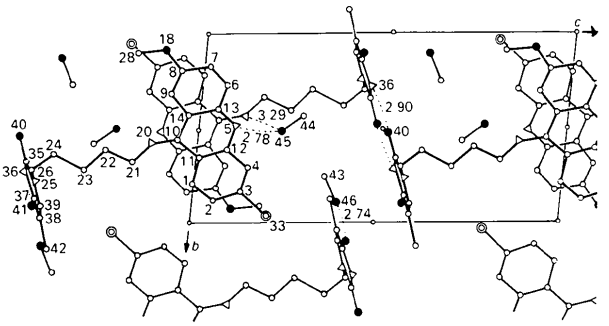

Fig. 3. Projection de la structure le long de **a**.

Une molécule de méthanol contribue à accroître la cohésion d'une pile en participant à deux liaisons hydrogène avec respectivement l'azote pyrimidique et l'azote amine de deux noyaux aminoacridines homologues par un centre de symétrie: O(45)–H[*x*,*y*,*z*]···N(5)[*x*,*y*,*z*] = 2,781 (5), N(20)–H(120)-(*x*,*y*,*z*)···O(45)[1−*x*, 1−*y*, −*z*] = 3,289 (5), H(120)-[*x*,*y*,*z*]···O(45)[1−*x*, 1−*y*, −*z*] = 2,34 (4) Å; N(20)–H(120)···O(45) = 157 (3)°.

L'autre molécule de méthanol est liée au noyau thymine par une seule liaison hydrogène: O(46)–H[*x*,*y*,*z*]···O(41)[−*x*,−2−*y*,−*z*] = 2,746 (7) Å.

On observe aussi une association par liaisons hydrogène de deux enchaînements thymine homologues par un centre de symétrie: N(36)[*x*,*y*,*z*]···O(40)[−1−*x*, 1−*y*, −1−*z*] = 2,895 (5), H(136)[*x*,*y*,*z*]···O(40)[−1−*x*, 1−*y*, −1−*z*] = 1,90 (4) Å; N(36)–H(136)···O(40) = 167 (4)° (Fig. 3).

En dehors de ces liaisons localisées assurant l'essentiel de la cohésion du cristal on observe quelques contacts de van der Waals: C(43)[*x*,*y*,*z*]···C(35)[1 + *x*, *y*, 1 + *z*] = 3,538 (8), C(2)[*x*,*y*,*z*]···C(7)[*x*, 1 + *y*, *z*] = 3,376 (7), O(46)[*x*,*y*,*z*]···C(39)[1−*x*, 2−*y*, −*z*] = 3,233 (7), C(10)[*x*,*y*,*z*]···C(7)[1−*x*, 1−*y*, −*z*] = 3,569 (6), C(12)[*x*,*y*,*z*]···C(10)[−*x*, 1−*y*, −*z*] = 3,514 (6), C(44)[*x*,*y*,*z*]···C(35)[−*x*, 1−*y*, −*z*] = 3,494 (8) Å.

Cette étude structurale confirme l'aptitude du noyau acridine à s'associer par liaisons de superposition à une distance interplanaire assez courte ($\bar{d} \simeq 3{,}55$ Å). Par contre on n'observe aucune superposition entre les noyaux acridine et thymine: en effet dans cette structure ces deux noyaux sont pratiquement perpendiculaires et correspondent à une structure en chevrons présentant quelques analogies avec le complexe acridine–cytosine (Shefter, 1968).

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Acta Cryst. (1983). C**39**, 248–250

Structure of 2,2-Dimethyl-4,6,6-triphenyl-1-thia-4-azaspiro[2.3]hexan-5-one, $C_{24}H_{21}NOS$

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(*Received* 1 *July* 1982; *accepted* 19 *August* 1982)

Abstract. $M_r = 371{\cdot}5$, triclinic, $P\bar{1}$, $a = 12{\cdot}524$ (8), $b = 10{\cdot}680$ (7), $c = 7{\cdot}690$ (6) Å, $\alpha = 89{\cdot}51$ (3), $\beta = 74{\cdot}73$ (3), $\gamma = 79{\cdot}99$ (3)°, $V = 976{\cdot}4$ Å^3, $Z = 2$, $D_c = 1{\cdot}26$ Mg m^{-3}, μ(Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å) = $1{\cdot}52$ mm^{-1}, $F(000) = 392$. The structure was solved by direct methods and refined to an R value of 0·054 for 2466

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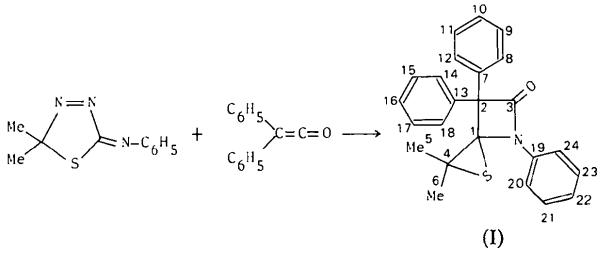
reflections. The β-lactam system is characterized by an almost planar four-membered ring, which contains a planar N atom. The phenyl group bonded to the N atom makes an angle of 34° relative to the β-lactam ring.

