C2—C2^{i}	1.535 (2)	C6—C6^{ii}	1.538 (3)
C3—O31	1.252 (2)	C7—O72	1.253 (2)
C3—O32	1.262 (2)	C7—O71	1.258 (2)
C4—O41	1.257 (2)	C8—O81	1.254 (2)
C4—O42	1.2607 (14)	C8—O82	1.254 (2)
C3—C1—C2	112.30 (9)	C7—C5—C6	111.67 (11)
C1—C2—C4	110.57 (10)	C8—C6—C6^{ii}	109.92 (13)
C1—C2—C2^{i}	110.72 (12)	C8—C6—C5	110.01 (10)
C4—C2—C2^{i}	109.69 (13)	C6^{ii}—C6—C5	111.83 (13)
O31—C3—O32	125.04 (12)	O72—C7—O71	122.99 (13)
O31—C3—C1	118.74 (11)	O72—C7—C5	119.58 (12)
O32—C3—C1	116.22 (12)	O71—C7—C5	117.43 (11)
O41—C4—O42	123.76 (12)	O81—C8—O82	123.89 (13)
O41—C4—C2	118.42 (10)	O81—C8—C6	117.80 (11)
O42—C4—C2	117.81 (11)	O82—C8—C6	118.29 (11)
O31—C3—C1—C2	117.12 (13)	O71—C7—C5—C6	55.0 (2)
C3—C1—C2—C2^{i}	177.27 (12)	C7—C5—C6—C6^{ii}	177.63 (12)
C3—C1—C2—C4	−60.95 (13)	C7—C5—C6—C8	55.19 (14)
C1—C2—C4—O41	−68.07 (14)	C5—C6—C8—O81	−121.02 (13)

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $-x, -y, 1-z$.

Using the area detector system, cell dimensions were refined from 250 reflections selected from two regions 90° apart and 5° wide at $\kappa = 0°$. For all compounds, the intensity standards were not measured by the area detector. Possible variations were checked by comparing intensities of common or symmetry-related reflections as they occurred during data collection. In this case, no variation was noted.

For all compounds, data collection: *MADNES* (Pflugrath & Messerschmidt, 1991); cell refinement: *MADNES*; data reduction: *MADNES*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *PLATON*92 (Spek, 1992*a*) and *PLUTON*92 (Spek, 1992*b*)

The authors thank EPSRC and Professor M. Hursthouse (University of Wales, Cardiff) for the data collections.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: LI1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,12-Dimesityl-6,8-dimethyl-5,10-dioxa-1,2,4,11-tetraazatricyclo[7.3.1.0^{2,6}]trideca-3,7,11-triene and 3,12-Dimesityl-6,8,13-trimethyl-5,10-dioxa-1,2,4,11-tetraazatricyclo[7.3.1.0^{2,6}]trideca-3,7,11-triene

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Abstract

X-ray crystallographic study of the title compounds, $C_{27}H_{32}N_4O_2$ and $C_{28}H_{34}N_4O_2$, respectively, allows us to establish the structures of the diadducts obtained from the condensation of nitrile oxides and 1,2-diazepines. The conformations of the five-, six- and seven-membered rings forming the tridecatriene are described.

Comment

As part of our program on the synthesis of bi- or triheterocyclic systems which have biological activity, we are studying cycloaddition reactions on seven-membered heterocycles (Hasnaoui, El Messaoudi & Lavergne, 1985; El Messaoudi, Hasnaoui, El Mouhtadi, Goupil & Lavergne, 1988; Hasnaoui, Baouid & Lavergne, 1991; El Messaoudi, Hasnaoui, El Mouhtadi & Lavergne, 1992; Baouid, Benharref, Hasnaoui & Lavergne, 1994). In a recent publication (El Messaoudi, Hasnaoui & Lavergne, 1994; El Messaoudi, Hasnaoui, Lavergne & Pierrot, 1995), we have described, in particular, the 1,3-dipolar cycloadditions of the 1,2-diazepines (I) and (II) with nitrile oxides, which lead to the title compounds (III) and (IV), respectively. X-ray diffraction study of compounds (III) and (IV) allows us to assign, without ambiguity, a structure of type *A*.

