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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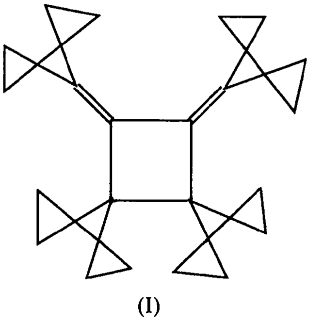
Abstract

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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gulanes [1.480 and 1.528 Å (Lukin, Kozhushkov, Zefirov, Yufit & Struchkov, 1993)]. The bond lengths in the central rings of dispiro[2.0.2.1]heptane moieties are also very similar to corresponding values in ethyl pentaspiro[2.0.2.0.0.2.0.2.0.1]tetradec-14-ylideneacetate (II) (Yufit, Struchkov, Kozhushkov & De Meijere, 1993) and in 7,7′-bis(dispiro[2.0.2.1]heptylidene) (III) (Boese, 1993). The exocyclic double bonds C15═C17 and C16═C24 (average 1.330 Å) are a little shorter than those in simple methylenecyclopropane derivatives (Boese, 1993) and in (III), but very close to that found in (II). Of interest is the similarity of the four-membered ring geometry in molecules (I) and (II) in spite of the presence of the spiro annulated cyclopropane ring in (II) instead of the dispiro[2.0.2.1]heptylidene substitutient in (I). The only difference between the four-membered cycles in (I) and (II) is a slight shortening of the C15—C16 bond in (I) to 1.480 (2) Å, compared to the analogous bond length in (II) of 1.496 (4) Å; this probably results from the more effective conjugation between two double-bond π systems in (I) than between a double-bond π system and Walsh's molecular orbital of the cyclopropane ring in (II). The steric repulsion between bulky substituents in (I) results in some twisting around the C15—C16 bond [torsion angle C17—C15—C16—C24 12.8 (3) °] and slight bending of the four-membered ring [the dihedral angle between the C8—C7—C16 and C8—C15—C16 planes is 9.1 (1) °].

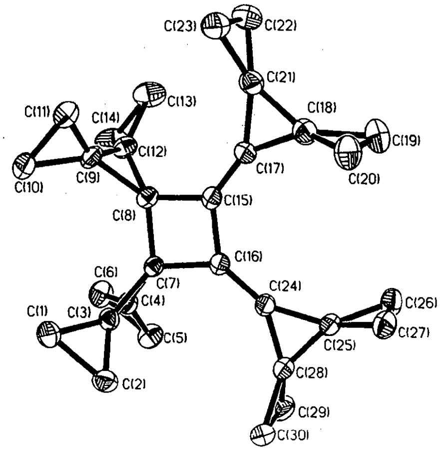

Fig. 1. View of molecule (I) showing the labelling of non-H atoms (H atoms are omitted for clarity).

Experimental

Crystal data

$C_{30}H_{32}$
M_r = 392.56
Triclinic
$P\bar{1}$
a = 10.344 (3) Å
b = 11.270 (3) Å
c = 12.361 (4) Å
α = 102.63 (3)°
β = 106.08 (3)°
γ = 115.82 (2)°
V = 1146.5 (6) Å^3
Z = 2

D_x = 1.137 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 11–12°
μ = 0.064 mm^{-1}
T = 293.0 (10) K
Transparent plate
0.5 × 0.3 × 0.2 mm
Colourless

Data collection

Siemens *P3/PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3918 measured reflections
3672 independent reflections
3087 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int}$ = 0.0221
$\theta_{\rm max}$ = 30.06°
$h = 0 \rightarrow 12$
$k = -15 \rightarrow 14$
$l = -17 \rightarrow 16$
2 standard reflections monitored every 98 reflections intensity variation: 1.5%

Refinement

Refinement on F^2
Final $R(F)$ = 0.0489
$wR(F^2)$ = 0.1142
S = 0.954
3670 reflections
399 parameters
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0490P)^2 + 1.3986P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max}$ = −0.006
$\Delta\rho_{\rm max}$ = 0.220 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.345 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

The final refinement was performed on F^2 for all reflections including those generally believed to be 'unobserved' [$F^2 < 2\sigma(F^2)$] and even those having F^2 negative but larger than $-3\sigma(F^2)$ (in fact, no reflections were rejected according to the latter criterium). The observed threshold $I > 2\sigma(I)$ is used only for calculating R(obs.) *etc.* given here for comparison with refinements on F. Non-H atoms were refined anisotropically, H atoms isotropically. The conventional R factor for the finally refined structure was calculated to be 0.0703 for all reflections and 0.0489 for 3087 'observed' reflections with $F > 4\sigma(F)$. The weighted $wR2$ factor (calculated on F^2) was equal to 0.1435 for all and 0.1142 for 'observed' data. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1992).

