identical interplanar angles are found in MPBZZP, though the diiron complex BAGRUW forms a slightly flatter boat with corresponding angles of 127 and 153°.

Most 1,2-diazepine rings, even with different double-bond positions and widely differing substituents, adopt similar boat conformations. It seems likely, therefore, that the parent 1,2-diazepine (2) would have a similar conformation to the oxide form. The lower barrier to inversion of the oxide compared to that for (2) is likely, therefore, to result mainly from the reduction of the N=N double-bond character. This would facilitate rotation about bonds and provide a lower-energy pathway for ring inversion.

The van der Waals contacts are mostly within the expected ranges. There is, however, a close intermolecular interaction between a methylene hydrogen atom and the azoxy group with distances H(11)···O(1) = 2·45, H(11)···N(2) = 2·71 and H(11)···N(3) = 2·61 Å.

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Structure of Diethyl (1*S*)-1-[(1*S*)-10-Camphorsulfonylamino]ethylphosphonate,* $C_{16}H_{30}NO_6PS$

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Abstract. $M_r = 395·4$, monoclinic, $P2_1$, $a = 11·040$ (2), $b = 17·367$ (4), $c = 12·381$ (3) Å, $\beta = 121·5$ (5)°, $V = 2024·0$ (9) Å^3, $Z = 4$, $D_m = 1·31$ (1), $D_x = 1·30$ Mg m^{-1}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 2·38$ mm^{-1}, $F(000) = 848$, $T = 292$ K, $R = 0·061$ for 3060 reflexions. There are two crystallographically independent molecules in the asymmetric unit. The main chain in both molecules adopts an anticlinal–[+synclinal(g$^+$)]–[+synclinal(g$^+$)] conformation. The O(ethyl)—P—O—C torsion angles are −120 (1), 81 (1)° for (I) and −153 (1), 92 (1)° for (II). The norbornane skeletons are undistorted with C—C—C bridge angles 93·4 (8) and 92·7 (9)°, respectively. The two independent molecules form a dimer through N—H···O(2) hydrogen bonds. The absolute configuration of the molecules was assigned as (*S*,*S*) with reference to the known *S* configuration of the (1*S*)-10-camphorsulfonyl moiety.

Introduction. 1-Aminoalkylphosphonic acids are often reported in the literature as mimetics of the naturally occurring amino acids (Hilderbrand, Curley-Joseph, Lubansky & Henderson, 1982). Although much attention has been paid to them because of their biological importance, the literature concerning the synthesis and absolute configuration of these compounds in optically active forms is still scarce (Głowiak, Sawka-Dobrowolska, Kowalik, Mastalerz, Soroka & Zoń, 1977; Vasella & Voeffray, 1982; Kowalik, Sawka-Dobrowolska & Głowiak, 1984).

In our research in this field we encountered some difficulties because of the lack of 1-aminoalkylphosphonic acids in crystalline form suitable for X-ray examination.

In this work we present the crystal structure and absolute configuration of the *N*-(10-camphorsulfonyl) derivative of diethyl 1-aminoethylphosphonate. We believe that this *N*-blocking group will be suitable in our attempts to solve the problem of absolute configuration of other optically active aminophosphonates.

Experimental. Title compound obtained by reaction of optically active diethyl 1-aminoethylphosphonate with (1*S*)-10-camphorsulfonyl chloride in the presence of

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* 10-Camphorsulfonic acid is 7,7-dimethyl-2-oxobicyclo[2.2.1]-hept-1-ylmethanesulfonic acid.

