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The Structure of Diethyl (1*R**,4*S**,5*R**,8*R**)-4,8-Dimethyl-2,3;6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene-4,8-dicarboxylate

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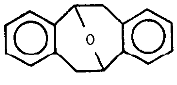
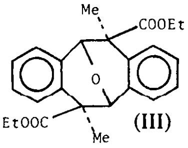
The reaction of ethyl 2-formyl-2-phenylpropionate with FSO_3H yields the unique compound diethyl 4,8-dimethyl-2,3;6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene-4,8-dicarboxylate. The compound crystallizes in space group $P1$ with a = 26·486 (10), b = 8·543 (3), c = 9·467 (8) Å, α = 104·38 (5), β = 102·45 (5), γ = 90·35 (3)°, V = 2022 (2) Å^3 and Z = 4. Counter techniques were used to collect 5620 independent reflections. The structure was solved by application of direct-method techniques, and the model was refined by least-squares procedures to an R factor of 0·076. The eight-membered cyclooctadiene ring exhibits a conformation intermediate between the tub and boat forms. The distortion is imposed by the 1,5-epoxy bridge and the 2,6-diene moieties. The conformations of the two independent molecules are identical except for a twist of one ethoxycarbonyl group.

Introduction

The reaction of phenylacetaldehyde with FSO_3H in CCl_4 was shown to give 2,3;6,7-dibenzo-9-oxabicyclo-[3.3.1]nona-2,6-diene (I) (Kagan, Chen, Agdeppa, Watson & Zabel, 1977; Zabel, Watson, Kagan, Chen & Agdeppa, 1978). The reaction of ethyl 2-formyl-2-phenylpropionate with FSO_3H yielded two compounds. Compound $X1$, m.p. 155–157 °C, comprised 40% of the product while compound $X2$, m.p. 116 °C, comprised 60%. Both compounds gave the same chemical analysis and an m/e peak at 394 which corresponded to $C_{24}H_{26}O_5$. The proton and ^{13}C NMR spectra of compound $X2$ were more complex but contained all the resonances found in $X1$. An X-ray crystal structure analysis of compound $X2$ showed it to be the *trans* isomer diethyl (1*R**,4*S**,5*R**,8*R**)-4,8-dimethyl-2,3;6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene-4,8-dicarboxylate (II). Considerations of steric interactions and interpretation of the proton and ^{13}C NMR spectra led to the assignment of compound $X1$ as probably the *cis* isomer diethyl (1*R**,4*R**,5*R**,8*R**)-4,8-dimethyl-2,3;6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene-4,8-dicarboxylate (III).


(I)



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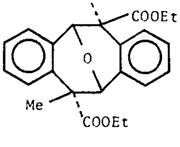

(II)

Experimental

Compound (II) was obtained by synthesis and recrystallization from CCl_4–diisopropyl ether. A crystal of dimensions 0·17 × 0·42 × 0·58 mm was selected for intensity measurements on a Syntex $P2_1$ diffractometer system by the $\theta/2\theta$ scanning technique using a variable scan speed for values of $2\theta \leq 120°$. Room-temperature lattice parameters were refined by a least-squares procedure utilizing 15 reflections whose angles were measured by a centering routine associated with the Syntex diffractometer. Crystal data and experimental parameters are given in Table 1.

5620 independent reflections were measured and Lorentz and polarization corrections were applied. No absorption correction was made. A periodically monitored reflection showed no significant crystal deterioration. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Table 1. *Crystal data and experimental parameters*

$C_{24}H_{26}O_5$, M_r = 394·47, space group $P\bar{1}$, a = 26·486 (10), b = 8·543 (3), c = 9·467 (8) Å, α = 104·38 (5), β = 102·45 (5), γ = 90·35 (3)°, V = 2022 (2) Å^3, Z = 4, d_c = 1·293 g cm^{-3}, μ = 7·4 cm^{-1}, λ(Cu $K\alpha$) = 1·54178 Å, graphite monochromator.

