given in Table1.* Bond lengths and bond angles are listed in Table 2.

The etheno bridge is in the 6β,14β position and the dimethylmethanol substituent is in the 7β position of the morphinan skeleton. This means that neither during the Ullmann reaction nor during the treatment in liquid ammonia has epimerization occurred. The synthetic pathway developed for the title compound is therefore applicable for the preparation of this novel class of deoxygenated etorphine analogues. These new etorphine analogues may contribute to the knowledge of structure–activity relationships of analgesics.

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* Lists of structure factors, anisotropic temperature factors, H-atom parameters and distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44251 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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17-Oxosparteine *N*(1)-Oxide Hydrochloride

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Abstract. $[C_{15}H_{25}N_2O_2]^+.Cl^-$, $M_r = 300{\cdot}7$, trigonal, $P3_2$, $a = 9{\cdot}365\ (1)$, $c = 15{\cdot}166\ (1)$ Å, $V = 1151{\cdot}9\ (4)$ Å^3, $Z = 3$, $D_x = 1{\cdot}301\ (1)$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 21{\cdot}19$ cm^{-1}, $F(000) = 486$, $T = 292$ K, $R = 0{\cdot}051$ for 900 unique reflections. The piperidine rings *A, B, C* and *D* have chair, chair, sofa and chair conformations respectively. The quinolizidine and quinolizidone moieties have *trans* and quasi-*trans* configurations. A hydrogen bond of 2·873 (5) Å is observed between the chloride anion and oxygen atom O(1) of the cation.

Introduction. The present work is a continuation of detailed studies on the chemistry and structure of sparteine *N*-oxides (Skolik, Łangowska & Wiewiórowski, 1971, 1974; Bratek-Wiewiórowska, Skolik & Wiewiórowski, 1977; Bratek-Wiewiórowska, Skolik, Łangowska & Wiewiórowski, 1974; Łangowska & Wiewiórowski, 1977), which have included the X-ray investigations of a number of sparteine *N*-oxide derivatives: sparteine *N*(16)-oxide sesquiperchlorate (Srivastava & Przybylska, 1969), episparteine *N*(16)-oxide sesquiperchlorate (Małuszyńska & Okaya, 1977*a*), episparteine *N*(16)-oxide semiperchlorate (Małuszyńska, Kałuski & Wiewiórowski, 1987), sparteine *N*(16)-oxide monoperchlorate (Kałuski & Małuszyńska, 1978), 2-phenylsparteine *N*(16)-oxide monoperchlorate (Małuszyńska & Okaya, 1977*b*), episparteine *N*(16)-oxide dihydrochloride trihydrate (Katrusiak, Hoser & Kałuski, 1983) and lupanine *N*(16)-oxide monoperchlorate (Kałuski, Gusiev, Struchkov, Skolik, Baranowski & Wiewiórowski, 1972). 17-Oxosparteine *N*(1)-oxide hydrochloride [hereinafter referred to as (I)] is the first *N*(1)-oxide of sparteine and the second oxo-derivative of sparteine *N*-oxide investigated by X-rays so far; unlike lupanine *N*(16)-oxide perchlorate [hereinafter (II)] it has the oxo-substituent located in the inner ring of the sparteine skeleton, in ring *C*. The X-ray analysis of the title compound was undertaken to establish the conformation of the cation and the geometry of its intermolecular hydrogen bonding.

Experimental. Recrystallization from ethanol solution, colourless prisms, crystal fragment 0·40 × 0·35 × 0·30 mm, Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Unit cell from least-squares fit to 15 reflections ($12 \leq 2\theta \leq 25°$); 2θ–θ mode with variable scan speed depending on the reflection intensity: 2·0 to 29·3° min^{-1}, two control reflections monitored every 50 current measurements, 1108 reflections measured up to $2\theta = 115°$, index range

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Table 1. *Final atomic parameters and U_{eq} for the refined atoms and for the hydrogen atom of the hydroxy group*

