C1—O2—HO2	110 (3)	C5—C10—C9	120.7 (2)
C11—O4—HO4	106 (3)	O3—C11—O4	123.8 (2)
O1—C1—O2	124.1 (2)	O3—C11—C12	120.7 (2)
O1—C1—C2	120.2 (2)	O4—C11—C12	115.5 (2)
O2—C1—C2	115.7 (2)	C11—C12—C13	116.6 (2)
C1—C2—C3	119.8 (2)	C11—C12—C14	117.1 (2)
C1—C2—C4	115.0 (2)	C13—C12—C14	126.3 (2)
C3—C2—C4	125.1 (2)	C12—C13—C15	133.1 (2)
C2—C3—C5	130.2 (2)	C12—C13—H13	114 (1)
C2—C3—H3	118 (2)	C15—C13—H13	113 (1)
C5—C3—H3	112 (2)	F4—C14—F5	105.2 (2)
F1—C4—F2	106.3 (2)	F4—C14—F6	106.2 (2)
F1—C4—F3	106.7 (2)	F4—C14—C12	112.1 (2)
F1—C4—C2	113.5 (2)	F5—C14—F6	106.8 (2)
F2—C4—F3	104.8 (2)	F5—C14—C12	112.8 (2)
F2—C4—C2	112.5 (2)	F6—C14—C12	113.2 (2)
F3—C4—C2	112.5 (2)	C13—C15—C16	123.5 (2)
C3—C5—C6	119.0 (2)	C13—C15—C20	118.0 (2)
C3—C5—C10	122.3 (2)	C16—C15—C20	118.2 (2)
C6—C5—C10	118.7 (2)	C15—C16—C17	120.3 (2)
C5—C6—C7	120.2 (2)	C16—C17—C18	120.8 (3)
C6—C7—C8	120.1 (2)	C17—C18—C19	119.5 (2)
C7—C8—C9	120.6 (2)	C18—C19—C20	120.6 (2)
C8—C9—C10	119.7 (2)	C15—C20—C19	120.5 (2)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—HO2···O3	0.77 (5)	1.87 (5)	2.638 (2)	176 (4)
O4—HO4···O1	0.84 (4)	1.79 (4)	2.627 (2)	171 (4)

Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974)

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980) (direct methods). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX* in *MolEN*.

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Lists of structure factors, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SX1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(2-Thiazolylhydrazono)indan-1,3-dione

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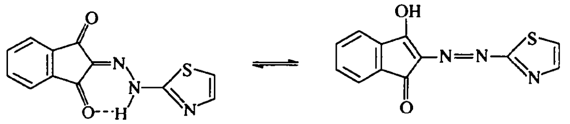
Abstract

The structural results clearly indicate that 2-(2-thiazolylhydrazono)indan-1,3-dione, $C_{12}H_7N_3O_2S$, exists as a keto–hydrazone tautomer in the solid state. The indandione and thiazolylhydrazone groups are connected by a C═N bond and the H atom bonded to N1 refines with a normal temperature factor. The entire molecule is essentially planar. There is a strong intramolecular hydrogen bond between the hydrazone H atom and a carbonyl O atom. The asymmetric unit contains two molecules which have a 'local' screw relationship.

Comment

Disperse monoazo dyes prepared from enol-type coupling components are known to exhibit azo–hydrazone tautomerism. Knowledge of the tautomeric form of the dyestuff present in the solid phase is primarily important for dye–fiber interaction. Several papers have been published concerning the relationships between the structure and various properties for a large number of monoazo disperse dyes (Desiraju, 1983; Guggenberger & Teufer, 1975; Connor, Kennedy, Dawes, Hursthouse & Walker, 1990; Bart, Calcaterra & Cavigiolo, 1985; McIntosh, Freeman & Singh, 1989). In a continuation of our work on the structural investigation of azo compounds, we introduce here a novel potential dyestuff, namely, 2-(2-thiazolylhydrazono)indan-1,3-dione, which is expected to exhibit azo–hydrazone tautomerism. The structure determination of the title compound, (I), was undertaken in order to establish whether the hydrazone or azo form is present in the solid phase.



(Ia) (Ib)

Our previous studies involving 2-phenylazo-1,3-indandione and 2-methyl-2-(*n*-substituted phenylazo)-1,3-indandione compounds have already been reported

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(İde, Kendi, Özbey & Ertan, 1994; İde, Kendi & Ertan, 1994; Özbey, Kendi, Hocaoğlu, Uyar & Mak, 1993; Özbey *et al.*, 1994). The crystal structures of two compounds in the thiazolylazopyridone series have also been studied (Temel, Özbey & Ertan, 1995; Özbey, Kendi & Ertan, 1995).