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Structure determination and refinement

The direct-methods program *MULTAN* (Germain, Main & Woolfson, 1971) was used to calculate phases for the 400 $|E|$ values greater than 1·82. The phase set with the largest combined figure of merit was selected, and the E map calculated with these phases revealed the positions of most atoms of the two independent

Table 2. *Positional parameters for diethyl* (1R*,4S*,5R*,8R*)-4,8-*dimethyl*-2,3;6,7-*dibenzo*-9-*oxabicyclo*-[3.3.1]*nona*-2,6-*diene*-4,8-*dicarboxylate*

Nonhydrogen parameters are $\times 10^4$ and hydrogen parameters $\times 10^3$.

	x	*y*	*z*		*x*	*y*	*z*
Molecule *A*				Molecule *B*			
C(1)	922 (1)	7542 (4)	2171 (4)	C(1)	3979 (1)	2350 (4)	63 (4)
C(2)	841 (1)	6062 (4)	867 (4)	C(2)	4114 (1)	1563 (4)	1349 (4)
C(3)	969 (1)	6087 (4)	−477 (4)	C(3)	3974 (1)	2238 (4)	2701 (4)
C(4)	1195 (1)	7662 (4)	−661 (4)	C(4)	3703 (1)	3801 (4)	2929 (4)
C(5)	1380 (1)	8902 (4)	894 (4)	C(5)	3510 (1)	4270 (4)	1423 (4)
C(6)	1883 (1)	8651 (4)	1854 (4)	C(6)	3004 (1)	3436 (4)	458 (4)
C(7)	1896 (1)	7992 (4)	3074 (4)	C(7)	2995 (1)	2123 (4)	−762 (4)
C(8)	1398 (1)	7583 (4)	3479 (4)	C(8)	3495 (1)	1547 (4)	−1191 (4)
O(9)	968 (1)	8988 (3)	1673 (2)	O(9)	3909 (1)	4015 (3)	591 (3)
C(10)	605 (1)	4632 (4)	983 (4)	C(10)	4392 (2)	181 (5)	1193 (5)
C(11)	513 (2)	3283 (4)	−192 (4)	C(11)	4515 (2)	−601 (5)	2339 (5)
C(12)	646 (2)	3316 (4)	−1528 (4)	C(12)	4364 (2)	57 (5)	3672 (5)
C(13)	865 (1)	4730 (4)	−1669 (4)	C(13)	4107 (2)	1448 (4)	3852 (4)
C(14)	2352 (2)	9189 (5)	1626 (4)	C(14)	2542 (2)	4037 (4)	730 (4)
C(15)	2817 (2)	9072 (5)	2537 (5)	C(15)	2071 (2)	3328 (5)	−140 (4)
C(16)	2835 (2)	8400 (5)	3755 (5)	C(16)	2065 (2)	1998 (5)	−1360 (4)
C(17)	2371 (2)	7891 (4)	4010 (4)	C(17)	2523 (2)	1438 (4)	−1653 (4)
C(18)	793 (2)	8469 (4)	−1581 (4)	C(18)	4081 (2)	5191 (4)	3981 (4)
O(19)	316 (1)	7839 (3)	−1878 (3)	O(19)	4254 (1)	4921 (3)	5301 (3)
C(20)	−96 (2)	8568 (5)	−2756 (5)	C(20)	4589 (2)	6205 (5)	6449 (5)
C(21)	−192 (2)	7641 (7)	−4343 (6)	C(21)	4709 (2)	5574 (7)	7834 (5)
O(22)	896 (1)	9629 (3)	−2013 (3)	O(22)	4197 (2)	6383 (3)	3670 (3)
