

- LAP, B. V. & PADDON-ROW, M. N. (1979). *J. Org. Chem.* **44**, 4979–4981.
- LUFF, W. & KEESE, R. (1991). *Top. Stereochem.* **20**, 231–318.
- MOTHERWELL, W. D. S. & CLEGG, W. (1983). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- PINKERTON, A. A., SCHWARZENBACH, D., BIRBAUM, J.-L., CARRUPT, P.-A., SCHWAGER, L. & VOGEL, P. (1984). *Helv. Chim. Acta*, **67**, 1136–1153.
- WATSON, W. H., KASHYAP, R. P. & PLUMMER, B. F. (1991). *Acta Cryst.* **C47**, 584–587.
- WINSTEIN, S. & HANSEN, R. L. (1960). *Tetrahedron Lett.* pp. 4–8.

*Acta Cryst.* (1992). **C48**, 2151–2154

## Functionalized Hydrocarbons with Condensed Ring Skeletons. XII.\* A Methyltricyclo[8.4.0.0<sup>2,7</sup>]tetradec-8-ene and a Vinylcyclododeca-3,9-diene, Products from Competing Intramolecular Diels–Alder Cycloadditions

BY MARC DROUIN

*Laboratoire de Chimie Structurale et Modélisation Moléculaire, Université de Sherbrooke, Sherbrooke, Québec, Canada J1K 2R1*

SERGE LAMOTHE

*Laboratoire de Synthèse Organique, Université de Sherbrooke, Sherbrooke, Québec, Canada J1K 2R1*

AND ANDRÉ G. MICHEL†

*Laboratoire de Chimie Structurale et Modélisation Moléculaire, Université de Sherbrooke, Sherbrooke, Québec, Canada J1K 2R1*

(Received 16 July 1991; accepted 16 April 1992)

**Abstract.** (III) Tetramethyl 1,10-*trans*-1,2-*cisoid*-2,7-*trans*-1-methyltricyclo[8.4.0.0<sup>2,7</sup>]tetradec-8-ene-5,5,-12,12-tetracarboxylate, C<sub>23</sub>H<sub>32</sub>O<sub>8</sub>, *M<sub>r</sub>* = 436.50, tetragonal, *P*4<sub>2</sub>/*mbc*, *a* = 13.1959 (8), *c* = 26.6986 (11) Å, *V* = 4648.3 (2) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.247 Mg m<sup>-3</sup>, λ(Cu *Kα*) = 1.54056 Å, μ = 0.74 mm<sup>-1</sup>, *F*(000) = 1872.0, *T* = 293 K, final *R* = 0.063 for 2950 observed reflections. (IV) Tetramethyl (3*E*,9*Z*)-9-methyl-2-vinylcyclododeca-3,9-diene-1,1,-6,6-tetracarboxylate, C<sub>23</sub>H<sub>32</sub>O<sub>8</sub>, *M<sub>r</sub>* = 436.50, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 12.767 (5), *b* = 10.199 (3), *c* = 19.175 (3) Å, β = 109.21 (2)°, *V* = 2357 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.230 Mg m<sup>-3</sup>, λ(Cu *Kα*) = 1.54056 Å, μ = 0.73 mm<sup>-1</sup>, *F*(000) = 936.0, *T* = 293 K, final *R* = 0.046 for 2644 observed reflections. Structure (III) consists of three angularly fused six-membered rings *A*, *B* and *C*. The relative stereochemistry is *trans* for the *AB* ring junction, *syn* between the methyl group on C(1) and the C(2) proton and *trans* for the *BC* ring junction (abbreviated to TST). The double bonds Δ<sup>1,2</sup>, Δ<sup>7,8</sup> and Δ<sup>9,10</sup> in the allylic chloride precursor tetramethyl 14-chloro-5-methyltetradeca-4,10,12-triene-1,1,8,8-tetracarboxylate (I) and the

corresponding double bonds in the cyclization product (IV) are *cis*, *trans* and *trans*. The cyclization of the *cis*–*trans* allylic chloride (I) can produce two different compounds from competing *S<sub>N</sub>2* and *S<sub>N</sub>2'* intramolecular substitution reactions. The *S<sub>N</sub>2* displacement produces a reactive 14-membered ring (II) which rapidly undergoes Diels–Alder cycloaddition to tricyclic compound (III), while the *S<sub>N</sub>2'* reaction produces a final stable vinylic 12-membered ring (IV).