As shown in the scheme, there are two possible tautomeric forms of the title compound, *i.e.* the hydrazone form, (I*a*), and the azo form, (I*b*). The presence of two carbonyl bands in the IR spectrum suggests that this compound is in the hydrazone form in the solid phase. The structural results for the title compound support this finding. The location of the H atom on N1 rather than on O1 and the lengths of the N1—N2, N2—C2 and C3—O1 bonds indicate that the molecule exists as the keto–hydrazone form (I*a*) in the solid state. There are two molecules in the asymmetric unit (Fig. 1). The existence of a pseudo-screw axis, however, passing through a = 0.74, c = 0.25 and parallel to the b axis shows that there is a 'local' screw relationship between the two molecules. The N2—C2*a* and C3*a*—O1 bond distances of 1.300 (4) and 1.220 (4) Å, respectively, for molecule *A* [1.299 (4) and 1.224 (4) Å, respectively, for molecule *B*] are consistent with the partial double-bond character expected in the hydrazone form (Pendergrass, Paul & Curtin, 1972). The bond lengths and angles agree with those in 2-phenylazo-1,3-indandione (İde, Kendi, Özbey & Ertan, 1994) and 1-butyl-3-cyano-4-methyl-5-(2-nitrothiazolylhydrazono)pyridine-2,6-dione (Özbey, Kendi & Ertan, 1995), which are also in the hydrazone form. The N1—N2 and N1—C2 bond distances suggest considerable conjugation in this part of the molecule. The N1—C2′ bond is a single bond with bond lengths of 1.384 (4) and 1.376 (4) Å in molecules *A* and *B*, respectively. This bond is 1.37 (1) Å in 1-butyl-3-cyano-4-methyl-5-(2-nitrothiazolylhydrazono)pyridine-2,6-dione (Özbey, Kendi & Ertan, 1995).

The short O1···H1 and N1···O1 distances of 2.14 (3) and 2.78 (3) Å, respectively, for molecule *A* [2.10 (4) and 2.76 (4) Å, respectively, for *B*] suggest the presence of a hydrogen bond. These values are comparable to those reported for similar related structures. The O1—C3*a*—C2*a*—N2—N1—H1 ring completed by the hydrogen bond is essentially planar, with a maximum deviation from the least-squares plane defined by all atoms of 0.021 (4) Å for H1 in molecule *A* [this value is 0.023 (3) Å for the C3*b* atom in molecule *B*].

The thiazole ring planes are very slightly twisted from that of the hydrazone group [C2*a*—N2—N1—C2′*a* −178.0 (3) and C2*b*—N2′—N1′—C2′*b* −173.5 (3)° for molecules *A* and *B*, respectively] and the indandione groups as a whole are planar. In contrast, 2-methyl-2-(*n*-substituted phenylazo)-1,3-indandione compounds have *trans* geometry about the azo linkage and the five-membered rings are in an envelope conformation.

All intermolecular contacts were examined. The minimum intermolecular distances conform to normal van der Waals interactions. The molecules are disposed on top of one another in an antiparallel fashion with a displacement which avoids superposition of the thiazole rings (Fig. 2).

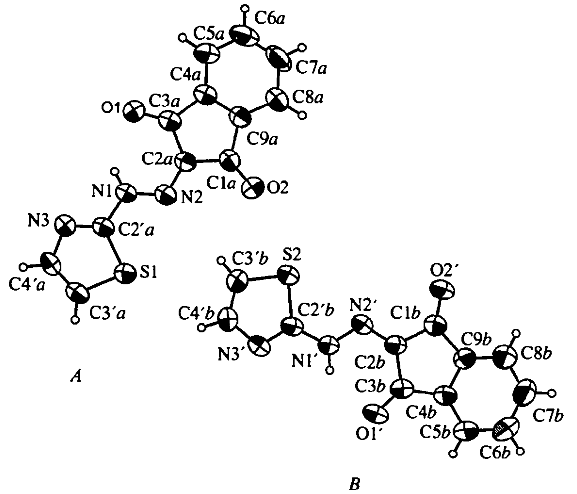

Fig. 1. *ORTEP* [(Johnson, 1965) in *MolEN* (Fair, 1990)] drawing of the two molecules of $C_{12}H_7N_3O_2S$ with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.