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triethylamine as HCl acceptor. Clear colourless crystals grown from diethyl ether at room temperature, dimensions $0{\cdot}1 \times 0{\cdot}15 \times 0{\cdot}2$ mm; D_m by flotation in dichloromethane/1,2-dichloroethane; monoclinic $P2_1/m$ or $P2_1$ from Weissenberg photographs, $P2_1/m$ excluded owing to known optical activity; Syntex $P2_1$ computer-controlled four-circle diffractometer, scintillation counter, graphite monochromator; cell parameters by least squares from setting angles of 15 reflexions with $25 \leq 2\theta(\mathrm{Cu}) \leq 35°$ measured on diffractometer; 3280 independent reflexions; $2\theta_{\max} = 125°$; variable θ–2θ scans, scan rate 2·0–29·3° min^{-1}, depending on intensity; two standards ($21\bar{4}$, $53\bar{2}$) every 50 reflexions, variation in intensities ±3%; correction for Lorentz and polarization factors, not for absorption; 3070 with $I > 1{\cdot}96\sigma(I)$ used for structure determination; index range h 0 to 12, k 0 to 20, l −13 to 12; calculations performed with Syntex (1976) *XTL* system; neutral atom scattering factors and anomalous dispersion from *International Tables for X-ray Crystallography* (1974); direct methods, Syntex (1976) version of *MULTAN* (Germain, Main & Woolfson, 1971); full-matrix least squares, minimizing $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$; 10 reflexions showing large $(F_o - F_c)/\sigma(F_o)$ ratio excluded in final stages of refinement; difference synthesis revealed 4 H atoms, 18 H atoms placed at computed positions; H atoms of CH_3 and C_2H_5 groups could not be located; non-H atoms refined anisotropically, H atoms with fixed coordinates and isotropic temperature factor, $B = 6{\cdot}5$ Å^2; ethyl and methyl moieties vibrate strongly; $(\Delta/\sigma)_{\max}$ in final LS cycle 0·06 for positional parameters and 0·12 for anisotropic temperature factors; max. and min. electron densities in final difference map 0·35 and −0·32 e Å^{-3}; $R = 0{\cdot}061$, $wR = 0{\cdot}077$, $S = 4{\cdot}92$ (3060 observations, 450 variables). Final positional parameters are given in Table 1.*

Table 1. *Positional parameters and B_{eq}(Å^2) values for the non-*H *atoms with e.s.d.'s in parentheses*

	x	y	y	B_{eq}*
P(1)	−0·0541 (3)	−0·1000	−0·9519 (2)	4·4 (2)
P(2)	−0·4939 (3)	−0·2603 (2)	−0·0483 (2)	4·5 (2)
S(1)	0·0494 (2)	−0·2947 (2)	−0·7558 (2)	4·2 (2)
S(2)	−0·4581 (2)	−0·2357 (2)	−0·3433 (2)	3·7 (2)
O(1.1)	0·0525 (7)	−0·1376 (6)	−0·9839 (7)	6·7 (8)
O(1.2)	−0·2023 (6)	−0·1030 (5)	−1·0556 (5)	4·5 (5)
O(1.3)	0·0062 (9)	−0·0164 (5)	−0·9029 (7)	6·3 (8)
O(1.4)	−0·0272 (7)	−0·3648 (4)	−0·8117 (6)	5·0 (6)
O(1.5)	0·1751 (7)	−0·2797 (5)	−0·7572 (6)	6·1 (6)
O(1.6)	0·0849 (9)	−0·1945 (4)	−0·4172 (7)	6·3 (7)
O(2.1)	−0·4999 (8)	−0·3451 (5)	−0·0964 (7)	5·6 (7)
O(2.2)	−0·3589 (6)	−0·2372 (5)	0·0637 (6)	5·0 (6)
O(2.3)	−0·6250 (7)	−0·2562 (6)	−0·0303 (7)	6·8 (8)
O(2.4)	−0·3216 (7)	−0·2382 (5)	−0·3288 (7)	5·6 (6)
O(2.5)	−0·5429 (7)	−0·3052 (4)	−0·3750 (6)	4·4 (5)
O(2.6)	−0·7533 (8)	−0·0441 (5)	−0·5311 (9)	8·1 (9)
N(1)	−0·0610 (7)	−0·2262 (5)	−0·8340 (6)	3·7 (6)
N(2)	−0·4325 (7)	−0·2036 (5)	−0·2106 (7)	3·8 (6)
C(1.1)	0·0008 (9)	−0·3109 (5)	−0·5485 (8)	3·5 (7)
C(1.2)	0·0256 (10)	−0·2568 (6)	−0·4411 (9)	4·4 (8)
C(1.3)	−0·0305 (11)	−0·2973 (7)	−0·3688 (10)	6·0 (9)
C(1.4)	−0·0911 (11)	−0·3726 (6)	−0·4476 (9)	4·7 (8)
C(1.5)	−0·2267 (10)	−0·3494 (6)	−0·5762 (9)	4·8 (8)
C(1.6)	−0·1631 (9)	−0·3055 (7)	−0·6430 (8)	4·5 (8)
C(1.7)	0·0169 (9)	−0·3912 (6)	−0·4864 (9)	3·8 (7)
C(1.8)	−0·0230 (13)	−0·4596 (6)	−0·5752 (12)	6·1 (10)
C(1.9)	0·1720 (10)	−0·4026 (7)	−0·3657 (11)	5·6 (10)
C(1.10)	0·1018 (9)	−0·2882 (6)	−0·5947 (8)	4·3 (7)
C(1.11)	−0·0157 (10)	−0·1441 (6)	−0·8067 (9)	4·0 (8)
C(1.12)	−0·0921 (13)	−0·1022 (7)	−0·7465 (10)	6·0 (10)
C(1.13)	0·0163 (14)	−0·1649 (7)	−1·1050 (11)	6·1 (11)
C(1.14)	0·0387 (15)	−0·1035 (9)	−1·1764 (11)	8·0 (13)
C(1.15)	−0·0193 (16)	0·0466 (7)	−0·9892 (12)	7·0 (12)
C(1.16)	0·0271 (19)	0·1195 (8)	−0·9105 (14)	9·2 (17)
C(2.1)	−0·5201 (8)	−0·0908 (5)	−0·4763 (8)	3·3 (6)
C(2.2)	−0·6462 (12)	−0·0346 (6)	−0·5306 (11)	5·9 (10)
C(2.3)	−0·6054 (13)	0·0327 (7)	−0·5820 (13)	7·1 (12)
C(2.4)	−0·4590 (11)	0·0087 (6)	−0·5559 (11)	5·2 (10)
C(2.5)	−0·3555 (14)	0·0165 (7)	−0·4130 (14)	7·0 (12)
C(2.6)	−0·4032 (12)	−0·0502 (6)	−0·3550 (10)	5·3 (9)
C(2.7)	−0·4753 (11)	−0·0799 (6)	−0·5764 (11)	5·5 (9)
C(2.8)	−0·5937 (15)	−0·1042 (9)	−0·7153 (11)	7·7 (13)
C(2.9)	−0·3328 (13)	−0·1202 (7)	−0·5408 (13)	7·8 (7)
C(2.10)	−0·5732 (9)	−0·1723 (5)	−0·4679 (9)	3·6 (7)
C(2.11)	−0·5468 (10)	−0·2004 (6)	−0·1843 (9)	4·1 (7)
C(2.12)	−0·5768 (14)	−0·1165 (7)	−0·1616 (12)	7·4 (12)
C(2.13)	−0·3753 (14)	−0·3877 (8)	−0·0768 (14)	7·4 (13)
C(2.14)	−0·3703 (21)	−0·4626 (9)	−0·0245 (18)	10·7 (20)
C(2.15)	−0·6135 (17)	−0·2733 (14)	0·0897 (14)	11·1 (22)
C(2.16)	−0·6071 (23)	−0·3550 (16)	0·1123 (20)	14·9 (30)