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}(Å^2)
Cl	0·5757 (2)	0·0475 (3)	0·0	0·068
N(1)	0·6705 (7)	0·6898 (6)	0·6562 (3)	0·045
O(1)	0·7139 (5)	0·6149 (5)	0·7249 (3)	0·053
C(2)	0·8176 (8)	0·8555 (9)	0·6360 (5)	0·056
C(3)	0·8727 (9)	0·9672 (10)	0·7172 (6)	0·072
C(4)	0·7345 (9)	0·9865 (10)	0·7543 (5)	0·060
C(5)	0·5805 (9)	0·8172 (9)	0·7730 (4)	0·061
C(6)	0·5274 (8)	0·7106 (9)	0·6899 (4)	0·049
C(7)	0·3695 (8)	0·5461 (8)	0·6986 (4)	0·047
C(8)	0·3216 (8)	0·4608 (9)	0·6078 (4)	0·047
C(9)	0·4543 (8)	0·4236 (8)	0·5829 (4)	0·046
C(10)	0·6205 (9)	0·5821 (8)	0·5745 (4)	0·049
C(11)	0·4544 (9)	0·2947 (8)	0·6490 (4)	0·051
C(12)	0·3317 (9)	0·1171 (8)	0·6182 (5)	0·059
C(13)	0·3342 (11)	−0·0086 (9)	0·6824 (5)	0·071
C(14)	0·3098 (9)	0·0264 (10)	0·7778 (6)	0·070
C(15)	0·4286 (9)	0·2061 (9)	0·8025 (5)	0·061
N(16)	0·4094 (7)	0·3151 (7)	0·7384 (3)	0·053
C(17)	0·3782 (8)	0·4312 (9)	0·7648 (5)	0·049
O(17)	0·3524 (6)	0·4487 (6)	0·8449 (3)	0·073
H(1)	0·4200	0·2234	0·3660	0·070

h −10/5, *k* 0/10 and *l* 0/16; profile analysis method of Lehmann & Larsen (1974), 900 independent reflections ($R_{int} = 0{\cdot}028$) with $I \geq 1{\cdot}96\sigma_I$; Lp corrections only.

The position of the Cl atom was located from the Patterson map. All remaining non-hydrogen atoms were found from a Fourier map. The absolute configuration of the cation and the correct enantiomorphic space group were assigned according to the previous determination of the absolute configuration of naturally occurring sparteine derivatives as C(7)*S* and C(9)*S* (Klyne, Scopes, Thomas, Skolik, Gawroński & Wiewiórowski, 1974). The hydrogen at O(1) was located from a difference Fourier map, the positions of all the other H atoms were calculated from the geometry for the cation and recalculated after each cycle of refinement. The H atoms were assigned isotropic temperature factors depending on the vibrations of their carriers. They contributed in the structure-factor calculations but were not refined.

Full-matrix least-squares refinement, minimization of $\sum w(|F_o| - |F_c|)^2$ where $w = 5{\cdot}5450/\sigma^2(F_o)$, final $R = 0{\cdot}051$, $wR = 0{\cdot}047$, $S = 1{\cdot}47$, max. parameter change in the last cycle of refinement $\Delta/\sigma = 0{\cdot}028$; max. and min. electron densities in the final difference Fourier map: 0·18 and −0·23 e Å^{-3}. The final atomic parameters are listed in Table 1.[*] Scattering factors including real and imaginary parts of anomalous dispersion from *International Tables for X-ray Crystallography* (1974); a RIAD-32 computer and *SHELX*76 program (Sheldrick, 1976).

Discussion. The stereodiagram in Fig. 1 shows the geometry of the cation as well as the atom numbering.

The bond lengths and valency angles of (I) are listed in Table 2. All are in good agreement with those obtained for similar sparteine derivatives (Kałuski, Skolik & Wiewiórowski, 1978). Bond distances N(16)—C(17) and C(17)—O(17) are comparable with the corresponding distances in the lactam groups of lupanine and oxosparteine derivatives (Doucerain,

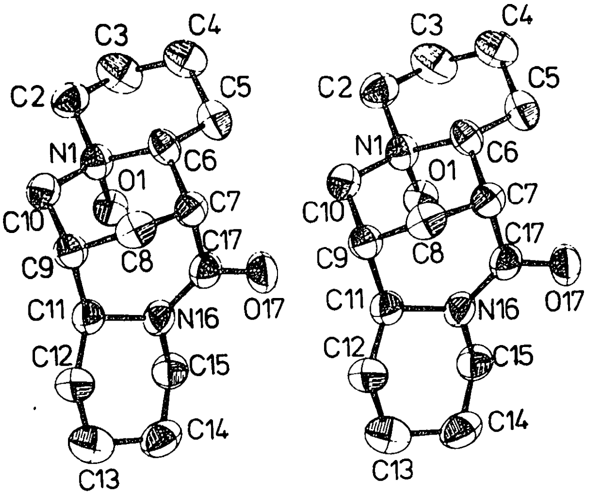

Fig. 1. *ORTEP* view (Johnson, 1965) of the cation at 50% probability. H atoms are omitted for clarity.