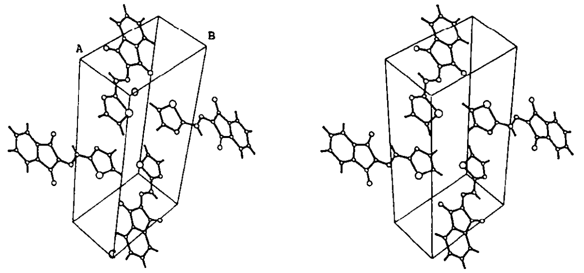

Fig. 2. A stereoscopic packing diagram of the molecules viewed along the c axis.

Experimental

The title compound was prepared by coupling 1,3-indandione with diazotized 2-aminothiazole in nitrosylsulfuric acid and recrystallized from an ethanol–dimethylformamide mixture. Details of the synthetic work will be published elsewhere (Ertan & Özgün, 1996).

Crystal data

$C_{12}H_7N_3O_2S$
M_r = 257.27
Mo $K\alpha$ radiation
λ = 0.71073 Å

Triclinic
$P\overline{1}$
$a = 5.377\,(1)$ Å
$b = 10.488\,(1)$ Å
$c = 20.320\,(2)$ Å
$\alpha = 77.41\,(1)°$
$\beta = 86.14\,(1)°$
$\gamma = 80.28\,(1)°$
$V = 1101.7\,(3)$ Å^3
$Z = 4$
$D_x = 1.551$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 8–18°$
$\mu = 0.29$ mm^{-1}
$T = 295$ K
Needle
0.64 × 0.24 × 0.12 mm
Light brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
$T_{\min} = 0.889$, $T_{\max} = 0.999$
4486 measured reflections
3561 independent reflections

2547 observed reflections $[I > 3\sigma(I)]$
$R_{\text{int}} = 0.023$
$\theta_{\max} = 26.3°$
$h = 0 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -24 \rightarrow 24$
3 standard reflections
frequency: 120 min
intensity decay: 2.7%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.041$
$S = 0.93$
2547 reflections
381 parameters
All H atoms were found from a difference map and refined isotropically

$w = 1/[\sigma(F)^2]$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{\text{eq}} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
S1	0.3339 (2)	0.24808 (8)	0.22587 (5)	3.03 (2)
O1	1.1388 (4)	0.4029 (2)	0.0659 (1)	3.61 (5)
O2	0.6268 (5)	0.7035 (2)	0.1806 (1)	4.03 (6)
N1	0.7446 (5)	0.3117 (3)	0.1479 (2)	3.09 (6)
N2	0.6906 (5)	0.4310 (3)	0.1620 (2)	2.91 (6)
N3	0.6406 (5)	0.1015 (3)	0.1598 (2)	3.52 (7)
C1*a*	0.7898 (6)	0.6545 (3)	0.1455 (2)	3.07 (8)
C2*a*	0.8338 (6)	0.5175 (3)	0.1342 (2)	2.79 (7)
C2′*a*	0.5939 (6)	0.2169 (3)	0.1744 (2)	2.71 (7)
C3*a*	1.0522 (6)	0.5023 (3)	0.0868 (2)	2.82 (7)
C3′*a*	0.2808 (7)	0.0894 (3)	0.2279 (2)	3.44 (8)
C4*a*	1.1372 (6)	0.6339 (3)	0.0690 (2)	2.85 (7)
C4′*a*	0.4582 (7)	0.0286 (3)	0.1916 (2)	3.65 (8)
C5*a*	1.3328 (7)	0.6726 (3)	0.0254 (2)	3.48 (8)
C6*a*	1.3730 (7)	0.8015 (4)	0.0174 (2)	4.17 (9)
C7*a*	1.2246 (8)	0.8887 (4)	0.0520 (2)	4.31 (9)
C8*a*	1.0284 (7)	0.8503 (3)	0.0955 (2)	3.72 (8)
C9*a*	0.9885 (6)	0.7212 (3)	0.1032 (2)	2.83 (7)
S2	0.1533 (2)	0.74352 (8)	0.27991 (5)	3.61 (2)
O1′	−0.6897 (4)	0.9175 (2)	0.4213 (1)	3.79 (6)
O2′	−0.1153 (4)	1.2090 (2)	0.3304 (1)	3.84 (6)
N1′	−0.2786 (5)	0.8224 (3)	0.3472 (2)	3.12 (6)
N2′	−0.2078 (5)	0.9395 (3)	0.3386 (1)	2.80 (6)
N3′	−0.1802 (5)	0.6025 (3)	0.3369 (2)	3.51 (7)
C1*b*	−0.2878 (6)	1.1612 (3)	0.3626 (2)	2.79 (7)
C2*b*	−0.3504 (6)	1.0274 (3)	0.3665 (2)	2.65 (7)
C2′*b*	−0.1245 (6)	0.7205 (3)	0.3250 (2)	2.84 (7)
C3*b*	−0.5817 (6)	1.0143 (3)	0.4086 (2)	2.74 (7)
C3′*b*	0.1971 (7)	0.5789 (3)	0.2763 (2)	3.78 (8)
C4*b*	−0.6499 (6)	1.1390 (3)	0.4335 (2)	2.77 (7)
C4′*b*	0.0058 (7)	0.5221 (3)	0.3083 (2)	3.75 (8)
C5*b*	−0.8444 (6)	1.1744 (3)	0.4776 (2)	3.34 (8)
C6*b*	−0.8639 (7)	1.2953 (4)	0.4942 (2)	3.88 (9)
C7*b*	−0.6937 (8)	1.3804 (4)	0.4678 (2)	4.16 (9)
C8*b*	−0.5005 (7)	1.3467 (3)	0.4235 (2)	3.68 (8)
C9*b*	−0.4794 (6)	1.2249 (3)	0.4068 (2)	2.68 (7)