**Introduction.** The intramolecular version of the Diels–Alder cycloaddition reaction has attracted considerable interest in the past 20 years. The reasons for this are that this reaction provides better regio- and stereocontrol as well as, for most cases, increased reactivity over the corresponding intermolecular process. Recent communications from these laboratories (Baettig, Dallaire, Pitteloud & Deslongchamps, 1987; Baettig, Marinier, Pitteloud & Deslongchamps, 1987; Bérubé & Deslongchamps, 1987; Lamothe, Ndibwami & Deslongchamps, 1988*a*) showed that selectivity and reactivity can be further improved when the diene and the dienophile are held together by two chains of atoms. One of these papers reported a general study of transannular

\* Part XI: Drouin, Michel-Dewez & Michel (1993).

† To whom correspondence should be addressed.

cycloaddition of 14-membered macrocyclic trienes yielding tricyclic compounds of predictable relative stereochemistry (Lamothe, Ndibwami & Deslongchamps, 1988b). One of the cases studied was that of the *trans-trans-cis* macrocycle (II) obtained from S<sub>N</sub>2 cyclization of allylic chloride (I) at 353 K. It was found that at this temperature, triene (II) underwent an intramolecular cycloaddition to give the tricyclic compound (III). The present study was undertaken in order to assign the structure of compound (III) as well as that of the major by-product (IV).