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

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

	x	y	z	$U_{\rm eq}$
C1	0.6844 (3)	−0.0640 (3)	0.9354 (2)	0.0325 (14)
C2	0.6511 (3)	0.0539 (3)	0.9276 (2)	0.0295 (13)
C3	0.5507 (3)	−0.0910 (2)	0.8284 (2)	0.0226 (12)
C4	0.4921 (3)	−0.1451 (2)	0.6958 (2)	0.0224 (12)
C5	0.5062 (3)	−0.0799 (3)	0.6057 (2)	0.031 (2)
C6	0.5378 (4)	−0.2003 (3)	0.6023 (3)	0.034 (2)
C7	0.3769 (3)	−0.1998 (2)	0.7500 (2)	0.0185 (11)
C8	0.2748 (3)	−0.3500 (2)	0.7438 (2)	0.0209 (12)
C9	0.3204 (3)	−0.4443 (2)	0.7911 (2)	0.0250 (13)
C10	0.4629 (3)	−0.4312 (3)	0.8756 (3)	0.0341 (14)
C11	0.3056 (3)	−0.5051 (3)	0.8846 (3)	0.0330 (15)
C12	0.2341 (3)	−0.4923 (2)	0.6596 (2)	0.0269 (14)
C13	0.0902 (4)	−0.6245 (3)	0.5615 (3)	0.041 (2)
C14	0.2441 (4)	−0.5536 (3)	0.5454 (3)	0.042 (2)
C15	0.1548 (3)	−0.3131 (2)	0.7578 (2)	0.0208 (12)
C16	0.2446 (3)	−0.1745 (2)	0.7486 (2)	0.0205 (11)

C17	0.0265 (3)	−0.3806 (2)	0.7759 (2)	0.0224 (11)
C18	−0.1053 (3)	−0.3842 (3)	0.7993 (2)	0.0268 (12)
C19	−0.2330 (3)	−0.3553 (3)	0.7550 (3)	0.036 (2)
C20	−0.1295 (3)	−0.2965 (3)	0.8911 (3)	0.036 (2)
C21	−0.0859 (3)	−0.5087 (2)	0.7842 (2)	0.0272 (12)
C22	−0.1887 (3)	−0.6653 (3)	0.7098 (3)	0.038 (2)
C23	−0.0929 (3)	−0.6080 (3)	0.8471 (3)	0.037 (2)
C24	0.2216 (3)	−0.0702 (2)	0.7362 (2)	0.0221 (12)
C25	0.1297 (3)	−0.0020 (2)	0.7315 (2)	0.0235 (12)
C26	−0.0304 (3)	−0.0367 (3)	0.6533 (2)	0.0318 (14)
C27	0.0368 (3)	0.0305 (3)	0.7924 (2)	0.0319 (14)
C28	0.2870 (3)	0.0711 (2)	0.7309 (2)	0.0243 (12)
C29	0.3547 (3)	0.1486 (3)	0.6588 (3)	0.035 (2)
C30	0.4370 (3)	0.2134 (3)	0.7979 (3)	0.0337 (15)

