ω-2θ scans of speed 4° min^{-1} and ω-scan width $(1.24 + 0.5\tan\theta)$°
Absorption correction: none
3133 measured reflections
3133 independent reflections

$\theta_{\max} = 50.0$°
$h = -21 \rightarrow 20$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F
Final $R = 0.053$
$wR = 0.047$
$S = 1.19$
2043 reflections
424 parameters
All H-atom parameters refined
Unit weights applied

$(\Delta/\sigma)_{\max} = 0.316$
$\Delta\rho_{\max} = 0.089$ e Å^{-3}
$\Delta\rho_{\min} = -0.016$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (*B*)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N(1)	0.1540 (1)	0.0892 (4)	0.5108 (3)	0.061 (2)
C(1)	0.1915 (2)	0.0468 (4)	0.6176 (3)	0.054 (3)
C(2)	0.2102 (2)	−0.0822 (5)	0.6304 (4)	0.063 (3)
C(3)	0.2474 (2)	−0.1212 (5)	0.7340 (4)	0.062 (3)
C(4)	0.2682 (2)	−0.0359 (4)	0.8255 (3)	0.053 (3)
C(5)	0.2474 (2)	0.0924 (4)	0.8128 (4)	0.052 (3)
C(6)	0.2096 (2)	0.1320 (5)	0.7104 (4)	0.055 (3)
C(7)	0.3086 (2)	−0.0900 (4)	0.9297 (4)	0.057 (3)
C(8)	0.3512 (2)	−0.0335 (4)	1.0112 (3)	0.049 (2)
C(11)	0.0973 (2)	0.1566 (4)	0.5239 (3)	0.054 (3)
C(12)	0.0596 (2)	0.1137 (5)	0.6060 (4)	0.064 (3)
C(13)	0.0050 (2)	0.1791 (5)	0.6222 (4)	0.066 (3)
C(14)	−0.0121 (2)	0.2860 (4)	0.5533 (4)	0.063 (3)
C(15)	0.0248 (2)	0.3273 (5)	0.4684 (4)	0.071 (3)
C(16)	0.0794 (2)	0.2633 (5)	0.4559 (4)	0.065 (3)
O(17)	−0.0648 (1)	0.3588 (3)	0.5616 (3)	0.090 (2)
C(18)	−0.1034 (3)	0.3217 (7)	0.6477 (6)	0.090 (4)
C(21)	0.1685 (2)	0.0532 (4)	0.3976 (3)	0.051 (3)
C(22)	0.2305 (2)	0.0287 (4)	0.3810 (4)	0.062 (3)
C(23)	0.2455 (3)	−0.0087 (5)	0.2707 (5)	0.073 (3)
C(24)	0.2003 (3)	−0.0218 (5)	0.1743 (5)	0.077 (4)
C(25)	0.1395 (3)	0.0039 (5)	0.1881 (4)	0.072 (3)
C(26)	0.1237 (2)	0.0417 (4)	0.2982 (4)	0.060 (3)
C(31)	0.3870 (2)	−0.1102 (4)	1.1089 (3)	0.049 (2)
C(32)	0.4306 (2)	−0.0518 (5)	1.1924 (5)	0.082 (3)
C(33)	0.4624 (3)	−0.1206 (7)	1.2873 (5)	0.089 (4)
C(34)	0.4529 (2)	−0.2494 (6)	1.2996 (4)	0.081 (4)
C(35)	0.4117 (3)	−0.3092 (6)	1.2163 (5)	0.103 (4)
C(36)	0.3799 (3)	−0.2406 (6)	1.1226 (5)	0.090 (4)
C(41)	0.3665 (2)	0.1066 (4)	1.0059 (3)	0.048 (2)
C(42)	0.3446 (2)	0.1972 (5)	1.0820 (4)	0.056 (3)
C(43)	0.3596 (2)	0.3261 (5)	1.0758 (4)	0.064 (3)
C(44)	0.3970 (2)	0.3690 (5)	0.9932 (5)	0.069 (3)
C(45)	0.4194 (2)	0.2800 (6)	0.9190 (5)	0.075 (3)
C(46)	0.4046 (2)	0.1512 (5)	0.9248 (4)	0.063 (3)

