Data collection

AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction: not applied
3649 measured reflections
3649 independent reflections
1786 observed reflections [$I > 3\sigma(I)$]
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = -16 \rightarrow 16$
3 standard reflections monitored every 150 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.0443$
$wR = 0.0494$
$S = 1.627$
1786 reflections
291 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.0003$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: not applied
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)

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

	x	y	z	U_{eq}
Cl1	1.0108 (1)	0.47234 (10)	0.28143 (10)	0.0589
Cl7	0.3397 (1)	0.35103 (9)	0.21595 (9)	0.0500
Cl14	0.6096 (2)	0.20949 (7)	0.23810 (9)	0.0539
N13	0.7972 (4)	0.3454 (2)	0.2547 (2)	0.0360
C1	0.8075 (5)	0.4431 (3)	0.2621 (3)	0.0365
C2	0.7390 (5)	0.4873 (3)	0.1681 (3)	0.0355
C3	0.8260 (6)	0.4707 (4)	0.0758 (4)	0.0547
C4	0.7207 (6)	0.4149 (3)	0.0115 (3)	0.0466
C5	0.5796 (5)	0.4009 (3)	0.0499 (3)	0.0388
C6	0.5716 (5)	0.4498 (3)	0.1463 (3)	0.0308
C7	0.5320 (5)	0.3919 (3)	0.2352 (3)	0.0334
C8	0.5512 (5)	0.4479 (3)	0.3320 (3)	0.0318
C9	0.5184 (6)	0.3960 (3)	0.4227 (3)	0.0401
C10	0.6513 (6)	0.3866 (3)	0.4854 (3)	0.0432
C11	0.7873 (7)	0.4331 (4)	0.4481 (3)	0.0483
C12	0.7256 (5)	0.4745 (3)	0.3490 (3)	0.0349
C14	0.6566 (5)	0.3217 (3)	0.2439 (3)	0.0324
C15	0.4664 (7)	0.3501 (3)	−0.0020 (4)	0.0526
C16	0.503 (1)	0.3127 (4)	−0.0914 (4)	0.0692
C17	0.6441 (9)	0.3290 (4)	−0.1280 (4)	0.0697
C18	0.7529 (8)	0.3793 (4)	−0.0776 (4)	0.0664
C19	0.6473 (8)	0.3392 (4)	0.5724 (4)	0.0615
C20	0.5093 (9)	0.3030 (4)	0.5955 (4)	0.0683
C21	0.3763 (9)	0.3146 (4)	0.5364 (4)	0.0674
C22	0.3787 (7)	0.3624 (4)	0.4493 (4)	0.0533

Table 2. *Selected geometric parameters* (Å, °)

Cl1—C1	1.809 (4)	C1—C12	1.507 (6)
Cl7—C7	1.771 (4)	C2—C6	1.559 (6)
Cl14—C14	1.729 (4)	C6—C7	1.557 (6)
N13—C1	1.468 (6)	C7—C8	1.568 (6)
N13—C14	1.263 (6)	C7—C14	1.503 (6)
C1—C2	1.528 (6)	C8—C12	1.558 (6)
N13—C1—C2	110.9 (3)	C6—C7—C14	104.6 (3)
N13—C1—C12	109.6 (4)	C8—C7—C14	106.2 (3)
C2—C1—C12	110.9 (4)	Cl14—C14—N13	120.0 (3)
C6—C7—C8	110.5 (3)	Cl14—C14—C7	120.8 (3)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *TEXSAN, MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71722 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1062]

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Acta Cryst. (1994). C**50**, 553–556

exo,exo-4,4,12,12,16,16-Hexakis(trifluoromethyl)-17-(3,3,3-trifluoro-2-trifluoromethyl-1-azapropenyl)-3,11,17-triazaheptacyclo[12.4.1.1^{6,9}.0^{2,13}.0^{3,11}.0^{5,10}.0^{15,18}]-icos-7-ene Formed *via* Novel 1,3-Dipolar Cycloaddition to Quadricyclane

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Abstract

The title compound, $C_{26}H_{16}F_{24}N_4$, crystallizes as a racemic mixture with two crystallographically independent molecules in the asymmetric unit, which differ only slightly in conformation. In both cases, the central diazo region bears a close resemblence to the struc-

ture of diethyl *trans*-4,4,8,8-tetrakis(trifluoromethyl)-1,5-diazabicyclo[3.3.0]octane-2,6-dicarboxylate [Burger, Schickaneder, Hein, Gieren, Lamm & Engelhardt (1992). *Liebigs Ann. Chem.* pp. 845-852], in which the N—N bond is bisected by a crystallographic inversion centre.

