Table 2. *Bond angles* (°)

O–P–N	112·5 (2)	C(3)–C(4)–C(5)	120·4 (5)
O–P–C(1)	114·8 (2)	C(4)–C(5)–C(6)	120·9 (5)
O–P–C(7)	109·3 (2)	C(1)–C(6)–C(5)	118·9 (5)
N–P–C(1)	104·9 (2)	P–C(7)–C(8)	120·2 (4)
N–P–C(7)	111·6 (2)	P–C(7)–C(12)	120·4 (4)
C(1)–P–C(7)	103·3 (2)	C(8)–C(7)–C(12)	119·4 (5)
P–C(1)–C(2)	116·5 (4)	C(7)–C(8)–C(9)	120·7 (5)
P–C(1)–C(6)	123·5 (4)	C(8)–C(9)–C(10)	119·9 (5)
C(2)–C(1)–C(6)	119·9 (5)	C(9)–C(10)–C(11)	119·7 (5)
C(1)–C(2)–C(3)	120·9 (5)	C(10)–C(11)–C(12)	120·5 (5)
C(2)–C(3)–C(4)	119·0 (6)	C(7)–C(12)–C(11)	119·7 (5)
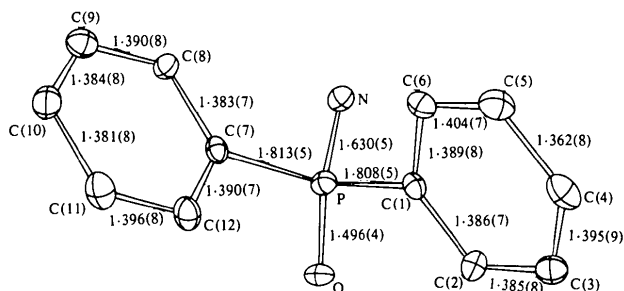
Fig. 1. View of the molecule showing the atom numbering and bond distances (Å).

stage. They were included with isotropic temperature factors and further refinement yielded a final $R = 0·033$ and $R_w = 0·035$. Unit weights were found appropriate. Atomic coordinates are listed in Table 1.* A final difference map was essentially flat.

Discussion. The molecule is depicted in Fig. 1 which also shows intramolecular bond distances. Angles between bonded atoms are given in Table 2. All bond lengths and angles are unexceptional. The phenyl groups are planar to within experimental accuracy with a dihedral angle of 76·2°. The phenyl C–C distances are essentially identical, with a mean value of 1·39 (1) Å. The phosphorous bond angles are close to tetrahedral, ranging between 114·8 (2) and 103·3 (2)°, as was found in the similar compound *N,N*-dimethyl-

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* Lists of structure factors, anisotropic thermal parameters and H atom positional parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35700 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

phosphinamide (Mazhar-ul-Haque & Caughlan, 1966). This rules out any possibility of the molecule possessing twofold symmetry, which indicates that the DPPA ligands in the lanthanide adducts, mentioned in the *Introduction*, must necessarily be disordered.

All calculations were performed on a PDP 11/45 computer with the *Enraf–Nonius SDP* crystallographic programs.

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2-Amino-1,3-thiazoline Hydrochloride

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Abstract. $C_3H_7N_2S^+.Cl^-$, $M_r = 138·62$, monoclinic, $P2_1/c$, $a = 7·240$ (7), $b = 6·176$ (2), $c = 13·462$ (8) Å, $\beta = 100·10$ (3)°, $D_m = 1·51$, $D_c = 1·55$ Mg m^{-3}, $Z = 4$, $\mu = 7·619$ mm^{-1}. Final $R = 0·110$ for 657 observed reflections. The nonplanar thiazoline ring assumes an envelope conformation. The crystal structure is stabilized by NH⋯Cl hydrogen bonds and van der Waals interactions.