Table 4. *Geometric parameters* (Å, °) *for compound* (*B*)

N(1)—C(1)	1.425 (5)	C(8)—C(31)	1.484 (5)
N(1)—C(11)	1.430 (5)	C(8)—C(41)	1.484 (6)
N(1)—C(21)	1.396 (5)	C(14)—O(17)	1.376 (6)
C(4)—C(7)	1.473 (5)	O(17)—C(18)	1.409 (8)
C(7)—C(8)	1.343 (5)		
C(1)—N(1)—C(11)	117.6 (3)	C(7)—C(8)—C(31)	121.0 (4)
C(1)—N(1)—C(21)	120.7 (3)	C(7)—C(8)—C(41)	122.1 (4)
C(11)—N(1)—C(21)	121.3 (3)	C(13)—C(14)—O(17)	124.5 (4)
C(3)—C(4)—C(7)	116.7 (4)	C(14)—O(17)—C(18)	117.9 (5)
C(4)—C(7)—C(8)	131.1 (5)		

Locations of H atoms in both compounds were found from difference Fourier maps, but the 12 H atoms bonded to the methyl C atoms C(17), C(27), C(17)′ and C(27)′ in (*A*) could not be located owing to their free rotation. The high R value for compound (*A*) may result from the poor crystal quality and exclusion of the 12 H atoms from the structure-factor calculations.

Data collection: MXC^3 control. Data reduction: Mac Science software. Program(s) used to solve structure: *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Program(s) used to refine structure: *DS*SYSTEM* (Okada & Koyama, 1991).

The author is grateful to Professors Y. Saito and M. Tokonami (The University of Tokyo) for valuable discussions, as well as Dr K. Tani. Mr K. Okada (Ricoh Company Ltd) is thanked for the modification of his program.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71127 (57 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1045]

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Two Novel Ten-Membered Ring Compounds: 1,2,5,8-Dithiadiazecine-6,7-diones

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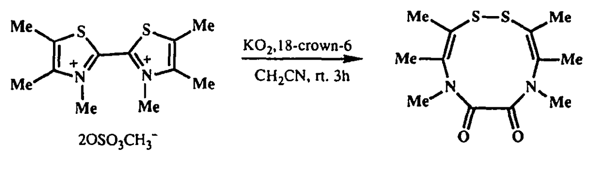
Abstract

The structures of two new ten-membered ring compounds, 3,4,5,8,9,10-hexamethyl-1,2,5,8-dithiadiazecine-6,7(5*H*,8*H*)-dione (I) and 11,14-dimethyldibenzo[*c*,*i*]-

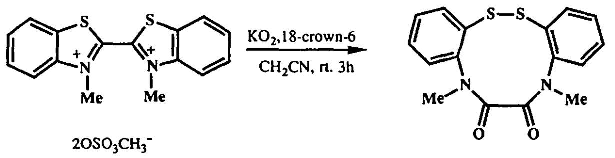
[1,2,5,8]dithiadiazecine-12,13-(11*H*,14*H*)-dione (II), have been determined. The two ten-membered rings, exhibiting asymmetric conformation, are almost identical. The disulfide bond lengths are 2.058 (1) and 2.053 (1) Å for (I) and (II), respectively.

Comment

In the course of our investigation of superoxides of electron-deficient heteroaromatics (Itoh, Nagata, Okada & Ohsawa, 1990, 1992; Itoh, Nagata, Okada, Takahashi & Ohsawa, 1991) we recently found that the reaction of 3,3′-dimethyl-2,2′-bithiazolium salts with potassium superoxide afford new ten-membered ring compounds: 1,2,5,8-dithiadiazecine-6,7-diones (Itoh, Nagata, Okada, Yamaguchi & Ohsawa, 1992). This reaction is specific for the superoxide, one of the most noteworthy active oxygen compounds.