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

C1—C3	1.482 (3)	C15—C17	1.323 (2)
C1—C2	1.526 (4)	C15—C16	1.480 (2)
C2—C3	1.480 (3)	C16—C24	1.328 (3)
C3—C4	1.455 (3)	C17—C21	1.455 (3)
C3—C7	1.507 (3)	C17—C18	1.456 (3)
C4—C5	1.469 (3)	C18—C21	1.482 (3)
C4—C6	1.480 (3)	C18—C19	1.484 (4)
C4—C7	1.494 (3)	C18—C20	1.486 (4)
C5—C6	1.523 (4)	C19—C20	1.521 (4)
C7—C16	1.511 (2)	C21—C23	1.483 (4)
C7—C8	1.526 (3)	C21—C22	1.485 (4)
C8—C9	1.500 (3)	C22—C23	1.520 (4)
C8—C12	1.507 (3)	C24—C28	1.459 (3)
C8—C15	1.510 (3)	C24—C25	1.457 (3)
C9—C12	1.458 (3)	C25—C28	1.473 (3)
C9—C11	1.477 (3)	C25—C27	1.486 (4)
C9—C10	1.475 (3)	C25—C26	1.487 (3)
C10—C11	1.521 (4)	C26—C27	1.523 (4)
C12—C13	1.478 (4)	C28—C29	1.483 (3)
C12—C14	1.482 (4)	C28—C30	1.485 (3)
C13—C14	1.533 (4)	C29—C30	1.520 (4)
C3—C1—C2	58.9 (2)	C17—C15—C16	136.97 (13)
C3—C2—C1	59.1 (2)	C17—C15—C8	132.47 (10)
C4—C3—C2	134.2 (2)	C16—C15—C8	90.51 (9)
C4—C3—C1	138.2 (2)	C24—C16—C15	136.8 (2)
C2—C3—C1	62.0 (2)	C24—C16—C7	132.2 (2)
C4—C3—C7	60.55 (15)	C15—C16—C7	90.87 (12)
C2—C3—C7	138.1 (2)	C15—C17—C21	148.6 (2)
C1—C3—C7	140.2 (2)	C15—C17—C18	150.2 (2)
C3—C4—C5	135.3 (2)	C21—C17—C18	61.2 (2)
C3—C4—C6	137.6 (2)	C17—C18—C21	59.3 (2)
C5—C4—C6	62.2 (2)	C17—C18—C19	140.7 (2)
C3—C4—C7	61.44 (15)	C21—C18—C19	138.6 (2)
C5—C4—C7	137.0 (2)	C17—C18—C20	138.0 (2)
C6—C4—C7	139.6 (2)	C21—C18—C20	135.7 (2)
C4—C5—C6	59.3 (2)	C19—C18—C20	61.6 (2)
C4—C6—C5	58.5 (2)	C18—C19—C20	59.3 (2)
C4—C7—C3	58.01 (15)	C18—C20—C19	59.1 (2)
C4—C7—C16	129.26 (9)	C17—C21—C18	59.4 (2)
C3—C7—C16	124.81 (8)	C17—C21—C23	141.6 (2)
C4—C7—C8	131.0 (2)	C18—C21—C23	137.1 (2)
C3—C7—C8	129.3 (2)	C17—C21—C22	138.2 (2)
C16—C7—C8	88.75 (9)	C18—C21—C22	135.9 (2)
C9—C8—C12	57.99 (15)	C23—C21—C22	61.6 (2)
C9—C8—C15	128.8 (2)	C21—C22—C23	59.2 (2)
C12—C8—C15	124.8 (2)	C21—C23—C22	59.2 (2)
C9—C8—C7	130.8 (2)	C16—C24—C28	147.4 (2)
C12—C8—C7	129.6 (2)	C16—C24—C25	151.5 (2)
C15—C8—C7	89.1 (2)	C28—C24—C25	60.7 (2)
C12—C9—C11	136.6 (2)	C24—C25—C28	59.7 (2)
C12—C9—C10	137.7 (2)	C24—C25—C27	141.1 (2)
C11—C9—C10	62.0 (2)	C28—C25—C27	139.2 (2)
C12—C9—C8	61.25 (15)	C24—C25—C26	137.6 (2)
C11—C9—C8	137.5 (2)	C28—C25—C26	134.4 (2)
C10—C9—C8	138.2 (2)	C27—C25—C26	61.6 (2)
C9—C10—C11	59.1 (2)	C25—C26—C27	59.2 (2)
C9—C11—C10	58.9 (2)	C25—C27—C26	59.2 (2)
C9—C12—C13	135.3 (2)	C24—C28—C25	59.6 (2)
C9—C12—C14	138.5 (2)	C24—C28—C29	141.1 (2)
C13—C12—C14	62.4 (2)	C25—C28—C29	135.6 (2)
C9—C12—C8	60.77 (15)	C24—C28—C30	137.9 (2)
C13—C12—C8	137.0 (2)	C25—C28—C30	138.2 (2)
C14—C12—C8	139.3 (2)	C29—C28—C30	61.6 (2)
C12—C13—C14	58.9 (2)	C28—C29—C30	59.3 (2)
C12—C14—C13	58.7 (2)	C28—C30—C29	59.1 (2)

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71089 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1043]

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Structure of *N*-Nitroso-2,4-diphenyl-3-azabicyclo[3.3.1]nonane

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Abstract

The title compound adopts a twin-chair conformation. The overall conformation is similar in both the molecules of the asymmetric unit. The phenyl rings are equatorially disposed with respect to the central ring system. The plane of the nitroso group makes angles of 31.0 (4) and 33.4 (5)° with the C(2)—N(3)—C(4) plane of the piperidine ring in molecules *A* and *B*, respectively. The N(3)···C(7) non-bonded separation is 3.19 (7) Å for both molecules *A* and *B*.