Table 2. *Bond lengths* (Å) *and valency angles* (°)

O(1)—N(1)	1·421 (9)	C(9)—C(8)	1·493 (14)
C(2)—N(1)	1·506 (8)	C(10)—C(9)	1·520 (8)
C(6)—N(1)	1·529 (12)	C(11)—C(9)	1·567 (12)
C(10)—N(1)	1·521 (9)	C(12)—C(11)	1·543 (9)
C(3)—C(2)	1·528 (12)	N(16)—C(11)	1·466 (10)
C(4)—C(3)	1·505 (15)	C(13)—C(12)	1·540 (13)
C(5)—C(4)	1·547 (9)	C(14)—C(13)	1·521 (13)
C(6)—C(5)	1·534 (10)	C(15)—C(14)	1·530 (11)
C(7)—C(6)	1·518 (8)	N(16)—C(15)	1·484 (12)
C(8)—C(7)	1·542 (10)	C(17)—N(16)	1·318 (13)
C(17)—C(7)	1·507 (12)	O(17)—C(17)	1·263 (10)

C(2)—N(1)—O(1)	108·4 (5)	C(10)—C(9)—C(8)	111·4 (7)
C(6)—N(1)—O(1)	107·7 (5)	C(11)—C(9)—C(8)	110·2 (6)
C(6)—N(1)—C(2)	110·4 (7)	C(11)—C(9)—C(10)	114·0 (7)
C(10)—N(1)—O(1)	109·4 (6)	C(9)—C(10)—N(1)	113·6 (6)
C(10)—N(1)—C(2)	109·8 (5)	C(12)—C(11)—C(9)	110·6 (6)
C(10)—N(1)—C(6)	111·1 (6)	N(16)—C(11)—C(9)	111·7 (8)
C(3)—C(2)—N(1)	111·3 (6)	N(16)—C(11)—C(12)	108·1 (5)
C(4)—C(3)—C(2)	111·7 (7)	C(13)—C(12)—C(11)	110·0 (6)
C(5)—C(4)—C(3)	111·3 (8)	C(14)—C(13)—C(12)	112·2 (9)
C(6)—C(5)—C(4)	110·7 (6)	C(15)—C(14)—C(13)	111·2 (6)
C(5)—C(6)—N(1)	109·5 (6)	N(16)—C(15)—C(14)	109·0 (6)
C(7)—C(6)—N(1)	112·1 (7)	C(15)—N(16)—C(11)	111·9 (8)
C(7)—C(6)—C(5)	115·0 (6)	C(17)—N(16)—C(11)	126·3 (7)
C(8)—C(7)—C(6)	109·2 (5)	C(17)—N(16)—C(15)	121·2 (7)
C(17)—C(7)—C(6)	114·1 (6)	N(16)—C(17)—C(7)	119·9 (7)
C(17)—C(7)—C(8)	109·2 (7)	O(17)—C(17)—C(7)	117·6 (9)
C(9)—C(8)—C(7)	106·5 (6)	O(17)—C(17)—N(16)	122·4 (8)

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44217 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

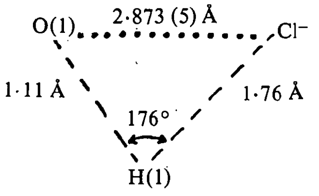
Table 3. *Torsion angles* (°)

