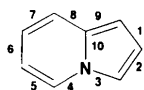
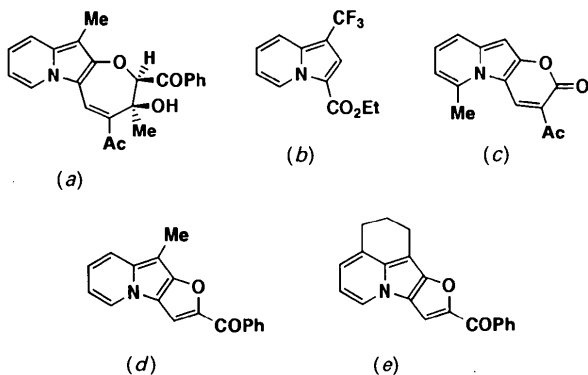


C2—C1—C13	116.0 (2)	C9—C10—C11	133.6 (2)
O3—C2—C1	101.0 (2)	C10—C11—C12	106.3 (2)
O3—C2—C3	111.9 (2)	C10—C11—C23	126.7 (3)
O3—C2—C20	109.4 (2)	C12—C11—C23	126.9 (3)
C1—C2—C3	117.1 (2)	O1—C12—C5	126.9 (2)
C1—C2—C20	108.8 (2)	O1—C12—C11	121.7 (2)
C2—C3—C4	122.3 (2)	C5—C12—C11	111.4 (2)
C2—C3—C21	118.1 (2)	O2—C13—C1	121.5 (2)
C4—C3—C21	119.1 (2)	O2—C13—C14	120.5 (2)
C3—C4—C5	128.7 (2)	C1—C13—C14	118.0 (2)
N1—C5—C4	122.2 (2)	O4—C21—C3	121.7 (3)
N1—C5—C12	104.3 (2)	O4—C21—C22	116.3 (3)
C4—C5—C12	133.4 (2)	C3—C21—C22	122.0 (3)

Table 3. Comparison of indolizine ring geometry



Compound	Fused ring	Bond length (Å)				
		1	2	3	4	5
(a)	7	1.378 (3)	1.409 (3)	1.406 (3)	1.379 (3)	1.346 (3)
(b)	None	1.382 (5)	1.370 (5)	1.407 (5)	1.372 (5)	1.362 (6)
(c)	6	1.368 (3)	1.411 (3)	1.403 (2)	1.386 (3)	1.367 (3)
(d)	5	1.388 (5)	1.373 (5)	1.379 (5)	1.381 (5)	1.335 (6)
(e)	5	1.394 (3)	1.384 (3)	1.379 (3)	1.377 (3)	1.350 (4)
		6	7	8	9	10
(a)	7	1.406 (4)	1.356 (4)	1.413 (4)	1.392 (3)	1.391 (3)
(b)	None	1.398 (6)	1.344 (5)	1.418 (5)	1.392 (5)	1.385 (5)
(c)	6	1.398 (4)	1.358 (4)	1.400 (3)	1.400 (3)	1.406 (3)
(d)	5	1.421 (6)	1.356 (6)	1.402 (6)	1.408 (5)	1.436 (5)
(e)	5	1.414 (4)	1.357 (4)	1.410 (3)	1.389 (3)	1.402 (3)



(a) 4-Acetyl-2-benzoyl-3-hydroxy-3,11-dimethyl-2,3-dihydrooxepino[2,3-*b*]indolizine (this work); (b) ethyl 1-trifluoromethylindolizine-3-carboxylate (Pritcher, 1988); (c) 3-acetyl-6-methyl-2*H*-pyrano[2,3-*b*]indolizine-2-one (Kakehi *et al.*, 1993); (d) 2-benzoyl-9-methylfuro[2,3-*b*]indolizine (Kakehi *et al.*, 1994); (e) 8-benzoyl-5,6-dihydro-4*H*-furo[2',3':4,5]pyrrolo[3,2,1-*ij*]quinoline (Kakehi *et al.*, 1994).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71449 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1052]

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*Acta Cryst.* (1994). **C50**, 141–144

## Structure of [1 $\alpha$ (*E*),5 $\alpha$ ,6 $\beta$ ,7 $\alpha$ ]-4-Methyl-7-(3-methyl-4-nitro-5-isoxazolyl)-5-nitro-6-phenyl-1-styryl-2-oxa-3-azabicyclo-[3.2.0]hept-3-ene

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## Abstract

The title compound, C<sub>24</sub>H<sub>20</sub>N<sub>4</sub>O<sub>6</sub>, is a dimer obtained by irradiation of 3-methyl-4-nitro-5-styrylisoxazole. The relative configuration of the stereocentres and the conformation of the 2-oxa-3-azabicycloheptene ring is established.