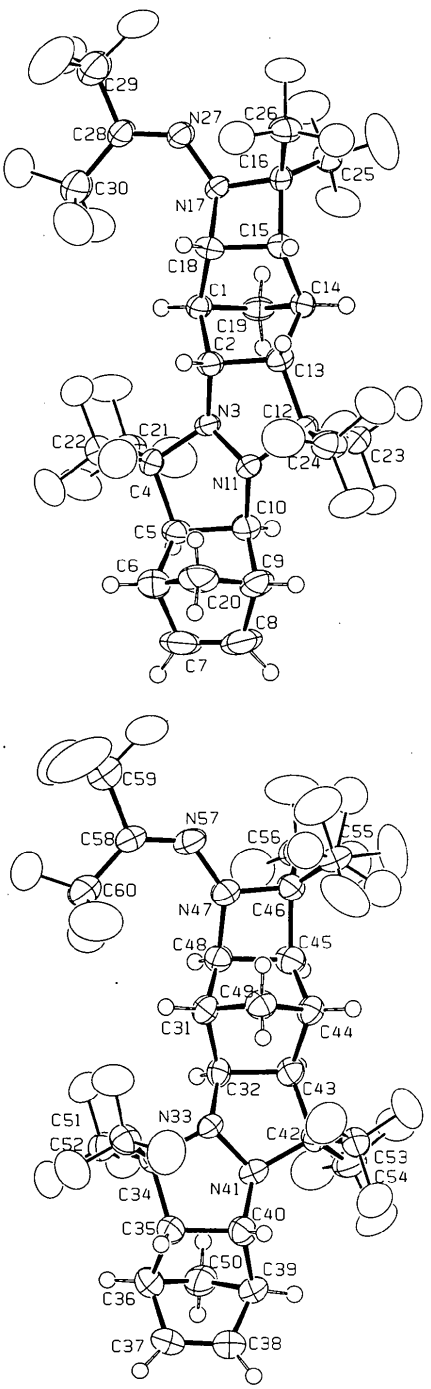
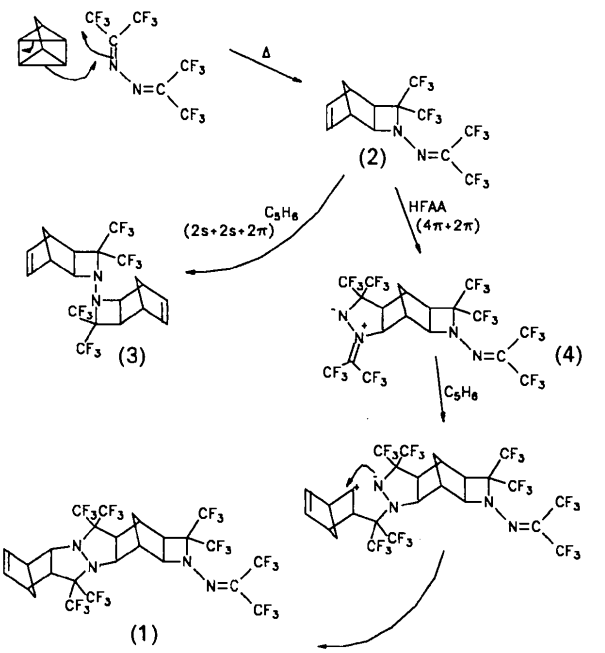


Fig. 1. The two crystallographically independent molecules of the title compound, including atomic numbering scheme, drawn using *ORTEP*II (Johnson, 1976). The F and H labels are based on those of the bonded C atoms.

Comment

The structure determination reported herein forms the basis of an investigation of novel 1,3-dipolar cycloadditions of azines and azomethine imines to quadricyclanes (Barlow, Suliman & Tipping, 1992). Cycloaddition ($\sigma^2s + \sigma^2s + \pi^2s$) of hexafluoroacetone azine (HFAA) to quadricyclane gives the 1:1 adduct (2). This can then undergo further cycloaddition with either (i) quadricyclane ($\sigma^2s + \sigma^2s + \pi^2s$) to give the 1:2 adduct (3) or (ii) the azine ($4\pi + 2\pi$) to afford the 2:1 adduct (4). 1,3-Dipolar cycloaddition of the 2:1 adduct (4) to quadricyclane by a two-step mechanism (a concerted $2s + 2s + 4\pi$ addition is disallowed thermally) then gives the title compound, the 2:2 adduct (1).