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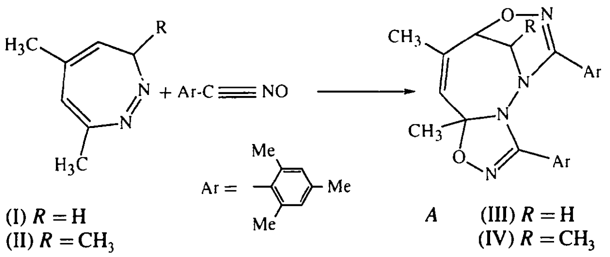
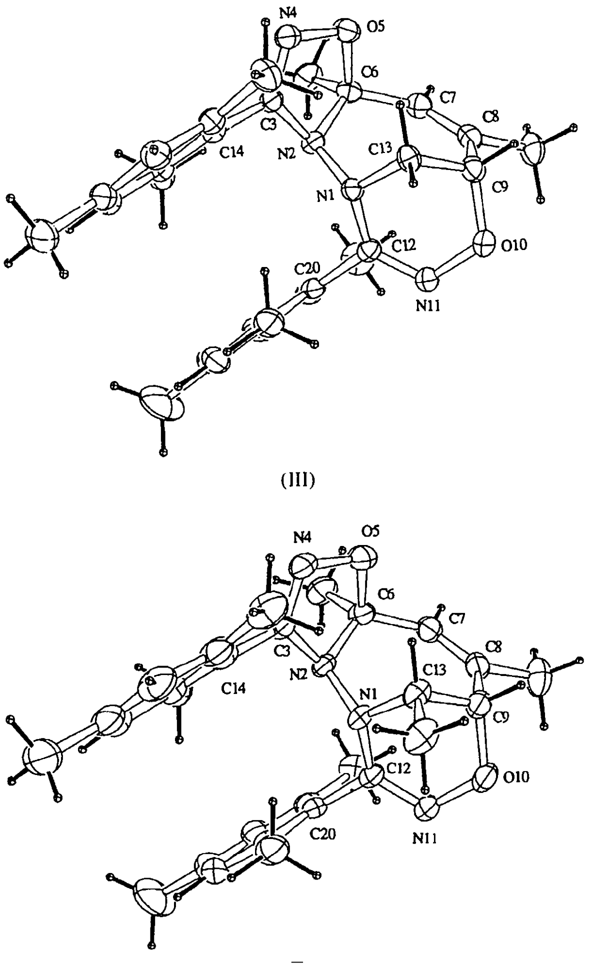


(I) $R = H$
(II) $R = CH_3$

(III) $R = H$
(IV) $R = CH_3$

Selected bond distances and angles (Table 2) show no significant differences between the two molecules: only two distances (C8—C9 and C9—O10) and one angle (C9—C13—N1) are slightly above the 3σ threshold. As can be seen in Fig. 1, the two mesityl groups are parallel, making angles of 2.0 (8) in (III) and 7.4 (5)° in (IV). The tridecadiene fragment is composed of three rings, the central seven-membered ring sharing one bond (N2—C6) with the five-membered ring and one angle (N1—C13—C9) with the six-membered ring. The conformation of these rings is imposed by the double-bond system. In each of these rings, one (five- or six-membered rings) or two (seven-membered ring) torsion angles are almost flat. The five-membered ring with the C6 atom out of the N2—C3—N4—O5 plane has an envelope conformation. The seven-membered ring is composed of a flat moiety C6—C7—C8—C9—C13 connected to a twist segment C13—N1—N2—C6 where the N1 and N2 atoms are located on the same side of the plane. In the six-membered ring the conjugated fragment due to the N11=C12 double bond is not N1—C12=N11—O10 (this torsion angle is equal to about 10° in both molecules) but C13—N1—C12=N11, which is connected to the twist segment N11—O10—C9—C13.



(III)

(IV)

Fig. 1. *ORTEP*II (Johnson, 1976) drawings of the title molecules with heavy atoms represented as 50% probability ellipsoids and H atoms as spheres of arbitrary radii. Phenyl rings are numbered sequentially (C14–C19 and C20–C25) with the number of each methyl substituent being appended by *A* (see Table 1).

Experimental

Crystallization was carried out at room temperature by adding a few drops of ethanol to a trichloromethane saturated solution.

