S(2)	0.56435 (8)	0.51396 (6)	0.14181 (6)	0.0350
O(21)	0.6551 (3)	0.5658 (2)	0.0322 (2)	0.0468
O(22)	0.3840 (2)	0.5447 (2)	0.1701 (2)	0.0504
C(21)	0.6893 (3)	0.5487 (2)	0.2288 (2)	0.0358
C(22)	0.8728 (4)	0.5439 (3)	0.2064 (2)	0.0418
C(23)	0.9711 (4)	0.5774 (3)	0.2706 (3)	0.0511
C(24)	0.8935 (5)	0.6154 (3)	0.3552 (3)	0.0571
C(25)	0.7108 (5)	0.6163 (4)	0.3779 (3)	0.0661
C(26)	0.6081 (4)	0.5835 (3)	0.3145 (3)	0.0567
C(27)	1.0065 (8)	0.6575 (5)	0.4197 (5)	0.0819

Table 2. *Selected geometric parameters (Å, °)*

C(1)—C(2)	1.422 (3)	C(5)—N(8)	1.346 (4)
C(1)—C(6)	1.371 (4)	N(7)—O(71)	1.231 (4)
C(1)—N(10)	1.401 (3)	N(7)—O(72)	1.232 (3)
C(2)—C(3)	1.368 (3)	N(10)—S(1)	1.643 (2)
C(2)—N(20)	1.427 (3)	S(1)—O(11)	1.429 (2)
C(3)—C(4)	1.389 (4)	S(1)—O(12)	1.428 (2)
C(4)—C(5)	1.408 (4)	S(2)—O(21)	1.424 (2)
C(4)—N(7)	1.443 (4)	S(2)—O(22)	1.426 (2)
C(5)—C(6)	1.407 (4)		

N—H distances	0.80 (3)–0.84 (3)	C—H distances	0.82 (3)–0.99 (3)

C(6)—C(1)—C(2)	119.0 (2)	N(8)—C(5)—C(4)	124.7 (3)
N(10)—C(1)—C(2)	117.6 (2)	N(8)—C(5)—C(6)	118.4 (3)
N(10)—C(1)—C(6)	123.4 (2)	C(5)—C(6)—C(1)	122.7 (3)
C(3)—C(2)—C(1)	119.3 (2)	O(71)—N(7)—C(4)	118.9 (3)
N(20)—C(2)—C(1)	119.5 (2)	O(72)—N(7)—C(4)	118.6 (3)
N(20)—C(2)—C(3)	121.2 (2)	O(72)—N(7)—O(71)	122.5 (3)
C(4)—C(3)—C(2)	121.3 (3)	O(11)—S(1)—N(10)	105.7 (1)
C(5)—C(4)—C(3)	120.8 (2)	O(12)—S(1)—N(10)	107.6 (1)
N(7)—C(4)—C(3)	117.3 (3)	O(21)—S(2)—N(20)	105.3 (1)
N(7)—C(4)—C(5)	121.9 (3)	O(22)—S(2)—N(20)	106.7 (1)
C(6)—C(5)—C(4)	116.9 (2)	O(22)—S(2)—O(21)	120.9 (1)

Data collection and cell refinement used *CAD*-4 *Software* (Enraf–Nonius, 1989). The structure was solved with *SHELXS*86 (Sheldrick, 1985). Most calculations were performed with the *CRYSTALS* package (Watkin, Carruthers & Betteridge, 1985). The drawings were prepared with *ORTEP*II (Johnson, 1976).

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2'-Methoxy-5'-methoxycarbonylspiro[acridine-9(10*H*),4'(5'*H*)-thiazole]

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Abstract

The structure of the title ester, methyl 2'-methoxyspiro[acridine-9(10*H*),4'(5'*H*)-thiazole]-5'-carboxylate, $C_{18}H_{16}N_2O_3S$, is described. The compound is of spiro type. The acridine molecule is not planar, but adopts an arch-like conformation. The thiazole ring is in an envelope form. One of the C—C bonds exhibits an unusual value of 1.610 (3) Å. A C=O···H—N-type intermolecular hydrogen bond takes part in the packing of the molecules.

Comment

Isothiocyanates are at present extensively studied as they are used as synthones of various heterocycles (Mukerjee & Ashare, 1991), fluorescence reagents for labelling of biomolecules (Russel & Boron, 1976), and important chemotherapeutics (Zhang & Talalay, 1994). Our interest lies in the study of such new functionalized 9-acridinyl derivatives (Mazagová *et al.*, 1994).

Our goal was to prepare a three-membered heterocycle by the reaction of sodium O-methyl-N-(9-acridinyl)-iminothiocarbonate with methyl bromoacetate. As the ¹H NMR, ¹³C NMR and IR spectra of the obtained compound were not in line with the expected composi-

tion, its structure was determined by X-ray diffraction. Details of the preparation of the title compound, (I), as well as some other derivatives and the results of their spectral studies, will be published elsewhere (Bernát *et al.*, 1995).