Ring *A*			
N(1)—C(2)—C(3)—C(4)	−55·9 (8)	C(4)—C(5)—C(6)—N(1)	57·4 (7)
C(2)—C(3)—C(4)—C(5)	53·9 (8)	C(5)—C(6)—N(1)—C(2)	−59·2 (7)
C(3)—C(4)—C(5)—C(6)	−55·4 (8)	C(6)—N(1)—C(2)—C(3)	58·1 (7)
Ring *B*			
N(1)—C(6)—C(7)—C(8)	−59·9 (7)	C(8)—C(9)—C(10)—N(1)	55·1 (7)
C(6)—C(7)—C(8)—C(9)	65·0 (7)	C(9)—C(10)—N(1)—C(6)	−47·3 (7)
C(7)—C(8)—C(9)—C(10)	−61·8 (6)	C(10)—N(1)—C(6)—C(7)	50·1 (6)
Ring *C*			
C(7)—C(17)—N(16)—C(11)	6·7 (7)	C(11)—C(9)—C(8)—C(7)	65·1 (7)
C(17)—N(16)—C(11)—C(9)	−2·9 (7)	C(9)—C(8)—C(7)—C(17)	−60·8 (7)
N(16)—C(11)—C(9)—C(8)	−34·1 (7)	C(8)—C(7)—C(17)—N(16)	25·7 (7)
Ring *D*			
N(16)—C(15)—C(14)—C(13)	54·7 (7)	C(13)—C(12)—C(11)—N(16)	−57·4 (7)
C(15)—C(14)—C(13)—C(12)	−51·0 (9)	C(12)—C(11)—C(16)—C(15)	64·5 (7)
C(14)—C(13)—C(12)—C(11)	52·0 (8)	C(11)—N(16)—C(15)—C(14)	−63·2 (7)
Exocyclic torsion angles			
O(1)—N(1)—C(10)—C(9)	71·5 (7)	C(4)—C(5)—C(6)—C(7)	−175·6 (7)
C(3)—C(2)—N(1)—C(10)	−179·5 (7)	C(5)—C(6)—C(7)—C(17)	−63·0 (7)
C(2)—N(1)—C(10)—C(9)	−169·3 (7)	C(6)—C(7)—C(17)—N(16)	−97·2 (7)
N(1)—C(10)—C(9)—C(11)	−69·3 (7)	C(6)—C(7)—C(17)—O(17)	83·8 (7)
C(10)—C(9)—C(11)—C(12)	−148·1 (7)	C(7)—C(17)—N(16)—C(15)	176·4 (7)
C(9)—C(11)—C(12)—C(13)	179·1 (7)	C(17)—N(16)—C(15)—C(14)	125·8 (7)

Chiaroni & Riche, 1976; Skrzypczak-Jankun & Kałuski, 1978; Małuszyńska, Hoser & Kałuski, 1979; Skrzypczak-Jankun, Hoser, Kałuski & Perkowska, 1980; Katrusiak, Hoser, Grzesiak & Kałuski, 1980; Hoser, Katrusiak, Kałuski & Perkowska, 1981) and indicate a conjugated bond system. The three C—N(1) bond lengths, with a mean of 1·519 Å, are significantly longer than an N—C(sp^3) bond involving a protonated N atom – 1·500 Å (Birnbaum, 1967). Such C—N bond lengthening was also reported for (II) and all other sparteine *N*-oxides.

Torsion angles are listed in Table 3. They indicate a chair conformation for ring *A*, chair for *B*, sofa for *C* and chair for *D*. The conformation of the sparteine skeleton of (I) is very similar to that observed for 17-oxosparteine (Katrusiak, Hoser, Grzesiak & Kałuski, 1980), apparently little changed by the oxidation at N(1).

The unit-cell contents are shown in Fig. 2. The cation and the chloride anion are connected by a hydrogen bond involving H(1) at O(1). The geometry of this hydrogen bond is presented schematically below:



By way of contrast, in the structure of (II) a hydrogen bond was formed between the oxygen at N(16) and the lactam oxygen at C(2) while the perchlorate anion was not involved in any hydrogen bonding. In episparteine *N*(16)-oxide sesquiperchlorate (Małuszyńska & Okaya, 1977*a*) and in sparteine *N*(16)-oxide sesquiperchlorate (Srivastava & Przybylaka, 1969) the very strong hydrogen bonds between the oxygens in the symmetry-equivalent *N*-oxide groups bound the cations into dimers. In all the sparteine *N*-oxides investigated so far the oxygen atoms of the *N*-oxide group show a strong tendency to form very short hydrogen bonds. No other intermolecular contacts, shorter than the sum of the appropriate van der Waals radii, are present in the structure of (I).