(I)



(II)

Figs. 1 and 2 show *ORTEP*II (Johnson, 1976) drawings of molecules (I) and (II), respectively, with the crystallographic numbering system. The ten-membered rings in (I) and (II) exhibit asymmetric conformation as shown in the figures. Two *N*-methyl amide moieties, connected with C6 and C7, are located in opposite directions for both compounds; this results from the repulsion between the two carbonyl O atoms. This probably gives rise to the break down of the symmetry in the ten-membered ring. The asymmetric conformation is also observed in solution by NMR spectroscopy. For both compounds each *N*-methyl amide conformation is *cis*, which is thought to be preferable because *N*-methylbenzanilide exists predominantly in a *cis*-amide conformation in solution and in the crystal form (Itai, Toriumi, Tomioka, Kagechika, Azumaya & Shudo, 1989). The bond lengths and torsion angles about the disulfide bonds are respectively 2.058 (1) Å and 110.6° in (I), and 2.053 (1) Å and 105.1 (2)° in (II). Two S—C torsion angles indicate inconsistent values for both compounds [S1—S2—C3—C4 −98.2 (2) in (I), −89.7 (3)° in (II) and S2—S1—C10—C9 −103.7 (2) in (I), −108.8 (3)° in (II)].

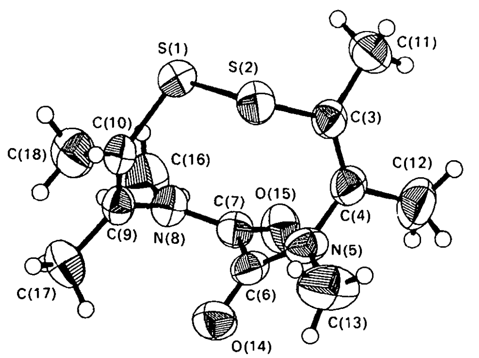


Fig. 1. *ORTEP*II drawing of (I). Ellipsoids are drawn at the 50% probability level while isotropic H-atom thermal parameters are represented by spheres of arbitrary size.

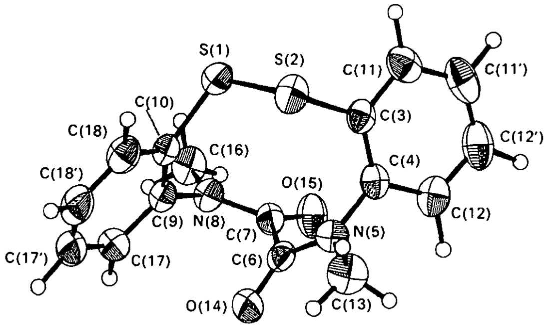


Fig. 2. *ORTEP*II drawing of (II). Ellipsoids are drawn at the 50% probability level while isotropic H-atom thermal parameters are represented by spheres of arbitrary size.

Experimental

Compound (I)

Crystal data

$C_{12}H_{18}N_2O_2S_2$
M_r = 286.41
Monoclinic
$P2_1/c$
a = 11.015 (3) Å
b = 12.870 (1) Å
c = 10.575 (1) Å
β = 94.78 (1)°
V = 1494.0 (5) Å^3
Z = 4
D_x = 1.273 Mg m^{-3}

Cu $K\alpha_1$ radiation
λ = 1.5405 Å
Cell parameters from 20 reflections
θ = 28.0–30.5°
μ = 3.157 mm^{-1}
T = 297 K
Prism
0.35 × 0.30 × 0.15 mm
Clear
Crystal source: chemical synthesis

Data collection

Rigaku AFC-5 diffractometer
$\omega/2\theta$ scans, ω-scan width (1.3 + 0.14tanθ)°, speed 32° min^{-1}
Absorption correction: none
2533 measured reflections
2218 independent reflections
1926 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int}$ = 0.020
$\theta_{\rm max}$ = 65°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 200 reflections
intensity variation: <3%