## Comment

During a study on the photoreactivity of 3-methyl-4-nitro-5-styrylisoxazole, as well as the expected photodimers deriving from 2 + 2 cycloaddition of two styryl moieties, the title dimer, obtained from 2 + 2 photocycloaddition of the double bond of the styryl moiety on the 4,5 positions of the isoxazole ring, was observed (Donati, Fiorenza & Sarti-Fantoni, 1979). To date, this case is the unique example of such unexpected photocycloaddition of isoxazoles. The latter photoreaction is highly stereo-

specific; in fact only one of the possible stereoisomers was found. This X-ray structure determination, as well as giving definite stereochemical information on the title compound, allowed us to obtain valuable data on the extent of conjugation of the 4-nitro group in 3-methylisoxazoles, whose reactivity is strongly influenced by the presence of the nitro group in the 4 position (Nesi, Giomi, Papaleo, Bracci & Dapporto, 1989; Nesi, Giomi, Papaleo & Corti, 1990; Sarti-Fantoni, Donati, Fiorenza, Moschi & Dal Piaz, 1980). Three X-ray structure determinations (Belicchi Ferrari, Gasparri Fava & Pellinghelli, 1973*a,b*; Bonamartini Corradi & Grasselli Palmieri, 1973) of the single 2-oxa-3-azabicyclo[3.2.0]hept-3-ene ring system have been reported previously.

The final atomic coordinates of the title compound are reported in Table 1 and bonds and angles in Table 2. As shown in Fig. 1, the styryl, isoxazolyl and nitro groups are *cis* to each other and *trans* with respect to the phenyl moiety. This configuration allows the phenyl ring to face the methyl group of the isoxazoline moiety; the line passing through the methyl C(31) and the centre of the ring makes an angle of 26.4 (2)° with the normal to the plane of the ring, so that the methyl group lies in the shielded region of the diamagnetic anisotropy. This datum is in agreement with the low value ( $\delta$  1.5) of chemical shift of the methyl protons observed in the NMR spectrum (Donati, Fiorenza & Sarti-Fantoni, 1979).

The isoxazoline has an envelope conformation with C(7) at the apex. The distance of C(7) from the plane of the other four atoms is 0.241 (6) Å, the dihedral angle between C(5)—C(6)—C(7) and C(7)—C(4)—C(5) in the cyclobutane ring is 150.8 (3)°; these

values agree with those reported for the dibromo-derivative of a similar oxazabicycloheptene (Belicchi Ferrari, Gasparri Fava & Pellinghelli, 1973*a*) and differ significantly from those of the corresponding dichloro derivative (Bonamartini Corradi & Grasselli Palmieri, 1973).

The plane of the nitro group of the isoxazole ring makes a dihedral angle of 17.6 (2)° with the plane of isoxazole; the N(32)—C(30) bond distance is 1.425 (4) Å. This value, which is shorter than those reported for substituted nitrobenzenes ( $\approx$  1.47 Å) (Domenicano, Shultz, Hargittai, Colapietro, Portalone, George & Boch, 1990), suggests a conjugation of the nitro group with the isoxazole ring. The deviation from coplanarity is mainly due to steric hindrance of H(6) on the cyclobutane ring [O(34)—H(6) = 2.33 (2) Å], while a smaller influence results from the methyl group [O(33)—H(313) = 2.61 (2) Å]. Previously reported data on the X-ray structure of 3-phenyl-4-nitro-5-methoxycarbonylisoxazole (Bautz, Blom, Boeyens & Michael, 1982) showed that the nitro group is forced to be perpendicular to the isoxazole ring by the steric hindrance of substituents at the 3 and 5 positions.

The isoxazole and the styryl planes in the title compound make dihedral angles of 97.2 (2) and 95.1 (2)°, respectively, with the mean-squares plane of the cyclobutane ring; the phenyl ring plane lies at 56.3 (2)° to this plane. Crystal packing is mainly due to van der Waals forces.