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

S1—C2′*a*	1.718 (3)	S2—C2′*b*	1.724 (3)
S1—C3′*a*	1.726 (4)	S2—C3′*b*	1.720 (4)
O1—C3*a*	1.220 (4)	O1′—C3*b*	1.224 (4)
O2—C1*a*	1.209 (4)	O2′—C1*b*	1.214 (4)
N1—N2	1.323 (4)	N1′—N2′	1.319 (4)
N1—C2′*a*	1.384 (4)	N1′—C2′*b*	1.376 (4)
N2—C2*a*	1.300 (4)	N2′—C2*b*	1.299 (4)
N3—C2′*a*	1.289 (4)	N3′—C2′*b*	1.290 (4)
N3—C4′*a*	1.385 (5)	N3′—C4′*b*	1.379 (5)
C1*a*—C2*a*	1.482 (5)	C1*b*—C2*b*	1.482 (5)
C1*a*—C9*a*	1.496 (5)	C1*b*—C9*b*	1.489 (5)
C2*a*—C3*a*	1.477 (5)	C2*b*—C3*b*	1.470 (4)
C3′*a*—C4′*a*	1.329 (5)	C3*b*—C4*b*	1.485 (5)
C3*a*—C4*a*	1.489 (5)	C3′*b*—C4′*b*	1.335 (5)
C4*a*—C9*a*	1.384 (5)	C4*b*—C9*b*	1.394 (5)
C2′*a*—S1—C3′*a*	87.6 (2)	C2′*b*—S2—C3′*b*	88.0 (2)
N2—N1—C2′*a*	120.1 (3)	N2′—N1′—C2′*b*	120.3 (3)
N1—N2—C2*a*	117.6 (3)	N1′—N2′—C2*b*	117.1 (3)
C2′*a*—N3—C4′*a*	108.4 (3)	C2′*b*—N3′—C4′*b*	108.9 (3)
O2—C1*a*—C2*a*	127.9 (3)	O2′—C1*b*—C2*b*	127.2 (3)
O2—C1*a*—C9*a*	126.9 (3)	O2′—C1*b*—C9*b*	127.0 (3)
C2*a*—C1*a*—C9*a*	105.2 (3)	C2*b*—C1*b*—C9*b*	105.8 (3)
N2—C2*a*—C1*a*	122.4 (3)	N2′—C2*b*—C1*b*	122.5 (3)
N2—C2*a*—C3*a*	128.6 (3)	N2′—C2*b*—C3*b*	128.9 (3)
C1*a*—C2*a*—C3*a*	109.0 (3)	C1*b*—C2*b*—C3*b*	108.5 (3)
S1—C2′*a*—N1	122.1 (2)	S2—C2′*b*—N1′	121.8 (2)
S1—C2′*a*—N3	117.1 (3)	S2—C2′*b*—N3′	116.4 (3)
N1—C2′*a*—N3	120.7 (3)	N1′—C2′*b*—N3′	121.8 (3)
S1—C3′*a*—C4′*a*	110.4 (3)	S2—C3′*b*—C4′*b*	110.2 (3)
O1—C3*a*—C2*a*	126.5 (3)	O1′—C3*b*—C2*b*	126.0 (3)
O1—C3*a*—C4*a*	127.9 (3)	O1′—C3*b*—C4*b*	127.9 (3)
C2*a*—C3*a*—C4*a*	105.6 (3)	C2*b*—C3*b*—C4*b*	106.2 (3)
N3—C4′*a*—C3′*a*	116.4 (3)	N3′—C4′*b*—C3′*b*	116.4 (3)
C3*a*—C4*a*—C9*a*	110.1 (3)	C3*b*—C4*b*—C9*b*	109.9 (3)
C1*a*—C9*a*—C4*a*	110.1 (3)	C1*b*—C9*b*—C4*b*	109.5 (3)