Compound (III)

Crystal data

$C_{27}H_{32}N_4O_2$
$M_r = 444.58$
Triclinic
$P\bar{1}$
$a = 10.169\,(3)$ Å
$b = 8.748\,(2)$ Å
$c = 15.254\,(5)$ Å
$\alpha = 104.45\,(5)°$
$\beta = 97.40\,(5)°$
$\gamma = 109.22\,(5)°$
$V = 1207\,(1)$ Å^3
$Z = 2$
$D_x = 1.223$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12–14°$
$\mu = 0.073$ mm^{-1}
$T = 293$ K
Prismatic
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3992 measured reflections
3775 independent reflections
2462 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.025$
$\theta_{\rm max} = 24°$
$h = -11 \rightarrow 11$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 17$
3 standard reflections frequency: 60 min intensity decay: 1%

Refinement

Refinement on F
$R = 0.041$
$wR = 0.054$
$S = 1.84$
2462 reflections
394 parameters
H atoms: see below
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.243$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.25$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (IV)

Crystal data

$C_{28}H_{34}N_4O_2$
$M_r = 458.61$
Triclinic
$P\overline{1}$
$a = 9.894$ (3) Å
$b = 9.908$ (3) Å
$c = 14.780$ (5) Å
$\alpha = 105.29$ (5)°
$\beta = 94.07$ (5)°
$\gamma = 112.20$ (5)°
$V = 1270$ (1) Å^3
$Z = 2$
$D_x = 1.199$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12–14°$
$\mu = 0.072$ mm^{-1}
$T = 293$ K
Prismatic
0.8 × 0.6 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4125 measured reflections
3950 independent reflections
2538 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.032$
$\theta_{\rm max} = 24°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: 1.6%

Refinement

Refinement on F
$R = 0.040$
$wR = 0.053$
$S = 1.85$
2538 reflections
409 parameters
H atoms: see below
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.25$ 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)*

