1957 (3)	7·5 (3)
O(4Cl)	4455 (5)	3548 (4)	2129 (2)	5·6 (2)
O(*W*)	222 (7)	0	0	4·9 (3)
N(1)	4379 (5)	8238 (4)	1268 (2)	3·5 (2)
C(2)	5702 (7)	8784 (5)	848 (3)	4·5 (3)
C(3)	7047 (8)	8010 (6)	951 (4)	5·0 (3)
C(4)	6743 (8)	6740 (6)	640 (4)	5·3 (3)
C(5)	5431 (7)	6209 (5)	1068 (4)	4·5 (3)
C(6)	4049 (7)	6978 (5)	976 (3)	4·2 (3)
C(7)	2670 (6)	6479 (5)	1372 (3)	3·8 (3)
C(8)	1359 (7)	7292 (7)	1207 (3)	5·1 (3)
C(9)	1693 (7)	8506 (5)	1570 (4)	4·3 (3)
C(10)	3047 (7)	9050 (5)	1190 (4)	4·6 (3)
C(11)	1838 (6)	8413 (5)	2483 (3)	3·8 (3)
C(12)	624 (7)	9103 (6)	2913 (4)	5·0 (3)
C(13)	730 (7)	8917 (6)	3789 (4)	5·0 (3)
C(14)	520 (6)	7605 (6)	3978 (3)	4·5 (3)
C(15)	1373 (6)	6753 (5)	3458 (3)	3·7 (3)
O(C15)	1548 (4)	5691 (4)	3671 (2)	4·7 (2)
N(16)	1871 (5)	7141 (4)	2737 (3)	3·4 (2)
C(17)	2803 (6)	6296 (5)	2280 (3)	3·9 (3)
H(2)	432	451	393	
H(2)	459	368	478	
H(3)	270	280	345	
H(3)	209	327	445	
H(4)	334	170	493	
H(4)	236	121	429	
H(5)	443	99	330	
H(5)	478	47	405	
H(6)	400	703	33	
H(7)	245	567	111	
H(8)	122	735	65	
H(8)	44	702	146	
H(9)	106	912	148	
H(10)	297	909	61	
H(10)	317	973	134	
H(11)	280	875	263	
H(12)	72	1002	271	
H(12)	18	388	226	
H(13)	174	927	400	
H(13)	−7	941	407	
H(14)	94	755	460	
H(14)	73	229	103	
H(17)	374	630	240	
H(17)	267	533	250	
H(N1)	463	814	182	
H(O*W*)	30	533	464	

* The chemical studies carried out up to now have shown that a *trans*-quinolizidine chair–chair arrangement fused in the 3 and 5 positions to another quinolizidine skeleton is rigid, *e.g.* it is unsusceptible to conformational changes.

$4{\cdot}36)^2$ if $F_o < 4{\cdot}36$; $w = 1$, if $4{\cdot}36 \leq F_o \leq 13{\cdot}78$ and $w = (13{\cdot}78/F_o)^2$ if $F_o > 13{\cdot}78$. The final R and R_w values were 0·052 and 0·062 respectively. The final positional and isotropic thermal parameters are listed in Table 1.* All calculations were performed on a NOVA 1200 minicomputer using programs included in the *XTL/E-XTL Structure Determination System* (Syntex, 1976).

Discussion. An illustration of the cation of the title compound with its bond lengths and valency angles is presented in Fig. 1. These values are similar to those obtained for other sparteine derivatives. The N(16)–C(15) and C(15)–O distances of 1·362 (7) and 1·241 (7) Å, respectively, are comparable with the distances observed in the lactam groups in lupanine and oxosparteine derivatives. The torsion angles of the 15-oxosparteine cation are presented in Fig. 2. The asymmetry parameters (Duax & Norton, 1975) for the piperidine rings are:

ring A $\Delta C_s^3 = 0{\cdot}7$, $\Delta C_2^{1,2} = 1{\cdot}58$, $\Delta C_2^{3,4} = 0{\cdot}7°$;
ring B $\Delta C_s^1 = 0{\cdot}6$, $\Delta C_2^{6,7} = 9{\cdot}6$, $\Delta C_2^{1,6} = 5{\cdot}5°$;
ring C $\Delta C_s^8 = 1{\cdot}6$, $\Delta C_2^{7,17} = 20{\cdot}1$, $\Delta C_s^9 = 37{\cdot}5°$;
ring D $\Delta C_2^{12,13} = 5{\cdot}5$, $\Delta C_s^{11} = 36{\cdot}5$, $\Delta C_s^{12} = 12{\cdot}9°$.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35671 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

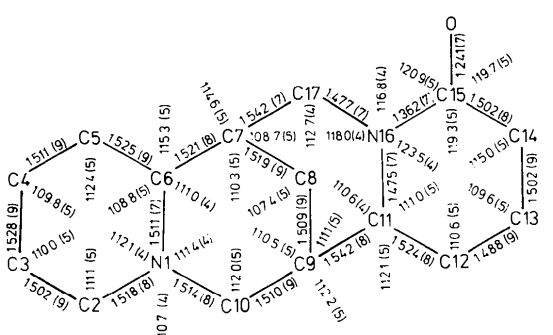


Fig. 1. Bond distances (Å) and valency angles (°) with their e.s.d.'s.