Refinement

Refinement on F^2
Final R = 0.0396
wR = 0.0464
S = 1.458
1926 reflections
235 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F) + 0.019F^2]$

$(\Delta/\sigma)_{max}$ = 0.16
$\Delta\rho_{max}$ = 0.230 e Å^{-3}
$\Delta\rho_{min}$ = −0.345 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$C_{16}H_{14}N_2O_2S_2$
M_r = 303.42
Monoclinic
$P2_1/n$
a = 27.229 (3) Å
b = 7.649 (1) Å
c = 7.358 (1) Å
β = 90.82 (2)°
V = 1532.5 (7) Å^3
Z = 4
D_x = 1.432 Mg m^{-3}

Cu $K\alpha_1$ radiation
λ = 1.5405 Å
Cell parameters from 20 reflections
θ = 28.5–30.5°
μ = 3.165 mm^{-1}
T = 297 K
Prism
0.30 × 0.20 × 0.25 mm
Clear
Crystal source: chemical synthesis

Data collection

Rigaku AFC-5 diffractometer
$\omega/2\theta$ scans, ω-scan width $(1.3 + 0.14\tan\theta)$°, speed 32° min^{-1}
Absorption correction: none
2937 measured reflections
2609 independent reflections
2333 observed reflections $[F > 3\sigma(F)]$

R_{int} = 0.023
θ_{max} = 65°
$h = -32 \rightarrow 32$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 8$
3 standard reflections monitored every 200 reflections
intensity variation: <3%

Refinement

Refinement on F^2
Final R = 0.0492
wR = 0.0480
S = 1.090
2333 reflections
255 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F) + 0.005F^2]$

$(\Delta/\sigma)_{max}$ = 0.14
$\Delta\rho_{max}$ = 0.239 e Å^{-3}
$\Delta\rho_{min}$ = −0.509 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (I)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S1	0.08380 (6)	0.05351 (6)	0.32954 (7)	0.062 (1)
S2	0.15129 (7)	0.17557 (6)	0.23109 (7)	0.060 (1)
C3	0.1533 (2)	0.1272 (1)	0.0747 (2)	0.051 (1)
C4	0.2542 (2)	0.0895 (2)	0.0323 (2)	0.059 (1)
N5	0.3623 (2)	0.0776 (1)	0.1155 (2)	0.064 (1)
C6	0.3843 (2)	−0.0041 (2)	0.1921 (2)	0.052 (1)
C7	0.2925 (2)	−0.0930 (1)	0.1812 (2)	0.047 (1)
N8	0.2509 (1)	−0.1265 (1)	0.2892 (1)	0.051 (1)
C9	0.2744 (2)	−0.0725 (2)	0.4070 (2)	0.049 (1)
C10	0.2121 (2)	0.0125 (2)	0.4311 (2)	0.052 (1)
C11	0.0351 (3)	0.1405 (3)	−0.0053 (3)	0.072 (1)
C12	0.2656 (5)	0.0591 (4)	−0.1041 (4)	0.103 (1)
C13	0.4564 (4)	0.1574 (3)	0.1100 (8)	0.109 (2)
O14	0.4782 (1)	−0.0152 (1)	0.2611 (2)	0.071 (1)
O15	0.2709 (1)	−0.1359 (1)	0.0780 (1)	0.067 (1)
C16	0.1807 (4)	−0.2233 (3)	0.2900 (4)	0.078 (1)
C17	0.3693 (3)	−0.1212 (2)	0.4977 (3)	0.068 (1)
C18	0.2369 (4)	0.0790 (4)	0.5469 (3)	0.082 (1)

Table 2. *Geometric parameters* (Å, °) *for compound* (I)