Introduction. Thiadiazolines have been applied in the synthesis of a variety of heterocyclic compounds. The title compound, (I), was synthesized through the reaction of 2,2-dimethyl-5-phenylimino-2,5-dihydro-1,3,4-thiadiazole with diphenylketene in excellent yield (Yamamoto, Abe, Nozawa, Motoyoshiya & Gotoh, 1981). The structure of the product (I) was based on spectral data and chemical properties. The present analysis was undertaken to reveal the conformation of the spiro β-lactam as well as to confirm the spectroscopic assignment.



(I)

Experimental. Crystal 0·2 × 0·3 × 0·3 mm, Ni-filtered Cu $K\alpha$ radiation, microcomputer-controlled four-circle diffractometer developed in our laboratory (Katsube, 1982); unit-cell dimensions from least-squares fit to observed values of $\pm\theta$ for 30 reflections; 2754 independent reflections, $2\theta_{max} = 120°$, θ–2θ scan technique; 2466 $I > 2\sigma(I)$ used for subsequent calculations; periodically monitored reflections showed no significant change in intensity; Lp corrections, no absorption correction; direct method (Germain, Main & Woolfson, 1971) using 220 reflections; E map calculated from phase set with highest combined figure of merit revealed all non-hydrogen atoms, although peak heights of some atoms were lower than that of highest noise; positional and anisotropic temperature factors for C, N, O and S refined by block-diagonal least-squares method (Ashida, 1973); H atoms from difference map, included in refinement with isotropic temperature factors; final $R = 0{\cdot}054$ for 2466 reflections; function minimized $\sum w|F_o - |F_c||^2$; $w = 1{\cdot}0$ for $F_o \le 20$, $w = [1{\cdot}0 + 0{\cdot}2(F_o - 20)]^{-1}$ for $F_o > 20$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are in Table 1.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38116 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. A stereoscopic view of the molecule is shown in Fig. 1. Bond lengths and angles for the non-hydrogen atoms are listed in Table 2. The β-lactam system is characterized by an almost planar four-membered ring. The geometry around the N atom is roughly planar; the displacement of the N atom from the plane through C(1), C(3) and C(9) is 0·05 Å. However, the phenyl group bonded to the N atom makes an angle of 34° with the β-lactam ring.

Several structures containing thiirane rings have been determined (Cunningham, Boyd, Myers, Gwinn & Le Van, 1951; Bates, Grady & Sneath, 1972; Utsumi-Oda & Koyama, 1973; Mugnoli & Simonetta, 1976; Wong-Ng & Nyburg, 1978). The C—S length seems to be reflected by the environment of the ring and shows a

Table 1. *Atomic coordinates and temperature factors* (Å²)