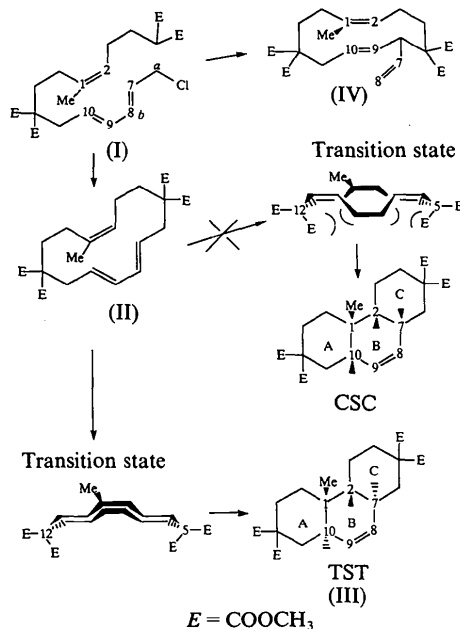


Table 1. Final coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B <sub>eq</sub>
(III)				
C(1)	0.56878 (18)	0.74542 (19)	0.02948 (6)	4.32 (13)
C(2)	0.53464 (19)	0.64571 (19)	0.05332 (7)	4.66 (13)
C(3)	0.53206 (19)	0.65357 (19)	0.11117 (8)	4.73 (14)
C(4)	0.63244 (19)	0.68981 (17)	0.13463 (7)	3.98 (13)
C(5)	0.67091 (19)	0.78482 (17)	0.10829 (7)	4.47 (13)
C(6)	0.67404 (19)	0.77132 (17)	0.05099 (7)	4.40 (13)
C(7)	0.71817 (21)	0.86175 (19)	0.02539 (7)	5.88 (15)
C(8)	0.4893 (5)	0.8296 (4)	0.03882 (14)	5.8 (3)
C(9)	0.60950 (19)	0.71730 (18)	0.18931 (8)	4.48 (14)
C(10)	0.6197 (3)	0.65868 (23)	0.27389 (8)	7.58 (20)
C(11)	0.71062 (19)	0.60489 (18)	0.13374 (7)	4.43 (14)
C(12)	0.87794 (22)	0.56020 (21)	0.15668 (11)	8.19 (20)
O(1)	0.57384 (15)	0.79386 (13)	0.20293 (5)	7.17 (11)
O(2)	0.63245 (15)	0.64085 (13)	0.21970 (5)	6.17 (10)
O(3)	0.69463 (13)	0.51929 (13)	0.12156 (5)	5.65 (10)
O(4)	0.79980 (12)	0.63855 (13)	0.14955 (6)	5.91 (10)
(IV)				
C(1)	0.74160 (20)	0.14900 (20)	-0.31610 (10)	3.66
C(2)	0.65910 (20)	0.1655 (3)	-0.28890 (10)	3.85
C(3)	0.65520 (20)	0.1216 (3)	-0.21560 (10)	4.11
C(4)	0.66390 (20)	0.23840 (20)	-0.16300 (10)	3.49
C(5)	0.67830 (20)	0.20290 (20)	-0.08180 (10)	3.36
C(6)	0.80310 (20)	0.19470 (20)	-0.03210 (10)	3.69
C(7)	0.85170 (20)	0.3297 (3)	-0.01430 (10)	4.27
C(8)	0.8919 (3)	0.3771 (4)	0.05320 (20)	6.49
C(9)	0.87040 (20)	0.10890 (20)	-0.06550 (10)	3.62
C(10)	0.96080 (20)	0.14270 (20)	-0.07870 (10)	3.64
C(11)	1.02280 (20)	0.05720 (20)	-0.11450 (10)	3.98
C(12)	1.04620 (20)	0.12550 (20)	-0.18030 (10)	3.73
C(13)	0.93930 (20)	0.18280 (20)	-0.23650 (10)	3.79
C(14)	0.85010 (20)	0.08370 (20)	-0.27400 (10)	3.96
C(15)	0.7331 (3)	0.1982 (3)	-0.39110 (10)	5.22
C(16)	1.10160 (20)	0.0348 (3)	-0.22080 (10)	4.15
C(17)	1.1539 (3)	-0.1807 (3)	-0.23820 (20)	6.91
C(18)	1.12770 (20)	0.2372 (3)	-0.15060 (10)	4.53
C(19)	1.3072 (3)	0.2850 (4)	-0.06890 (20)	6.17
C(20)	0.62550 (20)	0.30800 (20)	-0.04710 (10)	3.60
C(21)	0.58990 (20)	0.3604 (3)	0.06390 (10)	5.38
C(22)	0.61590 (20)	0.07600 (20)	-0.08000 (10)	4.38
C(23)	0.4391 (3)	-0.0201 (3)	-0.11900 (20)	7.64
O(1)	1.14190 (20)	0.07360 (20)	-0.26500 (10)	7.23
O(2)	1.10180 (10)	-0.08980 (20)	-0.20240 (10)	5.29
O(3)	1.11060 (20)	0.35080 (20)	-0.16410 (10)	6.60
O(4)	1.22330 (20)	0.19120 (20)	-0.10460 (10)	5.35
O(5)	0.59040 (10)	0.41190 (20)	-0.07330 (10)	4.93
O(6)	0.62690 (10)	0.26770 (20)	0.01960 (10)	4.46
O(7)	0.65710 (20)	-0.02540 (20)	-0.05240 (10)	6.36
O(8)	0.50810 (10)	0.09360 (20)	-0.11300 (10)	5.11

**Experimental.** An Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Cu K $\alpha$  radiation was used to collect data for a clear cubic crystal, 0.20  $\times$  0.20  $\times$  0.20 mm.