S1—S2	2.058 (1)	C6—O14	1.223 (3)
S1—C10	1.782 (2)	C6—C7	1.525 (3)
S2—C3	1.769 (2)	C7—O15	1.228 (3)
C3—C4	1.324 (4)	C7—N8	1.337 (3)
C3—C11	1.503 (4)	N8—C9	1.432 (3)
C4—N5	1.429 (3)	N8—C16	1.467 (4)
C4—C12	1.510 (5)	C9—C10	1.328 (3)
N5—C6	1.337 (3)	C9—C17	1.496 (4)
N5—C13	1.464 (5)	C10—C18	1.500 (5)
C10—S1—S2	103.2 (1)	N5—C6—C7	117.3 (2)
C3—S2—S1	103.7 (1)	O15—C7—N8	124.0 (2)
C4—C3—C11	124.2 (2)	O15—C7—C6	118.9 (2)
C4—C3—S2	121.5 (2)	N8—C7—C6	116.7 (2)
C11—C3—S2	114.1 (2)	C7—N8—C9	122.5 (2)
C3—C4—N5	120.7 (2)	C7—N8—C16	119.8 (2)
C3—C4—C12	123.9 (3)	C9—N8—C16	117.7 (2)
N5—C4—C12	115.4 (3)	C10—C9—N8	120.5 (2)
C6—N5—C4	123.8 (2)	C10—C9—C17	124.6 (2)
C6—N5—C13	118.9 (3)	N8—C9—C17	114.9 (2)
C4—N5—C13	117.2 (3)	C9—C10—C18	124.4 (2)
O14—C6—N5	123.6 (2)	C9—C10—S1	121.7 (2)
O14—C6—C7	118.7 (2)	C18—C10—S1	113.9 (2)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (II)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S1	0.87951 (4)	0.2787 (1)	0.7072 (1)	0.047 (1)
S2	0.92668 (4)	0.0953 (1)	0.6046 (1)	0.052 (1)
C3	0.9626 (1)	0.2073 (5)	0.4407 (5)	0.041 (1)
C4	0.9487 (1)	0.2198 (5)	0.2602 (5)	0.041 (1)
N5	0.9023 (1)	0.1544 (4)	0.1946 (4)	0.041 (1)
C6	0.8605 (1)	0.2483 (5)	0.1922 (5)	0.039 (1)
C7	0.8629 (1)	0.4346 (5)	0.2635 (5)	0.038 (1)
N8	0.8295 (1)	0.4741 (4)	0.3940 (4)	0.039 (1)
C9	0.8008 (1)	0.3397 (5)	0.4767 (5)	0.037 (1)
C10	0.8212 (1)	0.2308 (5)	0.6073 (5)	0.038 (1)
C11	1.0084 (1)	0.2713 (6)	0.4987 (7)	0.053 (1)
C11′	1.0401 (1)	0.3408 (6)	0.3730 (9)	0.062 (2)
C12	0.9814 (1)	0.2871 (6)	0.1346 (6)	0.052 (1)
C12′	1.0272 (1)	0.3471 (6)	0.1923 (8)	0.062 (1)
C13	0.9026 (2)	−0.0203 (6)	0.1157 (8)	0.060 (1)
O14	0.8220 (1)	0.1938 (4)	0.1266 (4)	0.055 (1)
O15	0.8896 (1)	0.5436 (4)	0.1962 (4)	0.054 (1)
C16	0.8235 (2)	0.6527 (6)	0.4591 (7)	0.052 (1)
C17	0.7519 (1)	0.3210 (6)	0.4243 (6)	0.048 (1)
C17′	0.7248 (1)	0.1845 (7)	0.4960 (6)	0.057 (1)
C18	0.7936 (1)	0.0936 (6)	0.6759 (6)	0.053 (1)
C18′	0.7457 (1)	0.0691 (7)	0.6150 (6)	0.060 (1)

Table 4. *Geometric parameters* (Å, °) *for compound* (II)

S1—S2	2.053 (1)	C7—N8	1.366 (5)
S1—C10	1.779 (4)	N8—C9	1.432 (5)