## Experimental

### Crystal data

C<sub>24</sub>H<sub>20</sub>N<sub>4</sub>O<sub>6</sub>  
*M<sub>r</sub>* = 460.445  
 Orthorhombic  
*Pbca*  
*a* = 20.838 (4) Å  
*b* = 19.880 (4) Å  
*c* = 10.861 (3) Å  
*V* = 4499 (1) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.36 Mg m<sup>-3</sup>

Cu K $\alpha$  radiation  
 $\lambda$  = 1.5418 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 12–17°  
 $\mu$  = 0.741 mm<sup>-1</sup>  
*T* = 293 K  
 Parallelepiped  
 0.7 × 0.3 × 0.1 mm  
 Colourless  
 Crystal source: ethanol solution

### Data collection

Philips PW100 diffractometer  
 $\omega/2\theta$  scans [width (0.8 + 0.1tan $\theta$ )°; rate 2.4° min<sup>-1</sup> in  $\theta$ ]  
 Absorption correction: none  
 2774 measured reflections  
 2774 independent reflections  
 1936 observed reflections  
 [*I* > 3 $\sigma$ (*I*)]

$\theta_{\max}$  = 55°  
*h* = 0 → 21  
*k* = 0 → 21  
*l* = 0 → 11  
 3 standard reflections monitored every 150 reflections  
 intensity variation: not significant

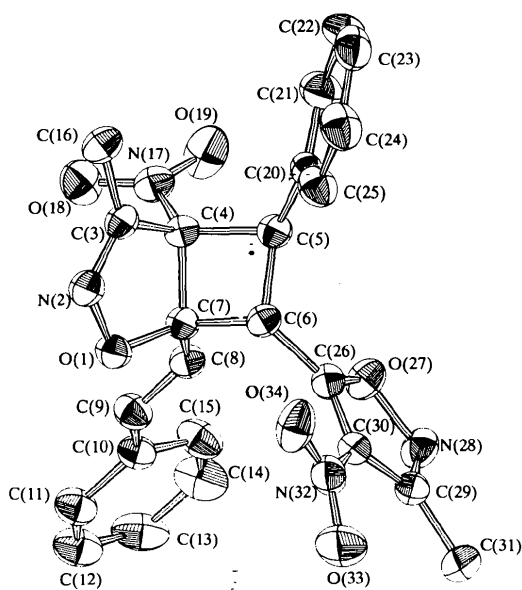


Fig. 1. ORTEP drawing (Johnson, 1965) of the asymmetric unit, with heavy atoms represented by 30% probability ellipsoids.

## Refinement

Refinement on  $F$  $R = 0.043$  $wR = 0.049$  $S = 0.088$ 

1936 reflections

368 parameters

Only coordinates of H atoms refined

$$w = [\sigma^2(F) + 0.002F^2]^{-1}$$

$$(\Delta/\sigma)_{\max} = 0.23$$

$$\Delta\rho_{\max} = 0.099 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.149 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Atomic scattering factors

from *SHELX76*

The structure was solved by direct methods using *SHELX76* (Sheldrick, 1976) and refined by full-matrix least squares with the same system of programs. The locations of the H atoms were obtained by difference Fourier maps. Non-H atoms were refined anisotropically, H atoms isotropically with a common temperature factor which refined freely to final  $U = 0.095$  (3)  $\text{\AA}^2$ .