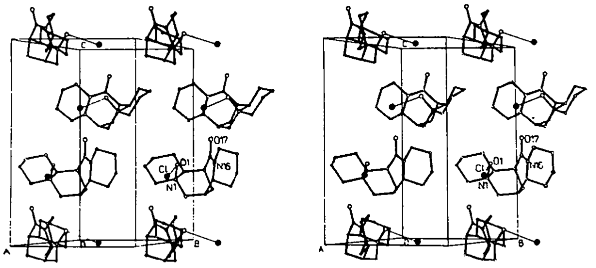


Fig. 2. Stereoscopic view of the unit-cell contents. H atoms have been omitted for clarity.

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4,4′-Biphenyldithiol

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Abstract. $C_{12}H_{10}S_2$, $M_r = 218{\cdot}3$, monoclinic, $P2_1/c$, $a = 11{\cdot}858\,(3)$, $b = 7{\cdot}462\,(1)$, $c = 6{\cdot}049\,(1)$ Å, $\beta = 96{\cdot}45\,(2)^\circ$, $V = 531{\cdot}9\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}36\,(1)$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu(\text{Cu } K\alpha) = 39{\cdot}95$ cm^{-1}, $F(000) = 228$, $T = 292$ K, $R = 0{\cdot}055$ for 548 unique reflections. The molecule is positioned on a centre of symmetry in the crystal and consequently the two phenyl rings are restricted by symmetry to be coplanar. There are slight deformations of the phenyl rings similar to those observed in biphenyl.

Introduction. Despite the considerable interest in aryl–sulfuric and alkyl–aryl–sulfuric compounds and their importance in the chemistry of polymers there are very few data describing their molecular and crystal structures. The present paper on the structure of 4,4′-biphenyldithiol [hereinafter referred to as (I)], a sulfuric derivative of biphenyl, is a continuation of our X-ray diffraction studies on this group of compounds (Andreetti, Garbarczyk & Królikowska, 1981; Garbarczyk, 1984). Our main interest connected with (I) concerned the planarity of the molecule and the way the S substituents influence the phenyl fragments, compared with the molecule of biphenyl.

Experimental. Crystals of (I) were obtained by slow evaporation of a cyclohexane solution oversaturated at 353 K. The crystal selected for data collection was a colourless prism of dimensions $0{\cdot}12 \times 0{\cdot}07 \times 0{\cdot}05$ mm. The crystallization proved to be very difficult and a number of crystallization techniques and different solvents were tried. The crystal used was the only specimen obtained which was suitable for X-ray investigation. It was mounted on a Syntex $P2_1$ diffractometer; graphite-monochromated Cu $K\alpha$ radiation was used. The unit-cell parameters were determined by a least-squares fit to 15 automatically centred reflections ($21{\cdot}0 \le 2\theta \le 71{\cdot}5^\circ$). The θ–2θ scan technique with a variable scan speed ($2{\cdot}1$ to $29{\cdot}3^\circ$min^{-1}) was applied. Two control reflections were monitored after each 50 intensity measurements and showed no systematic variation in intensity. Up to $2\theta = 115{\cdot}0^\circ$ 850 reflections were measured, with index ranges h −12/12, k 0/8, l 0/6. The background and integrated intensity for each reflection were calculated according to the profile-analysis method of Lehmann & Larsen (1974). Of 729 unique reflections ($R_{\text{int}} = 0{\cdot}035$) 548 reflections had $I \ge 1{\cdot}96\,\sigma_I$ and were used in the structure refinement. Only Lp corrections were applied. The structure was solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). After the non-hydrogen atoms were refined ansiotropically, all the H atoms, except for H at S(1), were located from a ΔF map. They were included in the refinement with isotropic temperature factors. An electron density peak was observed in the ΔF map at $1{\cdot}32$ Å from S(1) and could be considered to correspond to the hydrogen of the SH group. However, all attempts to refine this hydrogen were unsuccessful and finally it was excluded from the atom list. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0{\cdot}000170F_o^2]$. The final discrepancy indices were $R = 0{\cdot}0552$, $wR = 0{\cdot}0614$ and $S = 1{\cdot}4$. For all the parameters the final Δ/σ was smaller than $0{\cdot}06$, the max. and min. peaks on the final ΔF map were $0{\cdot}22$ and $-0{\cdot}23$ e Å^{-3}.

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