S2—C3	1.783 (4)	N8—C16	1.458 (6)
C3—C4	1.379 (6)	C9—C10	1.382 (5)
C3—C11	1.400 (6)	C9—C17	1.390 (5)
C4—C12	1.391 (6)	C10—C18	1.390 (6)
C4—N5	1.434 (5)	C11—C11′	1.381 (7)
N5—C6	1.346 (5)	C11′—C12′	1.371 (9)
N5—C13	1.457 (6)	C12—C12′	1.389 (7)
C6—O14	1.223 (5)	C17′—C18′	1.362 (7)
C6—C7	1.519 (6)	C17′—C17	1.386 (7)
C7—O15	1.216 (5)	C18′—C18	1.386 (7)
C10—S1—S2	105.5 (1)	C7—N8—C9	120.8 (3)
C3—S2—S1	105.8 (1)	C7—N8—C16	121.2 (3)
C4—C3—C11	120.0 (4)	C9—N8—C16	117.9 (3)
C4—C3—S2	122.7 (3)	C10—C9—C17	120.2 (3)
C11—C3—S2	117.1 (3)	C10—C9—N8	120.9 (3)
C3—C4—C12	119.7 (3)	C17—C9—N8	118.9 (3)
C3—C4—N5	121.8 (3)	C9—C10—C18	119.7 (3)
C12—C4—N5	118.3 (3)	C9—C10—S1	120.6 (3)
C6—N5—C4	123.9 (3)	C18—C10—S1	119.3 (3)
C6—N5—C13	119.6 (3)	C11′—C11—C3	119.5 (4)
C4—N5—C13	116.4 (3)	C12′—C11′—C11	120.6 (4)
O14—C6—N5	123.0 (4)	C12′—C12—C4	119.9 (4)
O14—C6—C7	119.2 (3)	C11′—C12′—C12	120.1 (5)
N5—C6—C7	117.7 (3)	C17′—C17—C9	119.0 (4)
O15—C7—N8	122.8 (3)	C18′—C17′—C17	120.9 (4)
O15—C7—C6	121.7 (3)	C18′—C18—C10	119.7 (4)
N8—C7—C6	115.1 (3)	C17′—C18′—C18	120.2 (4)

Data collection: *AFD* (Rigaku Corporation, 1985*a*). Program(s) used to solve structure: *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985). Program(s) used to refine (full-matrix least squares) structure: *RCRYSTAN* (Rigaku Corporation, 1985*b*). Molecular graphics: *ACV* (Stardent Computer Inc., 1990). Software used to prepare material for publication: *XPACK* (Yamaguchi, 1987).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71096 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1048]

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15,16-Bis(dispiro[2.0.2.1]hept-7-ylidene)-hexaspiro[2.0.2.0.0.0.2.0.2.0.2.0]hexadecane, $C_{30}H_{32}$

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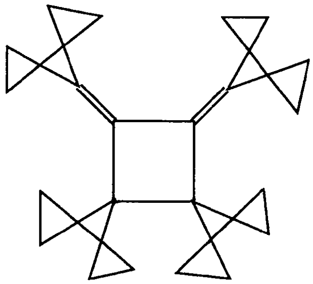
Abstract

The formation of the title compound as the 'head-to-head' dimer in the dimerization of bis(dispiro[2.0.2.1]hept-7-ylidene)methane has been established. The steric repulsion between bulky substituents results in slight bending of the four-membered cycle.

Comment

The X-ray structure of the title compound (I) has been determined as part of our investigations of the branched triangulanes and their derivatives (Yufit, Struchkov, Kozhushkov & De Meijere, 1993, and references therein). The compound (I) was obtained by spontaneous dimerization of bis(dispiro[2.0.2.1]hept-7-ylidene)methane. This [2+2]-cycloaddition reaction may result in the formation of two possible isomers; the present study was performed in order to confirm unequivocally the molecular structure of (I).



(I)

The molecule (I) is the 'head-to-head' isomer. The bond lengths of vicinal (relative to the spiro atoms; average 1.481 Å) and distal (average 1.523 Å) bonds in the terminal cyclopropane rings are in good agreement with those reported for previously studied trian-

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