For compound (III), lattice parameters were refined from 24 reflections with  $2\theta$  of 60–100°. Intensity measurements were made in the range  $2 \leq 2\theta \leq 143^\circ$  (index limits:  $h + 15$ ;  $k + 11$ ;  $l + 32$ ), using the  $2\theta/\omega$ -scan mode. One standard reflection, remeasured every 60 min, showed no significant deviation. A total of 8360 reflections was measured. 4416 reflections were unique of which 2950 were observed with  $I_{\text{net}} \geq 2.5\sigma(I_{\text{net}})$  ( $R_{\text{int}} = 0.019$ ). The structure was solved by direct methods and refined using the *NRCVAX* system (Gabe, Lee & Le Page, 1985). The structure of (III) was solved in the space group *P4<sub>2</sub>bc* with one molecule per asymmetric unit. Although not symmetrically related, two C(8) atoms with 50:50 occupancies appeared in a difference Fourier synthesis. The presence of static disorder was confirmed by another refinement using the space group *P4<sub>2</sub>* with two molecules per asymmetric unit, leading

again to 50:50 occupancies in both molecules for C(8) atoms. The refinement was completed using the space group *P4<sub>2</sub>/mbc*, revealed by the application of the *MISSYM* program (Le Page, 1987), and with one half molecule per asymmetric unit. No correction was made for absorption. All non-H atoms were refined anisotropically. All H-atom positions were calculated and refined. Weights based on counting statistics were used. The function  $\sum w(|F_o| - |F_c|)^2$  with  $w = 1/(\sigma^2 F_o)$  was minimized to give at convergence  $R = 0.063$ ,  $wR = 0.036$  and  $S = 3.959$ ;  $(\Delta/\sigma)_{\text{max}} = 0.08$  for 214 parameters. The secondary-extinction coefficient was 0.71 (4) (Larson, 1967; Zachariasen, 1963). The maximum and minimum peak heights in the final difference Fourier map were 0.40 and  $-0.36 \text{ e \AA}^{-3}$ . Relatively high values obtained for the goodness of fit are attributed to the disordered character of the crystal structure.

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

For compound (III), the following atoms are the symmetry equivalents: C(1a)  $x = 0.56878$ ,  $y = 0.74542$ ,  $z = -0.02948$  ( $x$ ,  $y$ ,  $-z$ ); C(7a)  $x = 0.71817$ ,  $y = 0.86175$ ,  $z = -0.02539$  ( $x$ ,  $y$ ,  $-z$ ).

