Table 4. *Mean torsion angles compared with those of the syn derivative*

E.s.d.'s are < 0·8°.

	This work	*syn* derivative*
N(4)–C(5)–C(7)–N(8)	−167·6°	16·5°
N(4)–C(5)–C(7)–C(11)	12·5	−163·7
C(6)–C(5)–C(7)–N(8)	14·2	−161·1
C(6)–C(5)–C(7)–C(11)	−165·8	18·8
C(5)–C(7)–N(8)–O(9)	−1·0	177·4
C(5)–C(7)–C(11)–O(12)	−103·3	101·7
C(5)–C(7)–C(11)–O(13)	77·0	−76·8
N(8)–C(7)–C(11)–O(12)	76·7	−78·5
N(8)–C(7)–C(11)–O(13)	−103·0	103·0
O(9)–N(8)–C(7)–C(11)	179·0	−2·5
C(7)–N(8)–O(9)–C(10)	180·0	−176·4

The difference results from the inversion of the methoxyimino and carboxyl groups by rotation (~180°) around C(5)–C(7).

* Laurent, Durant & Evrard (1981).

N(3)–H(31)···N(4) [N(3)···N(4)($-x + 1, -y + 1, -z + 2$) 3·032 (6) Å]. Moreover, the carbonyl groups are connected through the exocyclic amines of neighbouring dimers: O(12)···H(32)–N(3) [O(12)···N(3)($x + 1, y, z$) 3·002 (6) Å].

Table 5. *Hydrogen-bond lengths* (Å) *and angles* (°) *with e.s.d.'s*

N(3)–H(31)	0·90 (6)	H(32)–N(3)–C(2)	117 (5)
N(3)–H(32)	0·74 (6)	H(32)–N(3)–H(31)	128 (6)
C(6)–H(61)	0·84 (5)	H(61)–C(6)–S(1)	122 (4)
H(31)–N(3)–C(2)	115 (4)	H(61)–C(6)–C(5)	127 (4)

We thank Dr J. Perronnet (Roussel–Uclaf, Romainville, France) for providing the sample.

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Ethyl 2-Amino-α-(*E*-methoxyimino)-4-thiazoleacetate Hydrobromide

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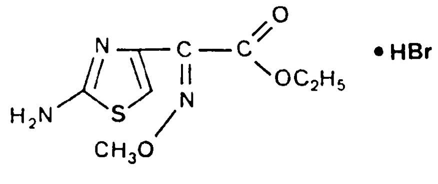
Abstract. $C_8H_{12}N_3O_3S^+.Br^-$, $M_r = 310·2$, monoclinic, $P2_1/c$, $a = 10·812$ (5), $b = 8·895$ (5), $c = 13·385$ (5) Å, $\beta = 109·34$ (5)°, $D_m = 1·71$ (2), $D_c = 1·70$ Mg m^{-3}, $Z = 4$, $T = 294$ K; $R = 0·048$ for 2093 reflexions. The three characteristic groups, aminothiazolyl, oxyimino and carboxyl, are nearly coplanar. The molecules are linked to each other by hydrogen bonds through exocyclic amines and Br^- ions.

Introduction. An X-ray crystallographic study of the title compound was undertaken to disclose any conformational change which might have occurred in the change from the free base (Laurent, Durant & Evrard, 1981) to the thiazolium salt.



Suitable crystals were obtained from different solutions: methanol/water, *n*-butanol/water/methanol, ethanol/water/acetone and ethyl acetate. They all have the same lattice parameters. The space group was determined from photographs. Final cell dimensions and intensities were measured on a Nonius CAD-4 four-circle computer-controlled diffractometer. No absorption corrections were made. Table 1 lists the instrumental settings for the data collection.

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Table 1. *Instrumental settings for the data collection*

Crystal size: 0·30 × 0·17 × 0·15 mm
Source: Cu $K\bar{\alpha}$; λ = 1·54178 Å
Graphite monochromator
$2 \le \theta \le 72°$
$\Delta 2\theta = 0{\cdot}80 + 0{\cdot}30\,\mathrm{tg}\,\theta\,(°)$
Aperture = $2{\cdot}5 + 0{\cdot}5\,\mathrm{tg}\,\theta$ (mm)
Confidence level: $2{\cdot}5\,\sigma$, where $\sigma^2 = S + B + (0{\cdot}03S)^2$, S being the scan and B the background count
Number of independent reflexions: 2421
Total observed: 2093