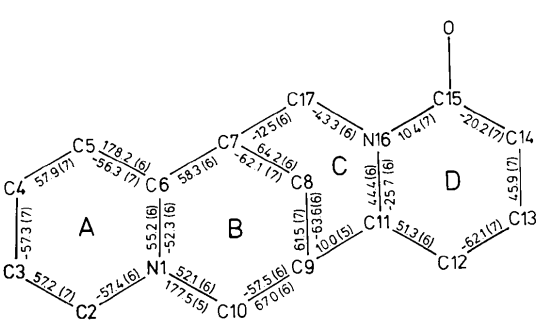


Fig. 2. Torsion angles (°) with their e.s.d.'s.

Table 2. *Least-squares planes*

Equations of planes

Plane 1: $0{\cdot}9312X + 0{\cdot}3623Y - 0{\cdot}040Z - 4{\cdot}7556 = 0$
Plane 2: $-0{\cdot}8646X - 0{\cdot}1990Y - 0{\cdot}4614Z + 5{\cdot}1959 = 0$

Deviations of atoms from the planes (Å)

Plane (1): C(7) 0·011 (6), C(17) −0·011 (6), C(9) −0·013 (6), C(11) 0·011 (6), C(8)* −0·759 (6), N(16)* −0·489 (4), χ^2 16·28

Plane (2): C(11) −0·026 (6), N(16) 0·032 (4), C(15) −0·051 (5), C(14) 0·028 (6), C(12)* 0·440 (6), C(13)* −0·281 (6), χ^2 198·43

* Atom not included in plane calculations.

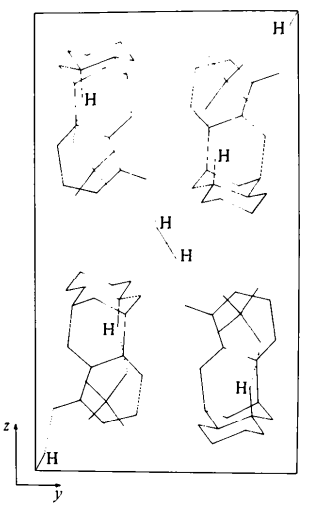
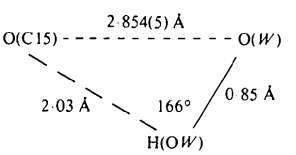


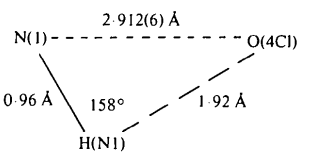
Fig. 3. Projection of the unit cell along [100].

Rings A and B have chair conformations. The conformation of ring C is a boat, slightly flattened at N(16) and sharpened at C(8). Deviations from mean-square planes for rings C and D, listed in Table 2, indicate a distorted half-chair conformation for ring D.

Two cations of 15-oxosparteine are linked together by hydrogen bonds utilizing one water molecule situated on the twofold axis (see Fig. 3). The geometry of the hydrogen bonds is presented below:



Another hydrogen bond involves the protonated N(1) atom and an O atom of the perchlorate anion:



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4-Hydroxy-4-phenylhexanamide, an Anticonvulsant Molecule

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Abstract. $C_{12}H_{17}NO_2$, orthorhombic, *Pcca*, $a = 23{\cdot}025$ (4), $b = 10{\cdot}366$ (2), $c = 10{\cdot}069$ (4) Å, $V = 2403{\cdot}6$ Å^3, $Z = 8$, $D_m = 1{\cdot}14$ (by flotation), $D_c = 1{\cdot}15$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 8·39 mm^{-1}. The structure was solved by application of direct methods, and refined by full-matrix least squares to a final R of 0·082 for 707 reflections with $I \geq 2\sigma(I)$. The bond distances in the benzene ring are rather short owing to libration. There is an intermolecular hydrogen bond [O(1)···O(2) = 2·768 (6) Å].

Introduction. The title compound [also known as γ-hydroxy-γ-ethyl-γ-phenylbutyramide (HEPB)] has long been known for its anticonvulsant activity. It was previously named EPP because it was believed to be 5-ethyl-5-phenyl-2-pyrrolidinone when it was designed and synthesized to penetrate the blood–brain barrier to inhibit γ-aminobutyric acid–α-oxoglutaric acid transaminase (GABA-T). In spite of the many papers published about its biological activity, its structure has only recently been revised by proton and carbon magnetic resonance (Joseph-Nathan, Massieu, Carvajal & Tapia, 1978) to establish its structural formula as HEPB.

The geometrical configuration of this substance may be an aid to the understanding of its mechanism of action at a molecular level. For this reason and to confirm unambiguously its structural formula a crystal structure determination was undertaken.

Crystals of adequate size for X-ray analysis were obtained, after many different trials, from H_2O–EtOH by slow evaporation at room temperature. A crystal of irregular shape with maximum and minimum linear dimensions of 0·6 and 0·3 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. 17 centred reflections and least-squares refinement produced the unit-cell dimensions and the orientation matrix for data collection. The θ–2θ scan technique at a rate of

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