Experimental

A mixture of quadricyclane (1.80 g, 19.56 mmol), hexafluoroacetone azine (3.50 g, 10.60 mmol) and dichloromethane (2 ml) was sealed *in vacuo* in a Rotaflo tube (*ca* 50 ml) and heated at 343 K for 2 d. The resulting material was washed from the tube with dichloromethane (10 ml) and the solvent removed (rotary evaporator), thus producing a mixture (4.22 g) of three products as shown by TLC (R_F 0.88, 0.74 and 0.65; eluant hexane). Chromatographic separation (Merck Kieselgel 60; eluant hexane) gave: (i) *exo,exo*-4,4-bis(trifluoromethyl)-3-{4,4-bis(trifluoromethyl)-3-azatricyclo[4.2.1.$0^{2,5}$]non-7-en-3-yl}-3-azatricyclo[4.2.1.$0^{2,5}$]non-7-ene (3) (0.21 g, 0.41 mmol, 8%; found M^+ 512.1115; $C_{20}H_{16}F_{12}N_2$ requires M 512.1008), m.p. 385-386 K; (ii) *exo*-4,4-bis(trifluoromethyl)-3-(3,3,3-trifluoro-2-trifluoromethyl-1-azapropenyl)-3-azatricyclo[4.2.1.$0^{2,5}$]-non-7-ene (2) (2.40 g, 5.71 mmol, 54%; found C 37.2, H 1.9, N 6.9, F 54.8, M^+ 420.0486; $C_{13}H_8F_{12}N_2$ requires C 37.1, H 1.9, N 6.7, F 54.3, M 420.0496), b.p. 392-394 K; and (iii) the title compound *exo,exo*-4,4,12,12,16,16-

hexakis(trifluoromethyl)-17-(3,3,3-trifluoro-2-trifluoromethyl-1-azapropenyl)-3,11,17-triazaheptacyclo[12.4.1.1^{6,9}.0^{2,13}.0^{3,11}.0^{5,10}.0^{15,18}]icos-7-ene (1) (1.38 g, 1.64 mmol, 31%; found C 37.3, H 2.0, F 54.3, M^+ 840; $C_{26}H_{16}F_{24}N_4$ requires C 37.2, H 1.9, N 6.4, F 54.3, M 840), m.p. 413–414 K, which was slowly crystallized from *n*-hexane.

Crystal data

$C_{26}H_{16}F_{24}N_4$
M_r = 840.40
Triclinic
$P\bar{1}$
a = 9.525 (8) Å
b = 12.936 (9) Å
c = 25.831 (9) Å
α = 78.16 (4)°
β = 83.79 (4)°
γ = 72.26 (5)°
V = 2963 (3) Å^3
Z = 4
D_x = 1.883 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 9.59–10.78°
μ = 0.2106 mm^{-1}
T = 296 K
Block
0.40 × 0.35 × 0.35 mm
Colourless

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: not applied
9070 measured reflections
8397 independent reflections
4988 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.035
θ_{max} = 24.97°
$h = 0 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -29 \rightarrow 30$
3 standard reflections
frequency: 2.5 h
intensity variation: none

Refinement

Refinement on F
R = 0.0501
wR = 0.0396
S = 2.111
4988 reflections
973 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max}$ = 0.0672
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
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)