C(23)	1645 (1)	7328 (4)	−1472 (4)	C(23)	3243 (1)	3702 (5)	3691 (4)
C(24)	1391 (2)	5925 (5)	3867 (4)	C(24)	3517 (2)	−288 (5)	−1522 (4)
O(25)	1705 (1)	4913 (3)	3252 (3)	O(25)	3257 (1)	−947 (3)	−733 (3)
C(26)	1684 (2)	3251 (5)	3433 (5)	C(26)	3295 (2)	−2690 (5)	−860 (5)
C(27)	2018 (2)	3140 (5)	4869 (6)	C(27)	2941 (2)	−3663 (5)	−2261 (6)
O(28)	1108 (1)	5631 (3)	4625 (3)	O(28)	3760 (1)	−1059 (4)	−2358 (3)
C(29)	1329 (2)	8916 (5)	4845 (4)	C(29)	3538 (2)	2052 (5)	−2632 (4)
H(1)	57 (1)	773 (3)	258 (3)	H(1)	435 (1)	220 (4)	−38 (4)
H(5)	138 (1)	1005 (4)	70 (3)	H(5)	349 (1)	561 (4)	173 (4)
H(10)	47 (1)	472 (4)	203 (4)	H(10)	450 (1)	−32 (4)	16 (4)
H(11)	37 (2)	211 (5)	−20 (5)	H(11)	473 (1)	−174 (5)	219 (5)
H(12)	62 (1)	221 (4)	−240 (4)	H(12)	448 (2)	−60 (6)	455 (6)
H(13)	93 (1)	479 (4)	−267 (4)	H(13)	406 (1)	195 (4)	497 (4)
H(14)	233 (1)	976 (4)	84 (4)	H(14)	256 (1)	504 (4)	159 (4)
H(15)	318 (1)	930 (4)	240 (4)	H(15)	172 (1)	370 (4)	−1 (4)
H(16)	319 (1)	823 (4)	436 (4)	H(16)	173 (1)	158 (4)	−198 (4)
H(17)	241 (2)	734 (5)	497 (5)	H(17)	254 (1)	47 (4)	−248 (4)
H(20*a*)	−46 (2)	850 (5)	−234 (5)	H(20*a*)	495 (2)	623 (5)	606 (5)
H(20*b*)	0 (2)	979 (6)	−268 (5)	H(20*b*)	438 (2)	727 (5)	654 (5)
H(21*a*)	−27 (2)	624 (7)	−437 (7)	H(21*a*)	491 (2)	652 (6)	871 (6)
H(21*b*)	−51 (2)	811 (6)	−480 (5)	H(21*b*)	436 (2)	558 (6)	815 (6)
H(21*c*)	12 (2)	771 (7)	−468 (6)	H(21*c*)	491 (2)	466 (7)	763 (6)
H(23*a*)	152 (1)	663 (4)	−258 (4)	H(23*a*)	300 (1)	266 (4)	306 (4)
H(23*b*)	182 (1)	836 (4)	−148 (4)	H(23*b*)	338 (1)	342 (4)	464 (4)
H(23*c*)	193 (1)	666 (4)	−96 (4)	H(23*c*)	306 (1)	469 (4)	388 (4)
H(26*a*)	130 (2)	289 (5)	328 (5)	H(26*a*)	372 (2)	−301 (5)	−80 (5)
H(26*b*)	187 (2)	250 (5)	269 (5)	H(26*b*)	318 (2)	−279 (5)	2 (5)
H(27*a*)	205 (2)	196 (5)	487 (5)	H(27*a*)	253 (2)	−316 (6)	−245 (5)
H(27*b*)	240 (2)	358 (6)	509 (5)	H(27*b*)	317 (2)	−375 (6)	−314 (6)
H(27*c*)	186 (2)	380 (5)	567 (5)	H(27*c*)	295 (2)	−476 (5)	−219 (5)
H(29*a*)	103 (1)	877 (4)	514 (4)	H(29*a*)	356 (1)	323 (4)	−238 (4)
H(29*b*)	167 (1)	898 (4)	567 (4)	H(29*b*)	389 (1)	152 (4)	−297 (4)
H(29*c*)	131 (1)	1001 (4)	459 (4)	H(29*c*)	320 (1)	166 (4)	−354 (4)