(III)			
C(1)—C(1a)	1.574 (3)	C(5)—C(6)	1.541 (3)
C(1)—C(2)	1.529 (3)	C(6)—C(7)	1.493 (3)
C(1)—C(6)	1.541 (3)	C(7)—C(7a)	1.356 (4)
C(1)—C(8)	1.548 (6)	C(9)—O(1)	1.172 (3)
C(2)—C(3)	1.548 (3)	C(9)—O(2)	1.330 (3)
C(3)—C(4)	1.541 (3)	C(10)—O(2)	1.476 (3)
C(4)—C(5)	1.525 (3)	C(11)—O(3)	1.194 (3)
C(4)—C(9)	1.534 (3)	C(11)—O(4)	1.327 (3)
C(4)—C(11)	1.523 (3)	C(12)—O(4)	1.473 (3)
C(1a)—C(1)—C(2)	114.59 (18)	C(4)—C(5)—C(6)	111.84 (18)
C(1a)—C(1)—C(6)	111.87 (18)	C(1)—C(6)—C(5)	111.80 (18)
C(1a)—C(1)—C(8)	99.27 (20)	C(1)—C(6)—C(7)	110.98 (19)
C(2)—C(1)—C(6)	107.53 (18)	C(5)—C(6)—C(7)	111.87 (18)
C(2)—C(1)—C(8)	110.5 (3)	C(6)—C(7)—C(7a)	117.24 (21)
C(6)—C(1)—C(8)	113.0 (3)	C(4)—C(9)—O(1)	125.32 (20)
C(1)—C(2)—C(3)	111.34 (18)	C(4)—C(9)—O(2)	110.87 (18)
C(2)—C(3)—C(4)	114.03 (19)	O(1)—C(9)—O(2)	123.77 (20)
C(3)—C(4)—C(5)	110.72 (18)	C(4)—C(11)—O(3)	125.47 (22)
C(3)—C(4)—C(9)	106.88 (19)	C(4)—C(11)—O(4)	110.45 (19)
C(3)—C(4)—C(11)	110.33 (19)	O(3)—C(11)—O(4)	124.06 (22)
C(5)—C(4)—C(9)	108.08 (18)	C(9)—O(2)—C(10)	116.83 (19)
C(5)—C(4)—C(11)	111.85 (19)	C(11)—O(4)—C(12)	115.28 (19)
C(9)—C(4)—C(11)	108.81 (17)		
(IV)			
C(1)—C(2)	1.331 (4)	C(12)—C(16)	1.523 (4)
C(1)—C(14)	1.509 (3)	C(12)—C(18)	1.521 (3)
C(1)—C(15)	1.493 (3)	C(13)—C(14)	1.515 (3)
C(2)—C(3)	1.491 (3)	C(16)—O(1)	1.194 (4)
C(3)—C(4)	1.541 (3)	C(16)—O(2)	1.319 (4)
C(4)—C(5)	1.549 (3)	C(17)—O(2)	1.440 (4)
C(5)—C(6)	1.567 (3)	C(18)—O(3)	1.192 (4)
C(5)—C(20)	1.530 (3)	C(18)—O(4)	1.334 (3)
C(5)—C(22)	1.526 (3)	C(19)—O(4)	1.431 (4)
C(6)—C(7)	1.503 (4)	C(20)—O(5)	1.195 (3)
C(6)—C(9)	1.509 (3)	C(20)—O(6)	1.338 (3)
C(7)—C(8)	1.317 (4)	C(21)—O(6)	1.450 (3)
C(9)—C(10)	1.307 (4)	C(22)—O(7)	1.201 (3)
C(10)—C(11)	1.489 (3)	C(22)—O(8)	1.324 (3)
C(11)—C(12)	1.554 (3)	C(23)—O(8)	1.438 (4)
C(12)—C(13)	1.548 (3)		
C(2)—C(1)—C(14)	123.48 (19)	C(13)—C(12)—C(16)	108.98 (15)
C(2)—C(1)—C(15)	121.26 (22)	C(13)—C(12)—C(18)	108.39 (17)
C(14)—C(1)—C(15)	115.25 (24)	C(16)—C(12)—C(18)	105.69 (22)
C(1)—C(2)—C(3)	127.41 (21)	C(12)—C(13)—C(14)	115.54 (17)
C(2)—C(3)—C(4)	111.54 (23)	C(1)—C(14)—C(13)	111.94 (17)
C(3)—C(4)—C(5)	115.86 (18)	C(12)—C(16)—O(1)	122.8 (3)
C(4)—C(5)—C(6)	112.61 (21)	C(12)—C(16)—O(2)	114.15 (21)
C(4)—C(5)—C(20)	110.47 (16)	O(1)—C(16)—O(2)	123.1 (3)
C(4)—C(5)—C(22)	109.31 (15)	C(12)—C(18)—O(3)	126.36 (21)
C(6)—C(5)—C(20)	106.16 (15)	C(12)—C(18)—O(4)	110.39 (23)
C(6)—C(5)—C(22)	112.02 (16)	O(3)—C(18)—O(4)	123.23 (24)
C(20)—C(5)—C(22)	106.04 (21)	C(5)—C(20)—O(5)	126.61 (21)
C(5)—C(6)—C(7)	110.52 (17)	C(5)—C(20)—O(6)	109.34 (17)
C(5)—C(6)—C(9)	112.33 (16)	O(5)—C(20)—O(6)	124.02 (23)
C(7)—C(6)—C(9)	112.19 (22)	C(5)—C(22)—O(7)	125.72 (23)
C(6)—C(7)—C(8)	123.9 (3)	C(5)—C(22)—O(8)	110.02 (18)
C(6)—C(9)—C(10)	126.29 (20)	O(7)—C(22)—O(8)	124.25 (23)
C(9)—C(10)—C(11)	124.92 (20)	C(16)—O(2)—C(17)	116.96 (23)
C(10)—C(11)—C(12)	112.66 (17)	C(18)—O(4)—C(19)	117.33 (24)
C(11)—C(12)—C(13)	111.92 (21)	C(20)—O(6)—C(21)	117.08 (19)
C(11)—C(12)—C(16)	112.79 (18)	C(22)—O(8)—C(23)	116.23 (21)
C(11)—C(12)—C(18)	108.80 (15)		