$B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} or B_{iso}
S	0·7757 (1)	0·7406 (1)	0·2969 (1)	4·7
O	0·5468 (2)	0·7847 (2)	0·9184 (3)	4·9
N	0·5859 (2)	0·7503 (2)	0·6044 (3)	3·6
C(1)	0·6998 (2)	0·6881 (3)	0·5104 (4)	3·6
C(2)	0·7303 (2)	0·6840 (3)	0·6974 (4)	3·5
C(3)	0·6062 (2)	0·7491 (3)	0·7715 (4)	3·7
C(4)	0·7270 (3)	0·5879 (3)	0·3644 (4)	4·3
C(5)	0·6343 (3)	0·5549 (3)	0·2909 (5)	5·1
C(6)	0·8233 (3)	0·4775 (4)	0·3565 (5)	5·4
C(7)	0·7516 (2)	0·5495 (3)	0·7655 (4)	3·6
C(8)	0·8517 (3)	0·4989 (3)	0·8041 (4)	4·5
C(9)	0·8702 (3)	0·3719 (3)	0·8575 (5)	5·8
C(10)	0·7874 (4)	0·2991 (3)	0·8718 (5)	6·4
C(11)	0·6857 (4)	0·3504 (3)	0·8402 (5)	6·1
C(12)	0·6676 (3)	0·4757 (3)	0·7857 (5)	4·8
C(13)	0·8131 (2)	0·7669 (3)	0·7197 (4)	3·6
C(14)	0·7855 (3)	0·8497 (3)	0·8698 (4)	4·1
C(15)	0·8613 (3)	0·9261 (3)	0·8917 (5)	5·3
C(16)	0·9631 (3)	0·9202 (3)	0·7673 (6)	5·7
C(17)	0·9924 (3)	0·8361 (3)	0·6191 (5)	5·3
C(18)	0·9180 (3)	0·7591 (3)	0·5962 (5)	4·4
C(19)	0·4868 (2)	0·8079 (3)	0·5555 (4)	3·6
C(20)	0·3840 (3)	0·8056 (3)	0·6753 (5)	4·6
C(21)	0·2862 (3)	0·8639 (4)	0·6350 (5)	5·5
C(22)	0·2917 (3)	0·9241 (3)	0·4705 (5)	5·2
C(23)	0·3940 (3)	0·9234 (3)	0·3520 (5)	4·9
C(24)	0·4931 (3)	0·8667 (3)	0·3903 (4)	4·4
H(1)	0·575 (3)	0·636 (3)	0·283 (5)	4·2
H(2)	0·660 (3)	0·511 (3)	0·175 (5)	4·3
H(3)	0·587 (3)	0·502 (4)	0·379 (5)	5·3
H(4)	0·884 (3)	0·504 (3)	0·411 (5)	3·7
H(5)	0·793 (3)	0·411 (4)	0·422 (5)	4·5
H(6)	0·864 (3)	0·450 (3)	0·224 (5)	4·3
H(7)	0·916 (3)	0·555 (3)	0·793 (5)	3·5
H(8)	0·950 (3)	0·335 (3)	0·882 (5)	4·4
H(9)	0·804 (3)	0·205 (4)	0·905 (5)	5·6
H(10)	0·621 (3)	0·298 (4)	0·856 (6)	5·9
H(11)	0·586 (3)	0·515 (3)	0·762 (5)	3·8
H(12)	0·707 (3)	0·856 (3)	0·965 (4)	3·0
H(13)	0·838 (3)	0·988 (3)	1·006 (5)	4·3
H(14)	1·019 (3)	0·979 (3)	0·784 (5)	4·3
H(15)	1·071 (3)	0·830 (3)	0·521 (5)	4·1
H(16)	0·941 (3)	0·696 (4)	0·485 (5)	4·7
H(17)	0·380 (3)	0·759 (4)	0·799 (5)	4·7
H(18)	0·208 (3)	0·863 (4)	0·729 (5)	4·7
H(19)	0·217 (3)	0·969 (3)	0·440 (5)	3·5
H(20)	0·399 (3)	0·966 (3)	0·226 (5)	4·1
H(21)	0·573 (3)	0·867 (3)	0·299 (4)	3·0

broad range, 1·78 to 1·91 Å. The lengths of the two C–S bonds in the present crystal, 1·806 (3) and 1·862 (3) Å, lie in this range. There is no abnormally short intermolecular distance in this crystal.