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

	x	y	z	U_{eq}
F21*A*	1.0426 (3)	0.2328 (3)	0.8795 (1)	0.0776
F21*B*	1.0465 (3)	0.2326 (3)	0.7970 (1)	0.0739
F21*C*	0.8576 (4)	0.3317 (3)	0.8340 (1)	0.0755
F22*A*	1.0913 (3)	0.0157 (3)	0.9022 (1)	0.0830
F22*B*	1.0995 (3)	0.0169 (2)	0.81934 (10)	0.0673
F22*C*	0.9506 (3)	−0.0559 (2)	0.8710 (1)	0.0676
F23*A*	0.3533 (3)	0.2585 (2)	0.7511 (1)	0.0679
F23*B*	0.4986 (3)	0.3313 (2)	0.77906 (10)	0.0581
F23*C*	0.3492 (3)	0.2637 (2)	0.83344 (10)	0.0595
F24*A*	0.4090 (3)	0.0400 (3)	0.7732 (1)	0.0728
F24*B*	0.5858 (3)	−0.0551 (3)	0.8226 (1)	0.0644
F24*C*	0.3919 (3)	0.0497 (3)	0.8551 (1)	0.0704
F25*A*	0.8043 (3)	0.3091 (2)	0.5299 (1)	0.0742
F25*B*	0.6049 (4)	0.2666 (2)	0.5459 (1)	0.0930
F25*C*	0.6686 (3)	0.3520 (2)	0.59625 (10)	0.0694
F26*A*	0.6904 (3)	0.0472 (2)	0.56783 (10)	0.0555
F26*B*	0.9154 (3)	−0.0226 (2)	0.58921 (10)	0.0529
F26*C*	0.8637 (3)	0.0963 (2)	0.51805 (10)	0.0558
F29*A*	1.2366 (3)	0.0994 (3)	0.5082 (1)	0.0833
F29*B*	1.3980 (4)	−0.0109 (3)	0.5606 (2)	0.1078
F29*C*	1.3816 (3)	0.1590 (3)	0.5427 (1)	0.0850
F30*A*	1.4009 (4)	0.0766 (3)	0.6474 (1)	0.1074
F30*B*	1.1986 (4)	0.1810 (3)	0.6726 (1)	0.0902
F30*C*	1.2298 (4)	0.0103 (3)	0.6865 (1)	0.1031
N3	0.7876 (4)	0.1551 (3)	0.8129 (1)	0.0306
N11	0.6592 (4)	0.1212 (3)	0.8406 (1)	0.0301
N17	0.9393 (4)	0.1593 (3)	0.6202 (1)	0.0332
N27	1.0602 (5)	0.1374 (3)	0.5872 (1)	0.0373
C1	0.8937 (5)	0.1606 (4)	0.7200 (2)	0.0326
C2	0.8212 (5)	0.0979 (3)	0.7671 (2)	0.0329
C4	0.8876 (5)	0.1396 (4)	0.8546 (2)	0.0357
C5	0.7810 (5)	0.1615 (4)	0.9042 (2)	0.0377
C6	0.7863 (6)	0.0746 (5)	0.9567 (2)	0.0596
C7	0.6885 (8)	0.1469 (5)	0.9936 (2)	0.0723
C8	0.5517 (8)	0.1720 (5)	0.9795 (2)	0.0664
C9	0.5533 (5)	0.1152 (4)	0.9339 (2)	0.0515
C10	0.6253 (5)	0.1775 (3)	0.8863 (2)	0.0390
C12	0.5594 (5)	0.1359 (4)	0.7990 (2)	0.0364
C13	0.6660 (5)	0.1139 (3)	0.7491 (2)	0.0312
C14	0.6599 (4)	0.1978 (3)	0.6962 (2)	0.0295
C15	0.7477 (5)	0.1216 (3)	0.6582 (2)	0.0293
C16	0.7998 (5)	0.1617 (3)	0.6006 (2)	0.0298
C18	0.9076 (5)	0.0984 (3)	0.6748 (2)	0.0334
C19	0.7645 (5)	0.2617 (3)	0.7038 (2)	0.0345
C20	0.6819 (6)	0.0125 (4)	0.9479 (2)	0.0587
C21	0.9583 (6)	0.2351 (5)	0.8407 (2)	0.0528
C22	1.0064 (6)	0.0295 (5)	0.8622 (2)	0.0556
C23	0.4407 (5)	0.2477 (4)	0.7906 (2)	0.0458
C24	0.4847 (6)	0.0428 (5)	0.8132 (2)	0.0517
C25	0.7175 (6)	0.2729 (4)	0.5685 (2)	0.0409
C26	0.8171 (6)	0.0705 (4)	0.5676 (2)	0.0426
C28	1.1945 (7)	0.1061 (4)	0.5989 (2)	0.0421
C29	1.3025 (7)	0.0879 (6)	0.5527 (2)	0.0641
C30	1.2574 (8)	0.0916 (6)	0.6509 (2)	0.0682
F51*A*	1.4144 (4)	0.2158 (2)	0.3048 (1)	0.0696
F51*B*	1.2682 (3)	0.2099 (2)	0.2495 (1)	0.0642
F51*C*	1.2369 (3)	0.1463 (2)	0.3307 (1)	0.0676
F52*A*	1.2188 (3)	0.4268 (2)	0.21972 (10)	0.0539
F52*B*	1.3566 (3)	0.4324 (2)	0.27843 (10)	0.0618
F52*C*	1.1304 (3)	0.5317 (2)	0.27656 (10)	0.0529
F53*A*	0.6185 (4)	0.2719 (3)	0.3606 (1)	0.0814
F53*B*	0.7561 (3)	0.2640 (2)	0.4218 (1)	0.0695
F53*C*	0.8490 (3)	0.1867 (2)	0.3560 (1)	0.0686
F54*A*	0.6540 (3)	0.4725 (3)	0.4115 (1)	0.0754
F54*B*	0.5358 (3)	0.4939 (3)	0.3426 (1)	0.0784
F54*C*	0.7027 (3)	0.5747 (2)	0.3406 (1)	0.0731
F55*A*	0.6307 (6)	0.2239 (4)	0.1069 (2)	0.1126
F55*B*	0.4589 (5)	0.3014 (3)	0.1570 (2)	0.0910
F55*C*	0.6515 (4)	0.1887 (3)	0.1889 (2)	0.0909
F56*A*	0.6437 (4)	0.5468 (3)	0.0947 (1)	0.0814
F56*B*	0.4452 (3)	0.5109 (3)	0.1265 (1)	0.0843
F56*C*	0.5573 (4)	0.4453 (3)	0.0594 (1)	0.1051
F55*A*2	0.5214	0.2874	0.1018	0.0633
F55*B*2	0.5245	0.2669	0.1860	0.0633
F55*C*2	0.7114	0.1801	0.1423	0.0633
F59*A*	0.8965 (5)	0.3243 (4)	−0.0185 (1)	0.1223
F59*B*	1.1150 (5)	0.2260 (3)	−0.0067 (1)	0.1259
F59*C*	1.0663 (4)	0.3960 (3)	−0.0316 (1)	0.1021
F60*A*	1.2457 (4)	0.3355 (4)	0.0472 (1)	0.1305
F60*B*	1.1780 (4)	0.2450 (3)	0.1190 (1)	0.0997
F60*C*	1.1159 (3)	0.4186 (3)	0.1054 (1)	0.0732
N33	1.0215 (4)	0.3390 (3)	0.3016 (1)	0.0333
N41	0.9304 (4)	0.3907 (3)	0.3450 (1)	0.0360
N47	0.8268 (4)	0.3434 (3)	0.1281 (1)	0.0446
N57	0.8730 (5)	0.3358 (3)	0.0783 (2)	0.0442
C31	0.9574 (5)	0.3285 (4)	0.2120 (2)	0.0380
C32	0.9340 (5)	0.3978 (3)	0.2557 (2)	0.0337
C34	1.1724 (5)	0.3408 (3)	0.3076 (2)	0.0319
C35	1.1757 (5)	0.3423 (3)	0.3678 (2)	0.0384
C36	1.2064 (6)	0.4387 (4)	0.3886 (2)	0.0520
C37	1.2333 (7)	0.3890 (5)	0.4462 (2)	0.0645
C38	1.1062 (7)	0.3820 (5)	0.4699 (2)	0.0618
C39	0.9905 (5)	0.4296 (4)	0.4300 (2)	0.0475
C40	1.0191 (5)	0.3453 (4)	0.3920 (2)	0.0396