For compound (IV), a total of 3371 reflections was measured. 3317 reflections were unique of which 2644 were observed with  $I_{\text{net}} \geq 3.0\sigma(I_{\text{net}})$ . Lattice parameters were determined using twelve reflections with  $2\theta$  of 25–35°. Lorentz–polarization corrections were applied along with an empirical absorption correction [absorption surface (Walker & Stuart, 1983)]. Maximum and minimum correction coefficients applied to  $F_o$  were 1.3176 and 0.7210. Intensity measurements were made in the range  $2 \leq 2\theta \leq 114^\circ$

(index limits:  $h + 13$ ;  $k + 11$ ;  $l \pm 19$ ), using the  $2\theta/\omega$ -scan mode. Three intensity standard reflections, remeasured every 250 min of X-ray exposure time, showed a mean change in intensity of  $-1.5$  (0.4)%; therefore no decay correction was applied. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). H atoms were placed from difference maps and at calculated positions and assigned temperature factors corresponding to those of their attached atoms. Positional and anisotropic thermal parameters for all non-H atoms and positional parameters for all H atoms were refined. The function  $\sum w(|F_o| - |F_c|)^2$  with  $w = 1/(\sigma F_o)^2$  was minimized to give at convergence  $R = 0.046$ ,  $wR = 0.057$  and  $S = 4.99$ ;  $(\Delta/\sigma)_{\text{max}} = 0.01$  for 376 parameters. The maximum and minimum peak heights in the final difference Fourier map were 0.23 and  $-0.20 \text{ e } \text{Å}^{-3}$ . Atomic scattering factors as stored in the *NRCVAX* program are those of Cromer & Waber (1974).\*

**Discussion.** Table 1 gives the positional parameters with their  $B_{\text{eq}}$  values. Fig. 1 shows a *PLUTO* perspective view (Motherwell & Clegg, 1978) of both compounds with the crystallographic numbering. Bond lengths and angles are given in Table 2. From molecular models, it can be deduced that (II) can

\* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55391 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0316]

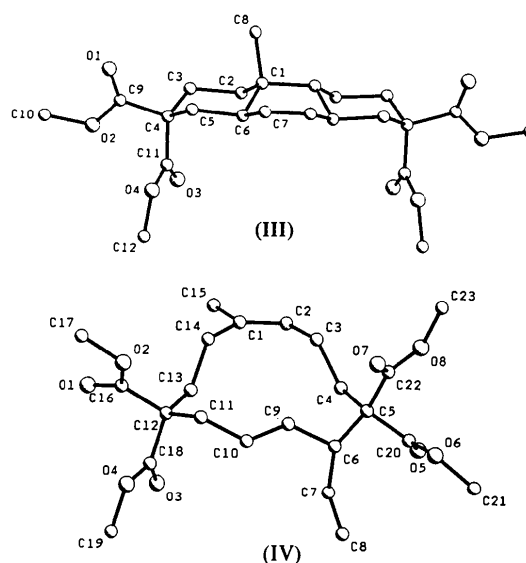


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) perspective view and crystallographic numbering; H atoms are omitted for clarity.