* $B_{eq} = \frac{1}{3}\sum_i B_{ii}$.

The absolute configuration was assigned as (*S*,*S*) with reference to the known *S* configuration of the (1*S*)-10-camphorsulfonyl moiety.

Discussion. The molecular structure of the two independent molecules and the atom numbering are shown in Fig. 1. Corresponding bond lengths and angles (see Table 2) in the molecules do not differ significantly.

The coordination around the P atoms is distorted tetrahedral, the angles varying from 115·8 (5) to 100·2 (5)°. All angles involving the non-ester O atoms are greater than the others. The lengths of the esterified P—O bonds are 1·566 (10)–1·579 (9) Å and are close to those observed in the diethyl phosphates of magnesium, propylguanidinium, arginine and 2-amino-5,5-dimethyl-1-pyrrolinium (Ezra & Collin, 1973; Furberg & Solbakk, 1972, 1973; Griffith, Rutherford & Robertson, 1982). The lengths of the non-esterified P—O bonds 1·461 (7) and 1·465 (7) Å, compare with similar bonds in the structures cited above. The C—O—P and C—C—O angles are close to 120 and 110°, mean 122·3 and 109·6° (e.s.d. *ca* 3°), respectively. In the phosphonate ethyl groups the C—C bond lengths are unusually short and the thermal motion is very high. Similar effects have been reported for other diethyl phosphates (Hazel & Collin, 1972; Ezra & Collin, 1973).

The geometry of the 10-camphorsulfonyl moiety agrees satisfactorily with those reported for other camphor and camphorsulfonyl moieties (Allen & Rogers, 1971; Bear & Trotter, 1975; Phillips & Trotter, 1976; Lebioda, Marshall & Flynn, 1984).