molecules. Subsequent least-squares refinements and difference Fourier maps led to the location of all atoms, including H. Block-diagonal least-squares refinement utilizing all 5620 reflections led to an R factor of 0·076 where $R = \sum||F_o| - |F_c||/\sum|F_o|$. The function minimized in the least-squares refinement was $\sum w(|F_o| - |F_c|)^2$ where $w = [1/\sigma(F_o)]^2$ was determined from counting statistics. Negative measured intensities were assigned a value of zero in all calculations. H atom thermal parameters were refined isotropically while those of the other atoms were refined anisotropically.

A final difference Fourier map showed no peak larger than 0·3 e Å^{-3}. Atomic positional parameters are given in Table 2 while interatomic distances and angles are presented in Tables 3 and 4.*

Discussion

The two independent molecules of diethyl 4,8-dimethyl-2,3;6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene-4,8-dicarboxylate are identified as *A* and *B* here and in the tables. Fig. 1 is an *ORTEP* drawing (Johnson, 1965) using the coordinates listed for molecule *A*, and the numbering system is the same as that used in the tables. Internal torsion angles are presented in Table 5.

The eight-membered ring exhibits a twisted conformation, intermediate between the normal tub and boat forms. The twist is imposed by the 1,5-epoxy and 2,6-diene moieties. The two six-membered rings exhibit half-chair conformations. The two phenyl rings are essentially planar with interplanar angles of 108 and 107° for the two independent molecules. The conformations of the two molecules are identical except for a twist of the ethoxycarbonyl side chain at C(4). The C(sp^2)=C(sp^2) bond distances average 1·388 (10) Å, the C(sp^2)–C(sp^3) 1·512 (7) Å, the C(sp^3)–C(sp^3) 1·554 (7) Å (excluding the side-chain methyl groups), the C(sp^2)=O 1·202 (9) Å, the C(sp^2)–O 1·319 (7) Å and the C(sp^3)–O 1·436 (8) Å. A better correspondence in bond distances occurs if molecule *B* is renumbered with C(5) designated as C(1); however, several distances still differ by more than 3σ. There are no close intermolecular contacts and the molecules are held together by van der Waals interactions.

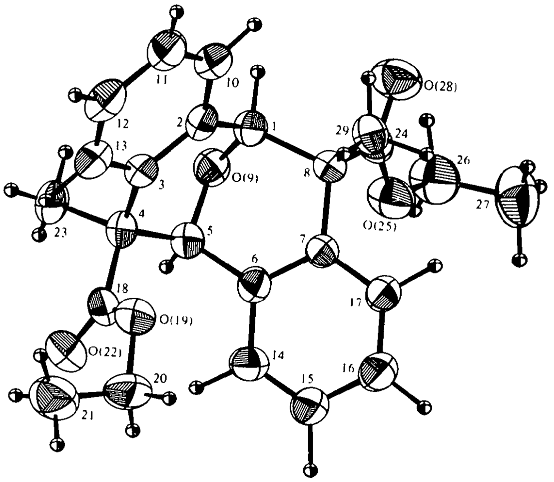

Fig. 1. *ORTEP* drawing of diethyl (1*R**,4*S**,5*R**,8*R**)-4,8-dimethyl-2,3;6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene-4,8-dicarboxylate. Anisotropic thermal parameters are represented at the 50% probability level while hydrogen thermal parameters are spheres of arbitrary size.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33779 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Interatomic distances* (Å) *with standard deviations in parentheses*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(1)–C(2)	1·509 (4)	1·512 (5)	C(8)–C(29)	1·541 (5)	1·555 (6)	C(15)–H(15)	1·03 (4)	1·02 (3)
C(2)–C(3)	1·390 (5)	1·396 (5)	C(8)–C(24)	1·550 (6)	1·524 (5)	C(16)–H(16)	1·03 (3)	0·97 (3)
C(3)–C(4)	1·534 (5)	1·510 (5)	C(24)–O(28)	1·207 (6)	1·201 (5)	C(17)–H(17)	1·12 (5)	1·00 (3)
C(4)–C(5)	1·561 (4)	1·558 (5)	C(24)–O(25)	1·321 (5)	1·331 (6)	C(20)–H(20*a*)	1·12 (5)	1·10 (5)
C(5)–C(6)	1·492 (5)	1·509 (5)	O(25)–C(26)	1·474 (5)	1·470 (5)	C(20)–H(20*b*)	1·06 (5)	1·07 (4)
C(6)–C(7)	1·400 (6)	1·392 (4)	C(26)–C(27)	1·479 (7)	1·495 (6)	C(21)–H(21*a*)	1·21 (6)	1·05 (4)
C(7)–C(8)	1·515 (6)	1·518 (6)	C(4)–C(23)	1·542 (6)	1·556 (6)	C(21)–H(21*b*)	1·00 (5)	1·03 (6)
C(1)–C(8)	1·558 (5)	1·565 (5)	C(4)–C(18)	1·509 (5)	1·541 (5)	C(21)–H(21*c*)	0·94 (7)	0·96 (6)
C(1)–O(9)	1·441 (5)	1·413 (4)	C(18)–O(22)	1·214 (5)	1·184 (5)	C(23)–H(23*a*)	1·05 (3)	1·08 (3)
C(5)–O(9)	1·434 (5)	1·436 (5)	C(18)–O(19)	1·313 (5)	1·313 (5)	C(23)–H(23*b*)	0·99 (4)	0·99 (4)
C(2)–C(10)	1·408 (5)	1·389 (6)	O(19)–C(20)	1·464 (5)	1·467 (4)	C(23)–H(23*c*)	1·07 (3)	0·97 (4)
C(10)–C(11)	1·367 (5)	1·393 (7)	C(20)–C(21)	1·481 (7)	1·511 (7)	C(26)–H(26*a*)	1·04 (4)	1·14 (4)
C(11)–C(12)	1·391 (6)	1·392 (7)	C(1)–H(1)	1·08 (3)	1·15 (4)	C(26)–H(26*b*)	1·04 (5)	0·97 (5)
C(12)–C(13)	1·385 (6)	1·365 (6)	C(5)–H(5)	1·04 (3)	1·12 (3)	C(27)–H(27*a*)	1·02 (5)	1·16 (5)
C(3)–C(13)	1·381 (4)	1·404 (6)	C(10)–H(10)	1·12 (4)	1·06 (4)	C(27)–H(27*b*)	1·03 (5)	1·11 (6)
C(6)–C(14)	1·399 (6)	1·380 (5)	C(11)–H(11)	1·09 (5)	1·13 (4)	C(27)–H(27*c*)	0·98 (5)	0·95 (5)
C(14)–C(15)	1·363 (5)	1·379 (5)	C(12)–H(12)	1·09 (3)	1·11 (6)	C(29)–H(29*a*)	0·92 (4)	0·96 (3)
C(15)–C(16)	1·403 (7)	1·402 (5)	C(13)–H(13)	1·02 (4)	1·08 (4)	C(29)–H(29*b*)	1·05 (4)	1·10 (4)
C(16)–C(17)	1·387 (6)	1·366 (6)	C(14)–H(14)	0·98 (4)	1·02 (3)	C(29)–H(29*c*)	1·02 (3)	1·09 (3)
C(7)–C(17)	1·390 (5)	1·381 (5)						