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1992). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP* (Johnson, 1965) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1205). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(5*S*)-3-Phenyl-5-[(6*R*,7*R*,14*R*)-1,8,13,16-tetraoxadispiro[5.0.5.4]hexadecan-14-yl]-4,5-dihydroisoxazole: the Major Adduct Resulting from Cycloaddition of Benzonitrile Oxide to (6*R*,7*R*,14*S*)-14-Vinyl-1,8,13,16-tetraoxadispiro[5.0.5.4]hexadecane

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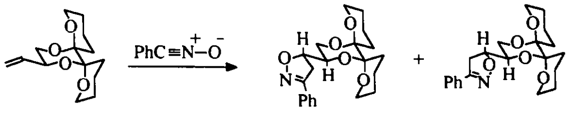
Abstract

This structure determination of $C_{21}H_{27}NO_5$, taken with the known absolute configuration of the starting alkene, establishes the configuration of the new stereogenic C5 centre in the title compound. The five-membered 4,5-dihydroisoxazole ring adopts a predominantly envelope conformation folded between the O1 and C4 atoms, and the two pyranoid rings and the 1,4-dioxane ring of the dispiroketal unit are in chair conformations.

Comment

As part of an investigation into the factors influencing stereoselectivity in nitrile oxide cycloadditions to chiral allyl ethers, the reaction of benzonitrile oxide ($PhC{\equiv}N^{+}{-}O^{-}$) with (6*R*,7*R*,14*S*)-14-vinyl-1,8,13,16-tetraoxadispiro[5.0.5.4]hexadecane, (1), has been examined (Gravestock, Paton & Todd, 1995). Two 4,5-dihydroisoxazole cycloadducts, (2) (52%) and (3) (16%), were isolated from the reaction mixture and the former provided crystals suitable for X-ray crystallography. Taken with the known absolute configuration of the starting alkene (1) (Ley, Woods & Zanotti-Gerosa, 1992), the present structure determination firmly establishes that the newly created stereogenic C5 centre in adduct (2) has an *S* configuration and that the minor isomer, (3), therefore has a 5*R* structure.



(1) (2) (3)

The Cremer & Pople (1975) puckering parameters for the four rings are given in Table 3. For the five-membered dihydroisoxazole ring, φ = 319.3 (5)°, which corresponds to a mainly envelope conformation *E*5 (φ = 324°), with the fold between the O1 and C4 atoms; the torsion angle for the O1—N2═C3—C4 unit is −2.6 (3)° and the C5 atom lies 0.406 (4) Å out of the best plane through these atoms. The θ values for the 1,4-dioxane ring and the two pyranoid rings indicate that the conformations of all three are very close to the ideal chair conformation (θ = 180°). No anomalies are found in the bond lengths and angles.

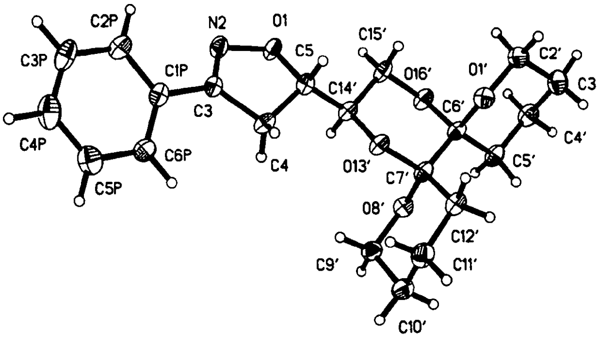


Fig. 1. A view of the title molecule, including the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was obtained by cycloaddition of benzonitrile oxide to (6*R*,7*R*,14*S*)-14-vinyl-1,8,13,16-tetraoxadispiro-[5.0.5.4]hexadecane as reported elsewhere (Gravestock, Paton & Todd, 1995).