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	$x$	$y$	$z$	$U_{\text{eq}}$
O(1)	0.1467 (1)	0.1429 (1)	0.2018 (2)	0.596 (7)
O(18)	0.0733 (1)	0.0097 (1)	0.0451 (2)	0.867 (10)
O(19)	0.0664 (2)	0.0526 (1)	-0.1355 (2)	1.090 (13)
O(27)	0.2043 (1)	0.2409 (1)	-0.1377 (2)	0.676 (8)
O(33)	0.2410 (2)	0.3902 (2)	0.1531 (2)	1.171 (14)
O(34)	0.1463 (2)	0.3480 (1)	0.1658 (2)	0.912 (12)
N(2)	0.0821 (1)	0.1401 (1)	0.2431 (2)	0.605 (9)
N(17)	0.0714 (1)	0.0571 (1)	-0.0245 (3)	0.649 (11)
N(28)	0.2559 (1)	0.2844 (2)	-0.1693 (3)	0.744 (11)
N(32)	0.1984 (2)	0.3539 (1)	0.1152 (3)	0.740 (12)
C(3)	0.0443 (1)	0.1296 (1)	0.1531 (3)	0.542 (10)
C(4)	0.0781 (1)	0.1260 (1)	0.0304 (2)	0.494 (6)
C(5)	0.0733 (1)	0.1875 (1)	-0.0559 (3)	0.537 (10)
C(6)	0.1271 (1)	0.2195 (1)	0.0228 (3)	0.518 (9)
C(7)	0.1457 (1)	0.1471 (1)	0.0707 (2)	0.507 (9)
C(8)	0.2029 (1)	0.1143 (2)	0.0153 (3)	0.554 (10)
C(9)	0.2469 (1)	0.0815 (2)	0.0757 (3)	0.611 (12)
C(10)	0.3046 (1)	0.0498 (2)	0.0245 (3)	0.604 (12)
C(11)	0.3487 (2)	0.0189 (2)	0.1019 (3)	0.710 (12)
C(12)	0.4040 (2)	-0.0099 (2)	0.0584 (5)	0.846 (17)
C(13)	0.4151 (2)	-0.0101 (2)	-0.0650 (5)	0.932 (19)
C(14)	0.3727 (2)	0.0195 (2)	-0.1447 (4)	0.964 (18)
C(15)	0.3177 (2)	0.0492 (2)	-0.1009 (4)	0.802 (15)
C(16)	-0.0259 (2)	0.1218 (2)	0.1712 (4)	0.686 (12)
C(20)	0.0091 (1)	0.2215 (2)	-0.0715 (3)	0.572 (11)
C(21)	-0.0360 (2)	0.1928 (2)	-0.1508 (3)	0.774 (14)
C(22)	-0.0970 (2)	0.2206 (3)	-0.1593 (4)	0.940 (19)
C(23)	-0.1141 (2)	0.2748 (3)	-0.0927 (4)	0.927 (17)
C(24)	-0.0701 (2)	0.3045 (2)	-0.0169 (4)	0.886 (17)
C(25)	-0.0084 (2)	0.2778 (2)	-0.0070 (3)	0.721 (13)
C(26)	0.1777 (1)	0.2618 (1)	-0.0323 (3)	0.521 (10)
C(29)	0.2587 (1)	0.3292 (2)	-0.0827 (3)	0.657 (12)
C(30)	0.2098 (1)	0.3164 (1)	0.0056 (3)	0.552 (10)
C(31)	0.3073 (2)	0.3833 (3)	-0.0881 (6)	1.007 (17)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

O(1)—N(2)	1.419 (3)	C(6)—C(26)	1.476 (4)
O(1)—C(7)	1.427 (3)	C(7)—C(8)	1.487 (4)
O(18)—N(17)	1.209 (3)	C(8)—C(9)	1.302 (4)
O(19)—N(17)	1.213 (3)	C(9)—C(10)	1.467 (4)
O(27)—N(28)	1.422 (4)	C(10)—C(11)	1.389 (4)
O(27)—C(26)	1.339 (3)	C(10)—C(15)	1.389 (4)
O(33)—N(32)	1.217 (4)	C(11)—C(12)	1.371 (4)
O(34)—N(32)	1.221 (4)	C(12)—C(13)	1.360 (6)