Table 4. *Interatomic angles* (°)

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(1)C(2)C(3)	122·0 (3)	120·6 (3)	C(16)C(17)C(7)	121·9 (3)	121·9 (3)	O(19)C(20)C(21)	108·8 (4)	105·2 (4)
C(2)C(3)C(4)	119·6 (3)	120·8 (3)	C(17)C(7)C(6)	119·2 (3)	119·0 (3)	O(22)C(18)O(19)	121·7 (3)	123·3 (4)
C(3)C(4)C(5)	110·4 (3)	111·3 (3)	C(3)C(2)C(10)	118·7 (3)	120·0 (3)	C(7)C(8)C(29)	108·3 (3)	108·9 (3)
C(4)C(5)C(6)	117·5 (3)	116·0 (3)	C(2)C(10)C(11)	120·7 (3)	121·3 (4)	C(1)C(8)C(29)	107·9 (3)	107·5 (3)
C(5)C(6)C(7)	120·9 (3)	120·9 (3)	C(10)C(11)C(12)	120·1 (3)	118·5 (4)	C(7)C(8)C(24)	113·1 (3)	112·5 (3)
C(6)C(7)C(8)	120·2 (3)	120·5 (3)	C(11)C(12)C(13)	119·6 (3)	120·5 (4)	C(1)C(8)C(24)	107·7 (3)	109·0 (3)
C(7)C(8)C(1)	111·0 (3)	111·3 (3)	C(12)C(13)C(3)	120·5 (3)	121·7 (4)	C(24)C(8)C(29)	108·7 (3)	107·5 (3)
C(8)C(1)C(2)	116·3 (3)	115·3 (3)	C(2)C(3)C(13)	120·2 (3)	118·0 (3)	C(8)C(24)O(28)	121·0 (3)	123·7 (3)
C(1)O(9)C(5)	110·7 (2)	111·7 (3)	C(3)C(4)C(23)	110·9 (3)	111·5 (3)	C(8)C(24)O(25)	113·3 (3)	113·0 (3)
C(6)C(5)O(9)	111·2 (3)	110·2 (3)	C(5)C(4)C(23)	110·7 (3)	110·1 (3)	C(24)O(25)C(26)	117·0 (3)	117·3 (3)
C(4)C(5)O(9)	107·9 (3)	109·1 (3)	C(5)C(4)C(18)	106·3 (3)	107·4 (3)	O(25)C(26)C(27)	111·4 (3)	110·9 (4)
C(2)C(1)O(9)	110·5 (3)	110·7 (3)	C(3)C(4)C(18)	111·2 (3)	109·9 (3)	O(28)C(24)O(25)	125·7 (3)	123·1 (3)
C(8)C(1)O(9)	107·6 (3)	109·2 (3)	C(23)C(4)C(18)	107·3 (3)	106·5 (3)	C(5)C(6)C(14)	120·6 (3)	119·7 (3)
C(7)C(6)C(14)	118·3 (3)	119·2 (3)	C(4)C(18)O(22)	122·8 (3)	125·0 (4)	C(17)C(7)C(8)	120·1 (3)	120·2 (3)
C(6)C(14)C(15)	122·3 (3)	121·6 (3)	C(4)C(18)O(19)	115·5 (3)	111·7 (3)	C(1)C(2)C(10)	119·1 (3)	119·4 (3)
C(14)C(15)C(16)	119·8 (4)	118·8 (3)	C(18)O(19)C(20)	118·5 (3)	117·4 (4)	C(13)C(3)C(4)	120·0 (3)	121·2 (3)
C(15)C(16)C(17)	118·5 (4)	119·4 (3)						