(I)
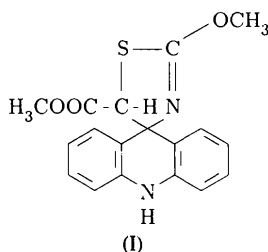
The results of the X-ray analysis (Table 1, Fig. 1) showed that during the above-mentioned reaction, the cyclization proceeds *via* C1—C2. As a consequence, a spiro compound is formed. A search in the Cambridge Structural Database (Allen *et al.*, 1993) showed that the title compound represents the first example of such a spiro compound containing dihydroacridine and thiazoline rings. On the other hand, a similar spiro compound containing quinazoline and thiazoline rings was described by Yamagishi *et al.* (1991).

Fig. 1. *ORTEP* (Johnson, 1965) view of the title compound. The displacement ellipsoids are drawn at the 50% probability level.

While acridine alone is an aromatic molecule and planar, the dihydroacridine ring in (I) adopts an arch shape. The central ring loses its aromatic character because of the formation of four covalent bonds at C2. In line with this, the observed distances of C2—C7 and C2—C4 (Table 2) are close to those of single bonds.

C2 and N2 are shifted away from the plane formed by the remaining four atoms of the ring by 0.442 (3) and 0.281 (3) Å, respectively, indicating the formation of a boat conformation. Both side rings are planar within experimental error and the angles between them and the plane formed by C4, C5, C6 and C7 are 14.1 (1) (ring containing C15 atom) and 15.4 (1)°.

As expected the thiazoline is not planar. The ring adopts an envelope conformation, as shown by the torsion angles. It should be noted that the C1—C2 bond distance exhibits an unusual value of 1.610 (3) Å, indicating weakening of this bond in the present compound. The reason for such an elongation may lie in the steric effects, *i.e.* repulsion between the ester group and the acridine ring. The shortest intramolecular contacts are: O2···C4 3.023 (3), O2···C5 3.198 (3) and C4···C16 2.805 (3) Å. A similar unusual value of 1.57 Å for the thiaxoline C—C bond was found in 2'-amino-3-methylspiro[quinazoline-4(1*H*),5'-thiazoline]-2,4'(3*H*)-dione (Yamagishi *et al.*, 1991). The other bond distances and angles in the molecule have usual values.

In the solid state, C=O···H—N-type hydrogen bonds play an important role in the packing of the molecules (Fig. 1), joining the molecules into chains. The geometric parameters of the hydrogen bond N2—H2···O1($\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$) are N···O 3.199 (2), H···O 2.42 (3) Å and N—H···O 148 (2)°. There is another short contact of 3.300 (4) Å between O3 and C9 [H9···O3 2.91 (3) Å]. No other contacts shorter than 3.41 Å are present. No intermolecular contact up to the limit of 3.6 Å for the S atom (with atoms other than H atoms) was found.
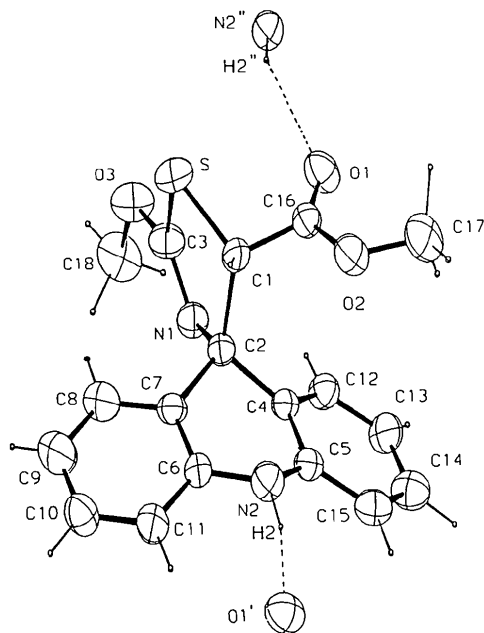
Experimental

The synthesis of the title compound will be described elsewhere (Bernát *et al.*, 1995). Monocrystals suitable for X-ray study were prepared by recrystallization from dichloromethane (m.p. 457–459 K). The density D_m was measured by flotation in *n*-hexane/tetrachloromethane mixture.

Crystal data

$C_{18}H_{16}N_2O_3S$	Cu $K\alpha$ radiation
$M_r = 340.396$	$\lambda = 1.54178$ Å
Monoclinic	Cell parameters from 25
$P2_1/n$	reflections
$a = 7.7640$ (8) Å	$\theta = 22.9–33.2°$
$b = 13.784$ (2) Å	$\mu = 1.879$ mm^{-1}
$c = 15.6920$ (15) Å	$T = 293$ (2) K
$\beta = 103.770$ (8)°	Paralellepiped
$V = 1631.1$ (3) Å^3	0.32 × 0.21 × 0.13 mm
$Z = 4$	Yellow
$D_x = 1.3862$ Mg m^{-3}	
$D_m = 1.38$ Mg m^{-3}	