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and the results of least-squares planes calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42192 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The norbornane skeletons have normal geometry; the angles between the C(1), C(7), C(4) plane and the four-atom planes of the six-membered ring, C(1), C(2), C(3), C(4) and C(1), C(4), C(5), C(6) are 126 (1) and 125 (1)° for (I) and 124 (1) and 127 (1)° for (II). The internal torsion angles of the six-membered ring are −3 (1), −71 (1), 71 (1), 2 (1), −72 (1), 73 (1)° for (I) and 1 (1), −73 (1), 69 (1), 5 (1), −74 (1), 72 (1)° for (II), compared with idealized values of 0, ±54, 0, ±54° (Bucourt, 1974). The C(7) bridgehead angles of 93·4 (8) and 92·7 (9)° are close to those observed in similar structures.

The main chain of the molecule is described by the χ_1 [P–C(11)–N–S], χ_2 [C(11)–N–S–C(10)] and χ_3 [N–S–C(10)–C(1)] torsion angles with values of 125 (1), 64 (1), 73 (1)° for (I) and 119 (1), 67 (1), 70 (1)° for (II). The molecules thus adopt the anticlinal–[+synclinal(g^+)]–[+synclinal(g^+)] conformation.

The conformation of the diethyl phosphonate group is synclinal–anticlinal [81 (1), −120 (1)°] for (I) and synclinal–antiperiplanar [92 (1), −153 (1)°] for (II). In contrast, other structures containing diethyl phosphonate groups have synclinal–synclinal conformation.

An interesting feature of the present structure is the formation of dimers. The two molecules are oriented in antiparallel fashion; N of molecule (I) is hydrogen bonded to O(2)(x, y, $-1+z$) of (II) with the H(1)···O(2) distance 1·99 Å [N···O 2·85 (1) Å, angle N–H···O 140°]. The N atom of molecule (II) is in turn hydrogen bonded to O(2)(x, y, $1+z$) of (I) with H(1)···O(2) 1·96 Å [N···O 2·85 (1) Å, angle N–H···O 147°].

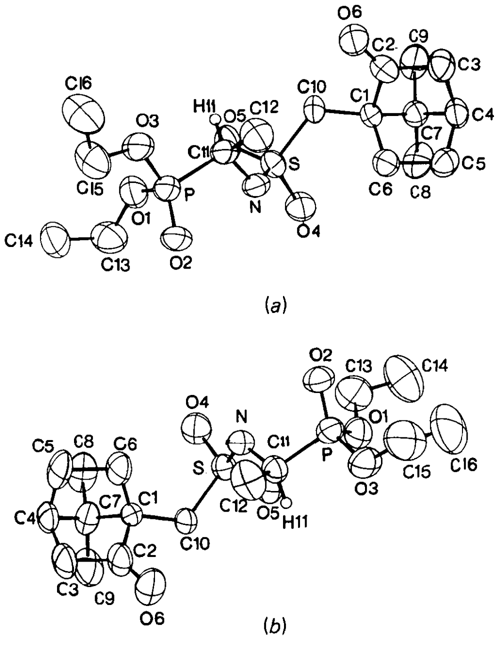

Fig. 1. An *ORTEP* (Johnson, 1976) drawing of the title compound with the atom-numbering scheme. (*a*) Molecule I, (*b*) molecule II.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