Table 5. *Torsion angles* (°)

Eight-membered ring

	A	*B*		*A*	*B*
1–2–3–4	0	−2	5–6–7–8	−3	−3
2–3–4–5	15	12	6–7–8–1	16	12
3–4–5–6	78	82	7–8–1–2	77	82
4–5–6–7	−103	−100	8–1–2–3	−103	−101

Six-membered rings

	A	*B*		*A*	*B*
1–2–3–4	0	−2	5–6–7–8	−3	−3
2–3–4–5	15	12	6–7–8–1	16	12
3–4–5–9	−49	−43	7–8–1–9	−48	−43
4–5–9–1	73	70	8–1–9–5	71	70
5–9–1–2	−56	−58	1–9–5–6	−58	−59
9–1–2–3	20	24	9–5–6–7	22	24

Phenyl rings

	A	*B*		*A*	*B*
2–10–11–12	−1	−2	6–14–15–16	0	−2
10–11–12–13	−1	−1	14–15–16–17	−1	0
11–12–13–3	2	2	15–16–17–7	2	1
12–13–3–2	−2	0	16–17–7–6	−1	−1
13–3–2–10	0	−2	17–7–6–14	0	−1
3–2–10–11	1	3	7–6–14–15	1	3

External torsion angles

	A	*B*		*A*	*B*
6–7–8–29	−103	−106	2–3–4–23	138	136
6–7–8–24	137	134	2–3–4–18	−103	−107
2–1–8–29	−165	−159	6–5–4–23	−45	−43
2–1–8–24	−48	−43	6–5–4–18	−162	−158
7–8–24–28	157	152	3–4–18–22	−171	121
7–8–24–25	−25	−31	3–4–18–19	9	−61

The ^{13}C NMR spectra of the *trans*-(1*R**,4*S**,5*R**,8*R**) and *cis*-(1*R**,4*R**,5*R**,8*R**) isomers can be assigned. The resonances are reported as p.p.m. from TMS with atom numbers indicated in parentheses: For the *trans* isomer these are: 175·3 (18), 174·2 (24), 137·0, 133·3 (7, 6), 134·0, 131·2 (3, 2), 129·9, 128·0, 127·6 (17, 13, 14, 10), 126·1, 125·4 (11, 12, 15, 16), 77·3 (20, 26), 61·5 (5), 60·8 (1), 53·5 (8), 50·0 (4), 29·3 (29), 21·7 (23), 14·0 (21, 27); and for the *cis* isomer: 174·4 (18, 24), 136·2, 132·7 (3, 7, 2, 6), 129·4, 127·5 (13, 17, 10, 14), 125·6, 125·5 (11, 12, 15, 16), 77·6 (20, 26), 60·7 (1, 5), 53·3 (4, 8), 29·3 (23, 29) and 13·4 (21, 27).

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