C42	0.7832 (5)	0.3823 (3)	0.3398 (2)	0.0339
C43	0.7755 (5)	0.4034 (3)	0.2780 (2)	0.0358
C44	0.7292 (5)	0.3295 (3)	0.2472 (2)	0.0371
C45	0.6992 (5)	0.4044 (3)	0.1929 (2)	0.0409
C46	0.6681 (6)	0.3682 (4)	0.1426 (2)	0.0417
C48	0.8563 (5)	0.4004 (3)	0.1686 (2)	0.0381
C49	0.8778 (5)	0.2448 (4)	0.2365 (2)	0.0421
C50	1.0518 (6)	0.5181 (4)	0.3949 (2)	0.0542
C51	1.2733 (7)	0.2283 (4)	0.2975 (2)	0.0451
C52	1.2184 (7)	0.4331 (4)	0.2705 (2)	0.0431
C53	0.7511 (7)	0.2752 (4)	0.3698 (2)	0.0483
C54	0.6690 (7)	0.4826 (5)	0.3590 (2)	0.0538
C55	0.6036 (8)	0.2719 (5)	0.1473 (2)	0.0599
C56	0.5771 (7)	0.4685 (5)	0.1052 (2)	0.0623
C58	0.9997 (7)	0.3342 (4)	0.0571 (2)	0.0413
C59	1.0192 (8)	0.3189 (5)	0.0002 (2)	0.0627
C60	1.1342 (7)	0.3359 (5)	0.0812 (2)	0.0617