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

	x	y	z	$B_{\rm eq}$
Compound (III)				
O5	0.2462 (2)	−0.0139 (2)	0.5009 (1)	3.83 (4)
O10	−0.1943 (2)	−0.2830 (2)	0.2469 (1)	3.82 (4)
N1	0.1016 (2)	−0.1713 (2)	0.2846 (1)	2.64 (4)
N2	0.2111 (2)	−0.1779 (2)	0.3502 (1)	2.76 (4)
N4	0.3565 (2)	0.0708 (2)	0.4608 (1)	3.61 (5)
N11	−0.1259 (2)	−0.3819 (2)	0.1974 (1)	3.37 (5)
C3	0.3323 (2)	−0.0202 (3)	0.3770 (2)	2.91 (5)
C6	0.1817 (2)	−0.1908 (3)	0.4409 (2)	3.22 (5)
C6*A*	0.2682 (3)	−0.2839 (3)	0.4768 (2)	4.50 (7)
C7	0.0279 (2)	−0.2672 (3)	0.4454 (2)	3.51 (6)
C8	−0.0929 (2)	−0.2621 (3)	0.4035 (2)	3.31 (6)
C8*A*	−0.2338 (3)	−0.3680 (3)	0.4180 (2)	4.58 (7)
C9	−0.1092 (2)	−0.1656 (3)	0.3361 (2)	3.35 (5)
C12	0.0110 (2)	−0.3303 (3)	0.2222 (1)	2.83 (5)
C13	0.0293 (2)	−0.0586 (3)	0.3202 (2)	3.08 (5)
C14	0.4165 (2)	0.0314 (3)	0.3100 (2)	3.08 (5)
C15	0.4794 (2)	−0.0746 (3)	0.2630 (2)	3.45 (6)
C15*A*	0.4725 (2)	−0.2365 (3)	0.2832 (2)	4.23 (6)
C16	0.5519 (2)	−0.0264 (3)	0.1979 (2)	4.28 (7)
C17	0.5655 (3)	0.1235 (3)	0.1774 (2)	4.33 (7)
C17*A*	0.6397 (3)	0.1703 (4)	0.1032 (2)	6.17 (9)
C18	0.5065 (3)	0.2283 (3)	0.2270 (3)	4.28 (7)
C19	0.4328 (2)	0.1873 (3)	0.2935 (2)	3.48 (6)
C19*A*	0.3759 (3)	0.3124 (3)	0.3459 (2)	4.65 (7)
C20	0.0787 (2)	−0.4401 (3)	0.1712 (2)	3.14 (5)
C21	0.0691 (3)	−0.5924 (3)	0.1880 (2)	4.09 (6)
C21*A*	−0.0043 (3)	−0.6504 (3)	0.2600 (2)	5.33 (8)
C22	0.1313 (3)	−0.6930 (3)	0.1364 (2)	5.61 (7)
C23	0.1988 (3)	−0.6466 (3)	0.0700 (2)	6.10 (8)
C23*A*	0.2578 (4)	−0.7642 (5)	0.0102 (3)	10.7 (1)
C24	0.2057 (3)	−0.4956 (4)	0.0532 (2)	5.32 (7)
C25	0.1462 (2)	−0.3900 (3)	0.1034 (2)	3.89 (6)
C25*A*	0.1527 (3)	−0.2311 (3)	0.0807 (2)	4.56 (7)
Compound (IV)				
O5	0.2148 (2)	−0.0324 (2)	0.5084 (1)	4.55 (4)
O10	−0.2301 (1)	−0.3428 (2)	0.2484 (1)	4.65 (4)
N1	0.0776 (2)	−0.2056 (2)	0.2881 (1)	3.21 (4)
N2	0.1917 (2)	−0.1932 (2)	0.3584 (1)	3.28 (4)
N4	0.3300 (2)	0.0442 (2)	0.4644 (1)	4.45 (5)
N11	−0.1479 (2)	−0.4201 (2)	0.2015 (1)	4.00 (4)
C3	0.3132 (2)	−0.0454 (2)	0.3806 (1)	3.62 (5)
C6	0.1581 (2)	−0.1953 (2)	0.4536 (1)	3.72 (5)
C6*A*	0.2548 (2)	−0.2588 (3)	0.4976 (2)	5.03 (6)
C7	−0.0013 (2)	−0.2773 (2)	0.4601 (2)	4.11 (6)
C8	−0.1289 (2)	−0.2936 (2)	0.4126 (2)	4.22 (6)
C8*A*	−0.2744 (3)	−0.3965 (3)	0.4306 (2)	6.45 (8)
C9	−0.1494 (2)	−0.2231 (2)	0.3379 (2)	4.15 (5)
C12	−0.0052 (2)	−0.3554 (2)	0.2272 (1)	3.26 (5)
C13	−0.0077 (2)	−0.1150 (2)	0.3185 (2)	3.92 (5)
C13*A*	−0.0381 (3)	−0.0473 (3)	0.2425 (2)	5.64 (7)
C14	0.4062 (2)	−0.0036 (3)	0.3106 (2)	4.49 (6)
C15	0.4733 (2)	−0.0999 (3)	0.2660 (2)	5.22 (7)
C15*A*	0.4625 (2)	−0.2390 (3)	0.2926 (2)	5.75 (7)
C16	0.5572 (3)	−0.0587 (4)	0.1987 (2)	7.4 (1)
C17	0.5784 (3)	0.0731 (4)	0.1749 (2)	8.4 (1)
C17*A*	0.6708 (4)	0.1125 (6)	0.1000 (2)	12.4 (2)
C18	0.5165 (3)	0.1681 (3)	0.2220 (2)	7.42 (9)
C19	0.4303 (3)	0.1333 (3)	0.2897 (2)	5.49 (7)
C19*A*	0.3709 (3)	0.2452 (3)	0.3400 (2)	6.88 (8)
C20	0.0767 (2)	−0.4423 (2)	0.1782 (1)	3.57 (5)
C21	0.0793 (2)	−0.5682 (2)	0.2025 (2)	4.35 (6)
C21*A*	0.0036 (3)	−0.6190 (3)	0.2793 (2)	5.81 (7)
C22	0.1570 (3)	−0.6459 (3)	0.1541 (2)	6.07 (7)
C23	0.2303 (3)	−0.6019 (3)	0.0840 (2)	6.89 (8)
C23*A*	0.3146 (4)	−0.6896 (4)	0.0318 (3)	11.7 (1)
C24	0.2249 (3)	−0.4784 (3)	0.0609 (2)	6.10 (8)
C25	0.1486 (2)	−0.3964 (3)	0.1069 (2)	4.47 (6)
C25*A*	0.1434 (3)	−0.2651 (3)	0.0769 (2)	5.78 (7)

Table 2. *Selected bond lengths (Å) and angles (°)*

	(III)	(IV)
N1—N2	1.424 (3)	1.426 (2)
N2—C3	1.435 (2)	1.434 (2)
C3—N4	1.269 (3)	1.276 (3)
N4—O5	1.427 (3)	1.418 (2)
O5—C6	1.461 (2)	1.462 (2)
C6—C7	1.503 (3)	1.500 (3)
C7—C8	1.329 (4)	1.332 (3)
C8—C9	1.510 (4)	1.493 (4)
C9—O10	1.438 (2)	1.449 (2)
O10—N11	1.417 (3)	1.414 (3)
N11—C12	1.284 (3)	1.288 (3)
C12—N1	1.399 (2)	1.399 (2)
N1—C13	1.456 (3)	1.461 (3)
N2—C6	1.476 (3)	1.473 (3)
C9—C13	1.501 (3)	1.510 (3)
C13—N1—N2	116.6 (2)	117.3 (1)
N1—N2—C6	117.8 (2)	117.8 (2)
N2—C6—C7	117.6 (2)	117.6 (2)
C6—C7—C8	133.5 (2)	132.7 (2)
C7—C8—C9	127.2 (2)	127.6 (2)
C8—C9—C13	114.4 (2)	115.3 (2)
C9—C13—N1	107.7 (2)	105.9 (2)
C6—N2—C3	102.1 (1)	101.7 (1)
N2—C3—N4	114.4 (2)	114.1 (2)
C3—N4—O5	107.7 (1)	108.0 (1)
N4—O5—C6	106.1 (2)	105.6 (1)
O5—C6—N2	103.0 (2)	103.3 (2)