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

S–C(1)	1·806 (3)	S–C(4)	1·862 (3)
O–C(3)	1·200 (4)	N–C(1)	1·461 (4)
N–C(3)	1·374 (4)	N–C(19)	1·427 (4)
C(1)–C(2)	1·581 (4)	C(1)–C(4)	1·488 (4)
C(2)–C(3)	1·545 (4)	C(2)–C(7)	1·529 (4)
C(2)–C(13)	1·518 (4)	C(4)–C(5)	1·511 (5)
C(4)–C(6)	1·522 (5)	C(7)–C(8)	1·382 (5)
C(7)–C(12)	1·397 (5)	C(8)–C(9)	1·412 (5)
C(9)–C(10)	1·382 (6)	C(10)–C(11)	1·378 (7)
C(11)–C(12)	1·397 (6)	C(13)–C(14)	1·391 (5)
C(13)–C(18)	1·393 (5)	C(14)–C(15)	1·397 (5)
C(15)–C(16)	1·369 (6)	C(16)–C(17)	1·387 (6)
C(17)–C(18)	1·390 (5)	C(19)–C(20)	1·376 (5)
C(19)–C(24)	1·402 (5)	C(20)–C(21)	1·384 (5)
C(21)–C(22)	1·405 (6)	C(22)–C(23)	1·362 (6)
C(23)–C(24)	1·386 (5)		
C(1)–S–C(4)	47·8 (1)	C(1)–N–C(3)	94·7 (2)
C(1)–N–C(19)	136·7 (3)	C(3)–N–C(19)	128·3 (3)
S–C(1)–N	122·2 (2)	S–C(1)–C(2)	128·8 (2)
S–C(1)–C(4)	68·1 (2)	N–C(1)–C(2)	88·5 (2)
N–C(1)–C(4)	124·5 (3)	C(2)–C(1)–C(4)	129·9 (3)
C(1)–C(2)–C(3)	83·7 (2)	C(1)–C(2)–C(7)	113·3 (2)
C(1)–C(2)–C(13)	116·6 (2)	C(3)–C(2)–C(7)	111·7 (2)
C(3)–C(2)–C(13)	113·1 (2)	C(7)–C(2)–C(13)	114·6 (3)
O–C(3)–N	132·0 (3)	O–C(3)–C(2)	134·8 (3)
N–C(3)–C(2)	93·2 (2)	S–C(4)–C(1)	64·1 (2)
S–C(4)–C(5)	117·4 (2)	S–C(4)–C(6)	113·0 (2)
C(1)–C(4)–C(5)	119·7 (3)	C(1)–C(4)–C(6)	119·0 (3)
C(5)–C(4)–C(6)	114·2 (3)	C(2)–C(7)–C(8)	121·9 (3)
C(2)–C(7)–C(12)	118·5 (3)	C(8)–C(7)–C(12)	119·6 (3)
C(7)–C(8)–C(9)	120·0 (3)	C(8)–C(9)–C(10)	119·6 (4)
C(9)–C(10)–C(11)	120·7 (4)	C(10)–C(11)–C(12)	119·8 (4)
C(7)–C(12)–C(11)	120·2 (4)	C(2)–C(13)–C(14)	119·5 (3)
C(2)–C(13)–C(18)	121·5 (3)	C(14)–C(13)–C(18)	119·0 (3)
C(13)–C(14)–C(15)	119·9 (3)	C(14)–C(15)–C(16)	120·8 (4)
C(15)–C(16)–C(17)	119·8 (4)	C(16)–C(17)–C(18)	120·0 (4)
C(13)–C(18)–C(17)	120·5 (3)	N–C(19)–C(20)	118·4 (3)
N–C(19)–C(24)	121·2 (3)	C(20)–C(19)–C(24)	120·3 (3)
C(19)–C(20)–C(21)	120·1 (3)	C(20)–C(21)–C(22)	120·0 (4)
C(21)–C(22)–C(23)	119·1 (4)	C(22)–C(23)–C(24)	121·8 (4)
C(19)–C(24)–C(23)	118·6 (3)		

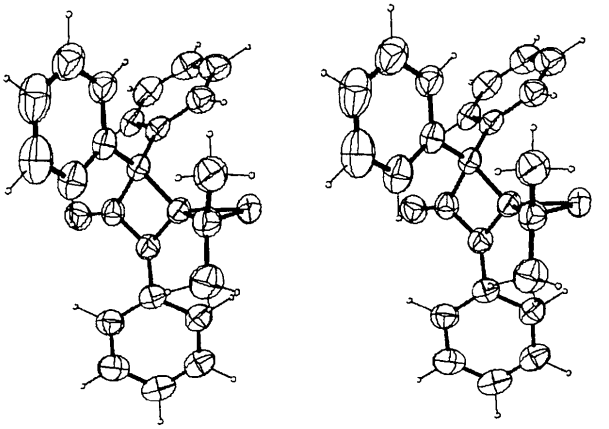


Fig. 1. A stereoscopic view of the molecule.

Computations were carried out on a HITAC M-150 computer at the Tottori University Computing Center. This work was supported by a grant (No. 57550502) from the Ministry of Education of Japan.

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Acta Cryst. (1983). C**39**, 250–253

Tetrafluorophthalic Acid (TFAC), $C_8H_2F_4O_4$

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(*Received* 4 *August* 1982; *accepted* 11 *October* 1982)

Abstract. $M_r = 238·1$, monoclinic, $P2_1/n$, $a = 11·450$ (3), $b = 5·559$ (1), $c = 13·775$ (2) Å, $\beta = 107·52$ (1)°, $V = 836·1$ Å^3, $Z = 4$, $D_x = 1·89$, $D_m = 1·82$ Mg m^{-3}, μ(Cu $K\alpha$) = 1·93 mm^{-1}, $T = 294$ K, Ni-

0108-2701/83/020250-04$01.50