Table 2. *Selected geometric parameters* (Å, °)

N3—N11	1.487 (5)	N33—N41	1.482 (5)
N3—C2	1.476 (6)	N33—C32	1.463 (5)
N3—C4	1.458 (6)	N33—C34	1.470 (7)
N11—C10	1.468 (6)	N41—C40	1.470 (6)
N11—C12	1.457 (6)	N41—C42	1.462 (7)
N17—C16	1.462 (7)	N47—C46	1.470 (7)
N17—C18	1.515 (5)	N47—C48	1.491 (7)
C1—C2	1.540 (6)	C31—C32	1.539 (7)
C1—C18	1.524 (7)	C31—C48	1.518 (6)
C1—C19	1.518 (5)	C31—C49	1.505 (7)
C2—C13	1.539 (7)	C32—C43	1.543 (7)
C4—C5	1.564 (6)	C34—C35	1.563 (6)
C5—C6	1.569 (6)	C35—C36	1.570 (8)
C5—C10	1.543 (7)	C35—C40	1.546 (7)
C6—C7	1.514 (8)	C36—C37	1.512 (7)
C6—C20	1.52 (1)	C36—C50	1.531 (7)
C7—C8	1.32 (1)	C37—C38	1.315 (9)
C8—C9	1.507 (9)	C38—C39	1.493 (7)
C9—C10	1.543 (7)	C39—C40	1.557 (7)
C9—C20	1.517 (7)	C39—C50	1.530 (8)
C12—C13	1.571 (6)	C42—C43	1.570 (6)
C13—C14	1.555 (5)	C43—C44	1.544 (8)
C14—C15	1.533 (6)	C44—C45	1.532 (6)
C14—C19	1.525 (7)	C44—C49	1.541 (6)
C15—C16	1.552 (5)	C45—C46	1.554 (7)
C15—C18	1.550 (7)	C45—C48	1.549 (7)
C2—N3—C4	125.7 (3)	C32—N33—C34	124.4 (3)
C10—N11—C12	125.8 (3)	C40—N41—C42	126.2 (3)
N3—C4—C5	103.4 (3)	N33—C34—C35	103.7 (3)
C4—C5—C6	123.7 (3)	C34—C35—C36	122.9 (4)
N11—C12—C13	103.5 (3)	N41—C42—C43	101.2 (3)

One of the cyclobutane CF_3 groups is disordered and has been treated as comprising a major [F55*A*, F55*B*, F55*C*; 81 (1)%] and a minor [F55*A*2, F55*B*2, F55*C*2; 19 (1)%] confomer, with a total occupancy constrained to be 1. The minor conformer has been treated as a rigid group with isotropic vibrational parameters. Data collection: Enraf-Nonius/CAD-4*AJ* diffractometer control software. Cell refinement: Enraf-Nonius/CAD-4*AJ* diffractometer control software. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature search: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71598 (74 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1061]

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endo-(1*R**,9*R**,10*R**)-9,10-Dimethoxy-12,15,15-trimethyltricyclo[9.3.1.$0^{3,8}$]-pentadeca-3(8),11-diene-4,13-dione

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Abstract

The X-ray analysis of the title compound, $C_{20}H_{28}O_4$, revealed a chair-boat-like conformation of the eight-membered ring which makes the whole molecule *endo* and the relative stereochemistry C1*R**, C9*R** and C10*R**.

Comment

A single-crystal X-ray analysis has been performed for the title compound (2), which was obtained in the course of a study towards the synthesis of taxusin (3) (a congener of taxane diterpenes). The synthetic process has been reported elsewhere (Horiguchi, Furukawa & Kuwajima, 1992). Tricyclic compound (2) was formed by the intramolecular cyclization of (1) and has the necessary

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