N(2)—C(3)	1.272 (4)	C(13)—C(14)	1.370 (6)
N(17)—C(4)	1.501 (4)	C(14)—C(15)	1.375 (4)
N(28)—C(29)	1.297 (4)	C(20)—C(21)	1.397 (4)
N(32)—C(30)	1.425 (4)	C(20)—C(22)	1.370 (4)
C(3)—C(4)	1.510 (4)	C(21)—C(22)	1.387 (6)
C(3)—C(16)	1.484 (4)	C(22)—C(23)	1.347 (7)
C(4)—C(5)	1.544 (4)	C(23)—C(24)	1.366 (6)
C(4)—C(7)	1.533 (4)	C(24)—C(25)	1.396 (4)
C(5)—C(6)	1.544 (4)	C(26)—C(30)	1.340 (4)
C(5)—C(20)	1.510 (4)	C(29)—C(30)	1.421 (4)
C(6)—C(7)	1.578 (4)	C(29)—C(31)	1.479 (6)
N(2)—O(1)—C(7)	107.7 (2)	C(4)—C(7)—C(8)	120.1 (2)
N(28)—O(27)—C(26)	109.3 (2)	C(6)—C(7)—C(8)	117.6 (2)
O(1)—N(2)—C(3)	110.6 (2)	C(7)—C(8)—C(9)	125.5 (3)
O(18)—N(17)—O(19)	124.6 (3)	C(8)—C(9)—C(10)	126.9 (3)
O(18)—N(17)—C(4)	117.4 (3)	C(9)—C(10)—C(11)	120.1 (3)
O(19)—N(17)—C(4)	118.0 (3)	C(9)—C(10)—C(15)	122.5 (3)
O(27)—N(28)—C(29)	106.0 (2)	C(11)—C(10)—C(15)	117.4 (3)
O(33)—N(32)—O(34)	123.6 (3)	C(10)—C(11)—C(12)	122.1 (4)
O(33)—N(32)—C(30)	118.1 (3)	C(11)—C(12)—C(13)	118.9 (4)
O(34)—N(32)—C(30)	118.3 (3)	C(12)—C(13)—C(14)	120.9 (4)
N(2)—C(3)—C(4)	113.3 (2)	C(13)—C(14)—C(15)	120.2 (4)
N(2)—C(3)—C(16)	121.7 (3)	C(10)—C(15)—C(14)	120.5 (4)
C(4)—C(3)—C(16)	124.9 (3)	C(5)—C(20)—C(21)	118.9 (3)
N(17)—C(4)—C(3)	110.5 (2)	C(5)—C(20)—C(25)	122.9 (3)
N(17)—C(4)—C(5)	118.4 (2)	C(21)—C(20)—C(25)	118.1 (3)
O(27)—C(4)—C(5)	118.0 (2)	C(20)—C(21)—C(22)	119.6 (4)
N(17)—C(4)—C(7)	116.7 (2)	C(21)—C(22)—C(23)	121.7 (4)
C(3)—C(4)—C(7)	99.4 (2)	C(22)—C(23)—C(24)	119.4 (4)
C(5)—C(4)—C(7)	90.9 (2)	C(23)—C(24)—C(25)	120.0 (4)
C(4)—C(5)—C(6)	86.7 (2)	C(20)—C(25)—C(24)	121.1 (4)
C(4)—C(5)—C(20)	118.7 (2)	O(27)—C(26)—C(6)	117.8 (3)
C(6)—C(5)—C(20)	121.4 (3)	O(27)—C(26)—C(30)	107.9 (2)
C(5)—C(6)—C(7)	89.2 (2)	C(6)—C(26)—C(30)	134.0 (3)
C(5)—C(6)—C(26)	121.9 (3)	N(28)—C(29)—C(30)	109.6 (3)
C(7)—C(6)—C(26)	118.5 (2)	N(28)—C(29)—C(31)	120.1 (4)
O(1)—C(7)—C(4)	106.4 (2)	C(30)—C(29)—C(31)	130.4 (4)
O(1)—C(7)—C(6)	112.7 (2)	N(32)—C(30)—C(26)	126.6 (3)
C(4)—C(7)—C(6)	85.9 (2)	N(32)—C(30)—C(29)	126.1 (3)
O(1)—C(7)—C(8)	111.5 (2)	C(26)—C(30)—C(29)	107.2 (3)

We thank the Istituto del CNR per lo Studio della Stereo-chimica ed Energetica dei Composti di Coordinazione of Florence for collection of intensity data.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and shortest intermolecular distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71415 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1037]

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## International Union of Crystallography

*Acta Cryst.* (1994). **C50**, 144

### Restructuring of the IUCr editorial office

Following the promotion of Mr Michael Dacombe from Technical Editor to Executive Secretary, it was considered an opportune time to restructure the editorial office to reflect better the greater variety of work now carried out and to clarify the responsibilities of the staff.

Mr Peter Strickland has been appointed as Managing Editor with overall responsibility for both the technical editing and the centralized checking. Mrs Sue King has been appointed as Technical Editor. Dr Amanda Berry has been appointed as Assistant Technical Editor with special responsibility for the centralized checking. There are three Senior Editorial Assistants and six Editorial Assistants. Mr Brian McMahon is the Research and Development Officer and his assistant is Dr M. Hoyland. The total number of graduate staff in the editorial office is 14.