C13—C9—O10	107.0 (2)	107.6 (2)
C9—O10—N11	114.7 (2)	114.9 (1)
O10—N11—C12	118.0 (1)	117.7 (1)
N11—C12—N1	125.4 (2)	125.5 (2)
C12—N1—C13	114.7 (2)	115.5 (1)

Table 3. *Torsion angles (°) and distances (Å) to the plane fragments*

	(III)	(IV)
O5—N4—C3—N2	−2.9 (3)	−2.5 (2)
C13—N1—C12—N11	−1.7 (3)	−4.3 (3)
C6—C7—C8—C9	−2.0 (4)	−3.6 (4)
C7—C8—C9—C13	−3.2 (3)	−3.3 (3)

		Distance of atom to plane	
Fragment	Atom	(III)	(IV)
N2—C3—N4—O5	C6	−0.391 (2)	−0.405 (2)
C13—N1—C12—N11	C9	−0.866 (2)	−0.884 (2)
C13—N1—C12—N11	O10	−0.257 (2)	−0.287 (1)
C6—C7—C8—C9—C13	N1	1.111 (2)	1.117 (2)
C6—C7—C8—C9—C13	N2	0.855 (2)	0.885 (2)

Structure solution was completed by Fourier synthesis. H atoms were introduced at idealized positions in the calculations before the last refinement cycle but not refined. Refinement was by full-matrix least-squares methods including anisotropic displacement parameters for all non-H atoms. Despite the large differences in cell parameters [*e.g.* b = 8.748 (3) in (III) and 9.908 (3) Å in (IV)], the two crystals are isostructural and the crystal structure of (IV) was obtained directly from the coordinates of (III) refined with the X-ray intensities measured on the crystal of (IV).

For both compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structures: *MULTAN*11/82 (Main *et al.*, 1982); program(s) used to refine structures: *SDP-Plus* (Frenz, 1985); molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1199). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis,trans-4,5-Diphenyl-1,3,2-dioxathiolane 2-Oxide, *cis,cis*-4,5-Diphenyl-1,3,2-dioxathiolane 2-Oxide and 4,4-Diphenyl-1,3,2-dioxathiolane 2-Oxide

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Abstract

All three title compounds, $C_{14}H_{12}O_3S$, adopt half-chair (envelope) conformations with the S═O group and the phenyl groups in pseudoaxial and pseudoequatorial positions, respectively. The steric effects of the phenyl groups are discussed in terms of S—O/C—O bond lengths and ring torsion angles.

Comment

The conformational analyses of methyl- and phenyl-1,3,2-dioxathiolane 2-oxides (methyl- and phenylethylene sulfites) have been thoroughly investigated by IR and ^{1}H and ^{13}C NMR solution studies (Hellier & Green, 1973*a*,*b*). It was concluded that rapid interconversion between various envelope forms is possible but is restricted to that involving rotation about the C—C bond. Complete pseudorotatory circuits around the ring are prevented by the relatively high potential barrier imposed by the sulfite group.

The crystal structure determinations of *trans*-4(*S*)-phenyl-1,3,2-dioxathiolane 2-oxide, (IV), and *trans,trans*-4,5-diphenyl-1,3,2-dioxathiolane 2-oxide, (V), have been reported previously (Lowe, Jones & Salamone, 1984) and fully confirm the half-chair conformation of these compounds.

In view of our interest in cyclic sulfites and the fact that neither the molecular structures of five-membered ring sulfites nor the effect of substituent groups have yet been studied in detail, we decided to investigate the structures of *cis,trans*-4,5-diphenyl-1,3,2-dioxathiolane 2-oxide, (I), *cis,cis*-4,5-diphenyl-1,3,2-dioxathiolane 2-