Data collection

CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
3524 measured reflections
3404 independent reflections
2793 observed reflections
[$I > 2\sigma(I)$]
$R_{int} = 0.0416$

$\theta_{max} = 76.43°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 19$
3 standard reflections
monitored every 85 reflections
intensity decay: 1.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0538$
$wR(F^2) = 0.1445$
$S = 1.104$
3399 reflections
266 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0898P)^2 + 0.4401P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.005$

$\Delta\rho_{max} = 0.382$ e Å^{-3}
$\Delta\rho_{min} = -0.443$ e Å^{-3}
Extinction correction: SHELXL93 (Sheldrick, 1993)
Extinction coefficient: 0.0025 (5)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

The positional parameters of the H atoms were freely refined with tied U values (1.5 × U_{eq} of parent atom for methyl groups and 1.2 × U_{eq} for others).

Data collection: *SDP Structure Determination Package* (B. A. Frenz & Associates Inc., 1985). Cell refinement: *SDP Structure Determination Package*. Data reduction: *SDP Structure Determination Package*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL93*. Geometric calculations: *PARST* (Nardelli, 1983); *SHELXL93*.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S	0.19655 (9)	0.09446 (5)	−0.00800 (4)	0.0582 (2)
O1	−0.0379 (2)	0.26980 (15)	−0.01456 (10)	0.0569 (5)
O2	−0.0209 (2)	0.26736 (14)	0.12977 (10)	0.0514 (4)
O3	0.3876 (3)	0.1541 (2)	−0.11433 (11)	0.0639 (5)
N1	0.4265 (2)	0.23850 (14)	0.01637 (11)	0.0423 (4)
N2	0.4168 (3)	0.2674 (2)	0.28005 (12)	0.0452 (4)
C1	0.1894 (3)	0.1748 (2)	0.08190 (14)	0.0419 (5)
C2	0.3739 (3)	0.23340 (15)	0.09963 (12)	0.0358 (4)
C3	0.3546 (3)	0.1730 (2)	−0.03660 (14)	0.0475 (5)
C4	0.3520 (3)	0.33405 (15)	0.13419 (12)	0.0355 (4)
C5	0.3649 (3)	0.3461 (2)	0.22399 (13)	0.0373 (4)
C6	0.5217 (3)	0.1956 (2)	0.25638 (13)	0.0402 (5)
C7	0.5119 (3)	0.1779 (2)	0.16755 (13)	0.0400 (5)
C8	0.6224 (3)	0.1082 (2)	0.1453 (2)	0.0549 (6)
C9	0.7382 (4)	0.0561 (2)	0.2098 (2)	0.0669 (8)
C10	0.7447 (4)	0.0725 (2)	0.2972 (2)	0.0626 (7)
C11	0.6367 (3)	0.1414 (2)	0.3206 (2)	0.0523 (6)
C12	0.3073 (3)	0.4139 (2)	0.07869 (14)	0.0456 (5)
C13	0.2703 (4)	0.5028 (2)	0.1106 (2)	0.0564 (6)
C14	0.2782 (4)	0.5126 (2)	0.1991 (2)	0.0564 (6)
C15	0.3250 (3)	0.4360 (2)	0.25545 (15)	0.0485 (5)
C16	0.0302 (3)	0.2415 (2)	0.05845 (13)	0.0428 (5)
C17	−0.1551 (4)	0.3420 (3)	0.1187 (2)	0.0652 (7)
C18	0.5320 (5)	0.2087 (3)	−0.1350 (2)	0.0771 (10)

Table 2. *Selected geometric parameters* (Å, °)

S—C3	1.772 (3)	N2—C6	1.387 (3)
S—C1	1.805 (2)	N2—C5	1.394 (3)
O1—C16	1.206 (3)	C1—C16	1.514 (3)
O2—C16	1.322 (3)	C1—C2	1.610 (3)
O2—C17	1.445 (3)	C2—C4	1.514 (3)
O3—C3	1.330 (3)	C2—C7	1.525 (3)
O3—C18	1.450 (4)	C4—C12	1.395 (3)
N1—C3	1.264 (3)	C4—C5	1.399 (3)
N1—C2	1.461 (3)	C6—C7	1.399 (3)

C3—S—C1	88.59 (10)	C7—C2—C1	108.5 (2)
C3—N1—C2	112.3 (2)	N1—C3—S	119.6 (2)
C6—N2—C5	119.1 (2)	C5—C4—C2	118.8 (2)
C2—C1—S	104.55 (14)	N2—C5—C4	118.9 (2)
N1—C2—C4	110.8 (2)	N2—C6—C7	119.7 (2)
N1—C2—C7	110.5 (2)	C6—C7—C2	118.1 (2)
C4—C2—C7	109.3 (2)	O1—C16—O2	124.4 (2)
N1—C2—C1	107.3 (2)	O1—C16—C1	125.1 (2)
C4—C2—C1	110.5 (2)	O2—C16—C1	110.5 (2)

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