	(I)	(II)		(I)	(II)
P–O(1)	1·566 (10)	1·577 (9)	P–O(1)–C(13)	125·0 (8)	123·9 (8)
P–O(2)	1·461 (7)	1·465 (7)	P–O(3)–C(15)	122·0 (9)	122·6 (10)
P–O(3)	1·579 (8)	1·576 (9)	O(1)–C(13)–C(14)	110·2 (11)	110·0 (13)
P–C(11)	1·790 (10)	1·798 (10)	O(3)–C(15)–C(16)	106·7 (12)	111·6 (17)
O(1)–C(13)	1·42 (1)	1·47 (2)	P–C(11)–N	107·9 (7)	106·9 (7)
C(13)–C(14)	1·49 (2)	1·44 (2)	P–C(11)–C(12)	112·0 (8)	111·7 (8)
O(3)–C(15)	1·45 (2)	1·46 (2)	N–C(11)–C(12)	110·7 (9)	111·6 (9)
C(15)–C(16)	1·51 (2)	1·44 (4)	C(11)–N–S	120·7 (7)	121·9 (7)
C(11)–C(12)	1·57 (2)	1·55 (2)	O(4)–S–O(5)	118·8 (5)	120·1 (5)
C(11)–N	1·49 (2)	1·46 (2)	O(4)–S–N	105·4 (5)	105·7 (5)
N–S	1·613 (9)	1·615 (8)	O(4)–S–C(10)	111·1 (5)	111·0 (5)
S–C(10)	1·766 (9)	1·778 (10)	O(5)–S–N	107·1 (5)	107·9 (4)
S–O(4)	1·438 (8)	1·422 (9)	O(5)–S–C(10)	104·9 (5)	102·3 (5)
S–O(5)	1·421 (9)	1·450 (8)	N–S–C(10)	109·3 (5)	109·5 (5)
C(10)–C(1)	1·55 (2)	1·55 (2)	S–C(10)–C(1)	121·8 (7)	119·9 (7)
C(1)–C(2)	1·53 (1)	1·54 (2)	C(2)–C(1)–C(6)	103·0 (8)	104·0 (9)
C(1)–C(6)	1·56 (1)	1·55 (1)	C(6)–C(1)–C(7)	102·3 (8)	105·4 (8)
C(1)–C(7)	1·56 (1)	1·57 (2)	C(7)–C(1)–C(2)	101·4 (8)	99·5 (8)
C(2)–C(3)	1·50 (2)	1·51 (2)	C(2)–C(1)–C(10)	109·2 (8)	108·6 (8)
C(2)–O(6)	1·22 (1)	1·19 (2)	C(6)–C(1)–C(10)	119·0 (8)	120·1 (8)
C(3)–C(4)	1·56 (2)	1·53 (2)	C(7)–C(1)–C(10)	119·5 (8)	116·7 (8)
C(4)–C(5)	1·56 (2)	1·53 (2)	C(1)–C(2)–O(6)	125·9 (10)	125·7 (11)
C(5)–C(6)	1·54 (2)	1·59 (2)	C(3)–C(2)–O(6)	127·4 (10)	128·3 (12)
C(7)–C(8)	1·52 (2)	1·58 (2)	C(1)–C(2)–C(3)	106·7 (9)	106·0 (10)
C(7)–C(9)	1·59 (2)	1·56 (2)	C(2)–C(3)–C(4)	101·3 (9)	102·4 (11)
C(7)–C(4)	1·54 (2)	1·56 (2)	C(3)–C(4)–C(5)	106·9 (9)	106·8 (11)
O(1)–P–O(2)	114·1 (5)	115·7 (5)	C(3)–C(4)–C(7)	102·9 (9)	102·5 (10)
O(1)–P–O(3)	105·0 (5)	103·6 (5)	C(5)–C(4)–C(7)	102·8 (9)	103·2 (10)
O(2)–P–O(3)	115·0 (5)	114·2 (5)	C(4)–C(5)–C(6)	102·3 (9)	103·5 (11)
O(1)–P–C(11)	105·1 (5)	104·9 (5)	C(5)–C(6)–C(1)	104·1 (8)	101·5 (9)
O(2)–P–C(11)	115·8 (5)	113·6 (5)	C(4)–C(7)–C(1)	93·4 (8)	92·7 (9)
O(3)–P–C(11)	100·2 (5)	103·5 (5)	C(1)–C(7)–C(8)	115·3 (9)	113·9 (10)
			C(1)–C(7)–C(9)	111·4 (8)	114·2 (10)
			C(4)–C(7)–C(8)	114·4 (9)	113·7 (10)
			C(4)–C(7)–C(9)	111·3 (9)	112·6 (10)
			C(8)–C(7)–C(9)	110·2 (9)	109·1 (10)