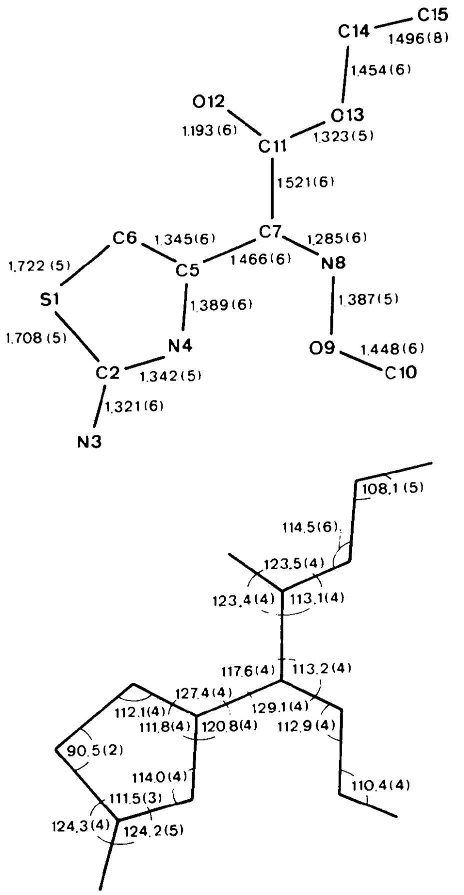

Fig. 1. Atom numbering, bond lengths (Å) and angles (°) with e.s.d.'s in parentheses.

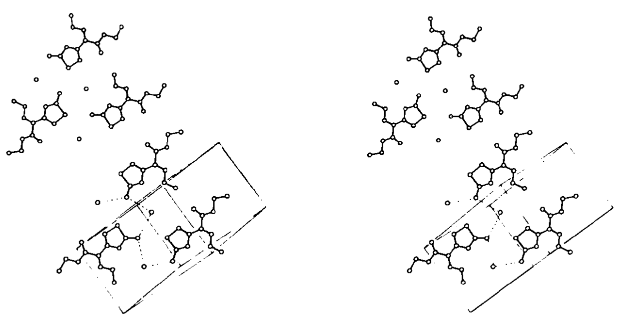

Fig. 2. Stereoview of the unit cell with a representation of the intermolecular hydrogen bonds.

Table 2. *Final coordinates* (×10^4, *for* H ×10^3), *with e.s.d.'s in parentheses and* U_{eq} *values* (Å^2 ×10^4, *for* H ×10^3)

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

	x	y	z	U_{eq}
S(1)	4046 (2)	5798 (2)	3432 (1)	465
C(2)	4015 (5)	7716 (6)	3464 (4)	379
N(3)	4536 (6)	8598 (6)	2915 (5)	608
N(4)	3378 (4)	8217 (5)	4104 (3)	324
C(5)	2915 (5)	7084 (6)	4600 (4)	295
C(6)	3190 (6)	5710 (6)	4310 (4)	387
C(7)	2208 (5)	7445 (6)	5334 (4)	292
N(8)	1905 (5)	8738 (5)	5614 (3)	384
O(9)	2362 (4)	9922 (4)	5159 (3)	497
C(10)	1938 (8)	11344 (7)	5460 (7)	571
C(11)	1694 (5)	6141 (6)	5819 (4)	348
O(12)	1766 (6)	4859 (5)	5582 (4)	722
O(13)	1141 (4)	6577 (4)	6516 (3)	440
C(14)	605 (7)	5361 (7)	6973 (5)	471
C(15)	−148 (7)	6041 (9)	7618 (6)	560
Br(16)	3892 (1)	3017 (1)	8565 (1)	418
H(31)	494 (6)	829 (7)	248 (5)	50
H(32)	458 (6)	955 (8)	296 (5)	50
H(41)	330 (5)	915 (6)	430 (4)	30
H(61)	301 (5)	476 (7)	454 (4)	37
H(101)	209 (6)	1145 (7)	628 (5)	54
H(102)	231 (6)	1205 (8)	501 (5)	54
H(103)	98 (6)	1140 (7)	524 (5)	54
H(141)	14 (6)	471 (7)	641 (5)	43
H(142)	133 (6)	470 (7)	730 (5)	43
H(151)	37 (6)	673 (8)	819 (5)	55
H(152)	−82 (6)	663 (7)	722 (5)	55
H(153)	−69 (6)	517 (7)	811 (5)	55

Table 3. *Mean torsion angles of the thiazolium salt compared with those of the corresponding base* (*Laurent, Durant & Evrard*, 1981)

E.s.d.'s are <0·6°.