Introduction. 2-Amino-1,3-thiazoline (2-AT) is one of the two components into which the aminothiol radio-protectant (S)-2-aminoethylisothiouronium bromide hydrobromide (AETBr.HBr) is converted in the body. Despite being toxic, 2-AT is also known to have the radioprotective properties of its parent compound, AET (Distefano, Leary & Little, 1959). The X-ray analysis of its hydrochloride was therefore undertaken

as part of a program of X-ray studies on the structural characteristics of chemical radioprotectants (Vijayan, Mani, Vedavathi & Ramaseshan, 1975; Vijayan & Mani, 1977; Vedavathi & Vijayan, 1977, 1979).

Transparent, needle-like crystals of 2-AT.HCl were obtained by slow evaporation from a dilute solution in absolute alcohol. The lattice parameters were refined by the least-squares method. Intensity data from five reciprocal levels hkl, $k = 0$ to 4, were recorded by the multiple-film equi-inclination Weissenberg method using Cu $K\alpha$ radiation. The intensities were estimated visually and were corrected for Lp, spot-shape and absorption effects.

The structure was solved by direct methods and refined by the block-diagonal least-squares procedure with anisotropic thermal parameters for all the non-hydrogen atoms. From a difference electron density map, five out of the seven structurally independent H atoms could be identified. However, in the subsequent structure factor calculations, the H atoms were included with the positional coordinates obtained from geometrical considerations and the equivalent B's of the heavy atoms to which they are attached. The coordinates of the H atoms were not refined. The final R value was 0·110 for 657 observed reflections. The weighting scheme was of the form $1/(a + bF_o + cF_o^2)$, where $a = 0·893$, $b = 0·0519$ and $c = 0·0011$. The scattering factors for the nonhydrogen atoms were taken from Cromer & Waber (1965) and for H from Stewart, Davidson & Simpson (1965). The final coordinates of the nonhydrogen atoms are given in Table 1.*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35659 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional coordinates* ($\times 10^4$) *and equivalent B values* (Å²)

The standard deviations are given in parentheses. Equivalent B values were calculated following Hamilton (1959).

	x	y	z	B
Cl	1519 (8)	7742 (10)	784 (4)	4·5 (0·3)
S	2994 (9)	4605 (12)	3106 (4)	4·8 (0·3)
C(1)	3464 (35)	2809 (44)	4157 (17)	5 (1)
C(2)	3433 (36)	607 (44)	3755 (17)	5 (1)
C(3)	2091 (27)	2556 (36)	2315 (14)	3·7 (1·0)
N(1)	2299 (24)	675 (31)	2721 (13)	4·2 (0·9)
N(2)	1268 (27)	2944 (33)	1392 (14)	4·9 (1·0)
H1(C1)	2447	2977	4587	
H2(C1)	4727	3142	4571	
H3(C2)	2810	−438	4189	
H4(C2)	4500	0	3667	
H5(N1)	1691	−632	2338	
H6(N2)	1171	4471	1134	
H7(N2)	738	1706	944	

Discussion. The dimensions of the molecule are given in Fig. 1. The exocylic bond C(3)−N(2) is short and its length is comparable, within limits of error, to that of the endocylic C(3)−N(1) bond. Also, the internal valency angles at C(3) and N(1) are slightly larger than those of the other internal angles in the five-membered ring. These features have, however, been observed in other aminothiazoline structures (Mornon & Raveau, 1971; Mornon & Bally, 1972; Bally, 1973; Bally & Mornon, 1973; Cohen-Addad & Viallet, 1978). The distribution of bond lengths indicates that the molecule can be considered as a resonance hybrid of the canonical forms (I) and (II) with nearly equal contributions from them.

(I)   (II)
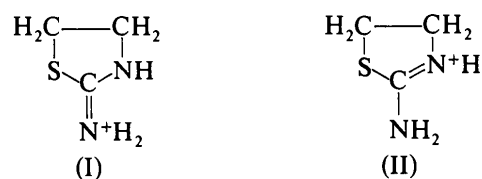
As observed in other similar structures, the thiazoline ring has an envelope conformation with the atoms S, C(3), N(1) and C(2) lying in a plane and the fifth ring atom C(1) deviating from this plane by 0·35 Å. The dihedral angles which define this conformation are given in Table 2.