present two different approaches of the diene and the dienophile which could result in two different tricyclic products: *cis-syn-cis* (CSC) and *trans-syn-trans* (TST) configurations. In the transition state the CSC conformation experiences two 1–3 diaxial interactions between the newly formed double bond and the axial methyl esters at C(5) and C(12) (see scheme above). The TST conformation does not have these interactions, resulting in a sterically and energetically favorable chair–boat–chair transition-state conformation. This intramolecular Diels–Alder reaction from non-activated dienophile and diene is realized at low temperature (353 K). The reaction of triene (I) (TTC) to form compound (III) is relatively fast. This can be explained because it is possible for the *trans-trans* diene easily to adopt the *cisoid* configuration since there are no important steric interactions inside the macrocycle. Another important reason is that the two entities are facing each other in the starting material (Lamothe, 1989). This result demonstrates the tremendous reactivity enhancement caused by the transannular arrangement although neither the diene nor the dienophile are activated by polar substituents. Interestingly, the competition for the formation of (II) and (IV) is important: 23% of (IV) was isolated. This compound was simply the result of an S<sub>N</sub>2' reaction of the malonate anion on the allylic chloride favoured by the proximity of the two centers *a* and *b* in the starting material (I).

No abnormally short contacts were observed in either structure.

We are grateful to James P. Springer (Merck Sharp and Dohme Research Laboratories, Rahway, NJ, USA) for his collaboration in the crystal-structure investigation of (IV).

#### References

- BAETTIG, K., DALLAIRE, C., PITTELOU, R. & DESLONGCHAMPS, P. (1987). *Tetrahedron Lett.* **28**, 5249–5252.  
 BAETTIG, K., MARINIER, A., PITTELOU, R. & DESLONGCHAMPS, P. (1987). *Tetrahedron Lett.* **28**, 5253–5254.  
 BÉRUBÉ, G. & DESLONGCHAMPS, P. (1987). *Tetrahedron Lett.* **28**, 5255–5258.  
 CROMER, D. T. & WABER, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)  
 DROUTIN, M., MICHEL-DEWEZ, N. & MICHEL, A. G. (1993). *Acta Cryst.* In the press.  
 GABE, E. J., LEE, F. L. & LE PAGE, Y. (1985). *The NRCVAX Crystal Structure System*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 167–174. Oxford Univ. Press.  
 LAMOTHE, S. (1989). PhD thesis. Univ. of Sherbrooke, Canada.  
 LAMOTHE, S., NDIBWAMI, A. & DESLONGCHAMPS, P. (1988a). *Tetrahedron Lett.* **29**, 1639–1640.  
 LAMOTHE, S., NDIBWAMI, A. & DESLONGCHAMPS, P. (1988b). *Tetrahedron Lett.* **29**, 1641–1644.  
 LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.  
 LE PAGE, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.  
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.  
 SHELDRIK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.  
 WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.  
 ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

*Acta Cryst.* (1992). **C48**, 2154–2157

## Structure of 2,3-Dihydro-1-methylquinolin-2-spiro-2'-indan-3'-spiro-2''-(1'',3''-dithiane)-4,1'-dione

BY ROY L. BEDDOES

*Chemistry Department, University of Manchester, Manchester M13 9PL, England*

ROSA GRIERA AND MERCEDES ALVAREZ

*Laboratory of Organic Chemistry, Faculty of Pharmacy, University of Barcelona, Barcelona 08028, Spain*

AND JOHN A. JOULE\*

*Chemistry Department, University of Manchester, Manchester M13 9PL, England*

(Received 20 December 1991; accepted 25 February 1992)

**Abstract.** C<sub>21</sub>H<sub>19</sub>NO<sub>2</sub>S<sub>2</sub>, *M<sub>r</sub>* = 381.51, monoclinic, *C*2/*c*, *a* = 24.911 (4), *b* = 10.028 (2), *c* = 17.042 (2) Å,

$\beta = 121.367(8)^\circ$ , *V* = 3634.8 (9) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.394 g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54178 \text{ \AA}$ ,  $\mu = 27.25 \text{ cm}^{-1}$ , *F*(000) = 1600, *T* = 295 K, *R* = 0.036 for 2385 observed [*I*/ $\sigma$ (*I*) ≥ 3] reflexions. The dithiane

\* Author to whom correspondence should be addressed