	This work	Corresponding base
N(4)–C(5)–C(7)–N(8)	0·9°	−167·6°
N(4)–C(5)–C(7)–C(11)	178·1	12·5
C(6)–C(5)–C(7)–N(8)	−178·5	14·2
C(6)–C(5)–C(7)–C(11)	−1·3	−165·8
C(5)–C(7)–N(8)–O(9)	−1·9	−1·0
C(5)–C(7)–C(11)–O(12)	−4·8	−103·3
C(5)–C(7)–C(11)–O(13)	176·2	77·0
N(8)–C(7)–C(11)–O(12)	172·9	76·7
N(8)–C(7)–C(11)–O(13)	−6·2	−103·0
O(9)–N(8)–C(7)–C(11)	180·0	179·0
C(7)–N(8)–O(9)–C(10)	177·2	180·0

A Patterson map revealed the Br position. All other non-H atoms were located by Fourier maps. The refinement was carried out by full-matrix least squares (Sheldrick, 1976). H atoms were found from difference maps, including one attached to N(4). With anisotropic temperature factors for all non-H atoms and isotropic for H (equal to those of the parent atom), the final $R = \sum||F_o| - |F_c||/\sum|F_o| = 0{\cdot}048$ for the 2093 observed reflexions. The final coordinates are given in Table 2. Fig. 1 gives bond lengths and angles and the atom numbering. Fig. 2 shows the packing.*

Discussion. Compared to the corresponding base, the main differences observed are the inversion of the oxyimino and carboxyl groups by half-rotation around C(5)—C(7) (Table 3) and the coplanarity of the three groups aminothiazolyl, oxyimino and carboxyl (Table 4). This coplanarity allows the formation of an intramolecular hydrogen bond between the protonated N(4) of the thiazolyl ring and O(9) of the oxyimino group; moreover an interaction [C(6)—H(61)···O(12)] occurs between the thiazolyl and carbonyl groups. This conformation leads to a close approach of O(13) and N(8), 2·548 (5) compared to 2·90 Å for the sum of the van der Waals radii (Pauling, 1960).

Each molecule is bonded to two neighbours by N—H···Br⁻ bonds: N(3)···Br⁻(x, $-y+\frac{3}{2}$, $z-\frac{1}{2}$) 3·272 (5), N(3)···Br⁻($-x+1$, $-y+1$, $-z+1$) 3·333 (5) Å.

We thank Dr J. Perronnet (Roussel–Uclaf, Romainville, France) for providing the sample.

Table 4. *Deviations from coplanarity and angles between planes*

Deviations (Å)

(1) Plane through S(1), C(2), N(3), N(4), C(5) and C(6): aminothiazolyl group

S(1)	−0·000	N(4)	−0·005
C(2)	0·005	C(5)	0·005
N(3)	−0·001	C(6)	−0·003

(e.s.d. 0·004 Å)

(2) Plane through C(5), C(7), N(8), O(9) and C(11): oxyimino group

C(5)	−0·004	O(9)	−0·002
C(7)	0·015	C(11)	−0·005
N(8)	−0·003		

(e.s.d. 0·008 Å)

(3) Plane through C(7), C(11), O(12) and O(13): carboxyl group

C(7)	−0·001	O(12)	−0·002
C(11)	0·005	O(13)	−0·002

(e.s.d. 0·003 Å)

Angles (°)

Planes	This work	Corresponding base
1–2	0·6	13·4
2–3	5·5	77·1
1–3	5·3	82·3

(e.s.d. <0·3°)

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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 35828 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,2′,4,4′,6,6′-Hexaisopropylazobenzene*†

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Abstract. $C_{30}H_{46}N_2$, orthorhombic, *Pbnn*, $a = 5{\cdot}9952$ (6), $b = 19{\cdot}630$ (2), $c = 23{\cdot}929$ (2) Å at 220 K giving $Z = 4$ and $D_c = 1{\cdot}03$ Mg m^{-3}. At room temperature, the space group is the same and the cell parameters are approximately 6·016, 19·68 and 24·08 Å. At 115 K the sample is a monoclinic twin with $\gamma = 90{\cdot}2°$. The transition temperature of this reversible transformation is about 200 K. The crystals are yellow to orange needles elongated parallel to **a** and limited by the {010} and {001} forms. A twofold axis relates the two parts of the molecule which extends

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[*] Structures of Hindered Azobenzenes. II.

[†] NRC No. 19079.

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