		(I)	(II)
ω	C(13)–O(1)–P–O(3)	−120 (1)	−153 (1)
ω^1	O(1)–P–O(3)–C(15)	81 (1)	92 (1)
	O(1)–P–C(11)–N	−69 (1)	−69 (1)
	O(2)–P–C(11)–N	58 (1)	58 (1)
	O(3)–P–C(11)–N	−178 (1)	−178 (1)
χ_1	P–C(11)–N–S	125 (1)	119 (1)
χ_2	C(11)–N–S–C(10)	64 (1)	67 (1)
χ_3	N–S–C(10)–C(1)	73 (1)	70 (1)
	C(11)–N–S–O(5)	−49 (1)	−44 (1)
	C(11)–N–S–O(4)	−177 (1)	−174 (1)
	C(10)–C(1)–C(2)–O(6)	18 (1)	21 (2)
	C(1)–C(2)–C(3)–C(4)	−3 (1)	1 (1)
	C(1)–C(6)–C(5)–C(4)	2 (1)	5 (1)
	C(2)–C(3)–C(4)–C(5)	−71 (1)	−73 (1)
	C(3)–C(4)–C(5)–C(6)	71 (1)	69 (1)
	C(5)–C(6)–C(1)–C(2)	−72 (1)	−74 (1)
	C(6)–C(1)–C(2)–C(3)	73 (1)	72 (1)

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The Ammonium Salt of 5-Nitrobarbituric Acid, $NH_4^+.C_4H_2N_3O_5^-$

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Abstract. M_r = 190·12, triclinic, $P\bar{1}$, a = 4·9184 (4), b = 7·6947 (5), c = 9·8195 (6) Å, α = 104·911 (5), β = 92·666 (6), γ = 105·085 (6)°, V = 344·2 (2) Å^3, Z = 2, D_m = 1·83 (1), D_x = 1·835 (1) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·164 mm^{-1}, $F(000)$ = 196, T = 295 K. R = 0·038 for 1596 unique observed reflections. The anion is nearly planar with only small deviations from mirror symmetry related to a mirror plane perpendicular to the molecular plane. Formation of the NH_4^+ salt of 5-nitrobarbituric acid (dilituric acid) does not increase the delocalization of π-electron density to the nitro group. The crystal structure is stabilized by intermolecular hydrogen bonds involving all the H atoms.

Introduction. The structure determination of the ammonium salt of 5-nitrobarbituric acid is part of a study of structural changes associated with the steps in the series: compounds, nitro compound, anion of nitro compound. The present structure determination was thus performed to study structural changes connected with 5-nitrobarbituric acid salt formation.

Experimental. Dilituric acid prepared as described by Hartman & Sheppard (1943). Colourless crystals (prisms) of the ammonium salt were obtained by slow cooling of a solution in boiling water (1·0 g ammonium salt needs 200 ml of boiling water). D_m by flotation in CH_2BrCl/CCl_4.

Crystal: 0·1 × 0·2 × 0·3 mm, Enraf–Nonius CAD-4*F* diffractometer, graphite-monochromatized Mo $K\alpha$, lattice parameters from setting angles for 25 reflections with 7·79 < θ < 13·52°. 1996 unique reflections (h 0→6, k $\overline{10}$→9, l $\overline{13}$→13) with 2·0 < θ < 30·0°, 1596 with $I > 2{\cdot}5\sigma(I)$ used in refinement process together with 'less-than' reflections [$I < 2{\cdot}5\sigma(I)$] with calculated values greater than the observed (1864 contributing reflections). Mixed $\omega/2\theta$ scan technique, scan angle = 1·40° + 0·35° tan θ. Standard reflections $10\bar{4}$, 022 and 031 used for orientation control every 100 reflections, $03\bar{3}$ used for intensity check every 10800 s of exposure time, standard intensity variations < 2·3% of mean value. Lp correction, absorption ignored; direct methods, full-matrix least-squares refinement of anisotropic non-H atoms. Positional H parameters from $\Delta\rho$ map (remaining peak heights less than half those associated with H atoms). Refinements of H(x,y,z) with sin θ/λ < 0·40 Å^{-1}, fixed isotropic H temperature factor. $\sum w(\Delta|F|)^2$ minimized, final R = 0·038, wR = 0·074,[*] weights $w = 1/\{1 + [(F_o - 6{\cdot}0)/8{\cdot}0]^2\}$ gave average $w\Delta(|F|)^2$ almost independent of $|F_o|$, S = 2·34. Ratio of maximum least-squares shift to error in final refinement cycle = 0·0018. Minimum and maximum in final $\Delta\rho$ map: −0·4 and 0·4 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs from *SHELX*76 (Sheldrick, 1976) and *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). The positional and thermal parameters are given in Table 1.

Discussion. The atoms in the anion show only small deviations from a least-squares plane defined by all the non-H atoms (except ammonium N). The deviations are within 0·03 (3) Å for endocyclic atoms and 0·05 (3) Å for exocyclic atoms. The anion does not

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[*] Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and $NH_4^+\cdots O$ distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42231 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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