The crystal structure is stabilized by NH···Cl hydrogen bonds and van der Waals interactions. The parameters of the hydrogen bonds are given in Table 2.
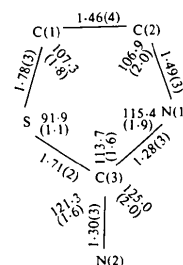
Fig. 1. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses.

Table 2. *Dihedral angles* (°) *and hydrogen-bond parameters*

The positions of the H atoms were not refined and the N−H distance in all cases is 1·0 Å.

C(3)−S−C(1)−C(2)	18 (2)
S−C(1)−C(2)−N(1)	−23 (2)
C(1)−C(2)−N(1)−C(3)	19 (3)
C(2)−N(1)−C(3)−S	−6 (3)
N(1)−C(3)−S−C(1)	−7 (2)

N(1)···Cl^{II}	3·14 (2) Å	N(2)···Cl^{III}	3·28 (2) Å
Cl^{II}···N(1)−H5(N1)	29°	Cl^{III}···N(2)−H7(N2)	42°
N(2)···Cl^{I}	3·09 (2) Å	N(2)···Cl^{II}	3·33 (2) Å
Cl^{I}···N(2)−H6(N2)	8°	Cl^{II}···N(2)−H7(N2)	32°

Symmetry code: (I) x,y,z; (II) $x, 1 − y, z$; (III) $\bar{x}, 1 − y, \bar{z}$.
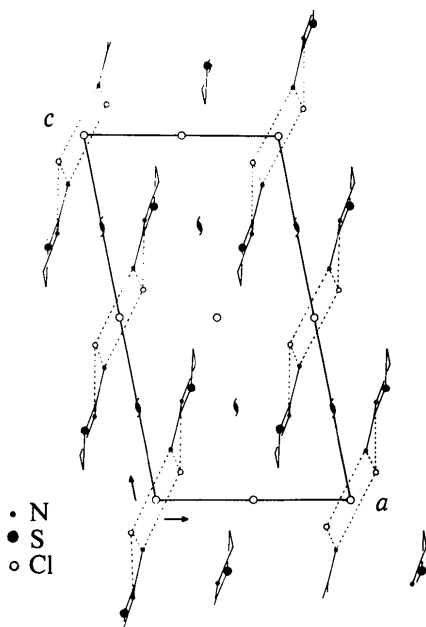
Fig. 2. View of the crystal structure as seen along the *b* axis. Broken lines represent the hydrogen bonds.

columns of 2-AT molecules. Thus, each double column has a central polar region consisting of (partially) positively charged NH and NH_2 groups and negatively charged chloride ions, flanked by non-polar regions made up of CH_2 groups and S atoms. The double columns are packed together in the crystal in such a way that each is surrounded by six other double columns.

Geometrical considerations indicate that two are linear hydrogen bonds whereas the third is a bifurcated hydrogen bond. These hydrogen bonds give rise to double columns of 2-AT molecules running parallel to the *b* direction and centered around $x = 0$, $z = 0$ and $x = 0$, $z = \frac{1}{2}$ respectively (Fig. 2). Each double column consists of two individual columns of 2-AT molecules which are related to each other by a linear array of inversion centers. The chloride ions in each double column are sandwiched between the two individual

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Structure of *trans*-3-*tert*-Butyl-4-cyano-1-cyclohexenyl Acetate: a Sofa Conformation*

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Abstract. $C_{13}H_{19}NO_2$, $M_r = 221.30$, m.p. 332 K, monoclinic, $P2_1/c$, $Z = 4$, $a = 8.498$ (4), $b = 18.094$ (7), $c = 10.630$ (4) Å, $\beta = 127.88$ (3)°, $V = 1290.1$ Å³, $d_m = 1.15$ (2), $d_x = 1.15$ Mg m⁻³, $\mu(Cu\,K\alpha) = 0.624$ mm⁻¹, $F(000) = 480$. With X-ray diffraction data collected on a three-circle diffractometer, the crystal structure was solved by direct methods and refined to an *R* of 0.053 for 1778 observed reflections. The molecular structure shows a sofa conformation; C(5) is the only C atom out of the mean plane of the other C atoms of the ring.

* Strained Cyclic Molecules. III.