

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<sub>3</sub> groups is disordered and has been treated as comprising a major [F55A, F55B, F55C; 81 (1)%] and a minor [F55A2, F55B2, F55C2; 19 (1)%] conformer, 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-4AJ diffractometer control software. Cell refinement: Enraf-Nonius/CAD-4AJ diffractometer control software. Data reduction: TEXSAN PROCESS (Molecular Structure Corporation, 1985). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: TEXSAN LS. Molecular graphics: ORTEPII (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-(1R\*,9R\*,10R\*)-9,10-Dimethoxy-12,15,15-trimethyltricyclo[9.3.1.0<sup>3,8</sup>]-pentadeca-3(8),11-diene-4,13-dione**

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## Abstract

The X-ray analysis of the title compound, C<sub>20</sub>H<sub>28</sub>O<sub>4</sub>, revealed a chair-boat-like conformation of the eight-membered ring which makes the whole molecule *endo* and the relative stereochemistry C1R\*, C9R\* and C10R\*.

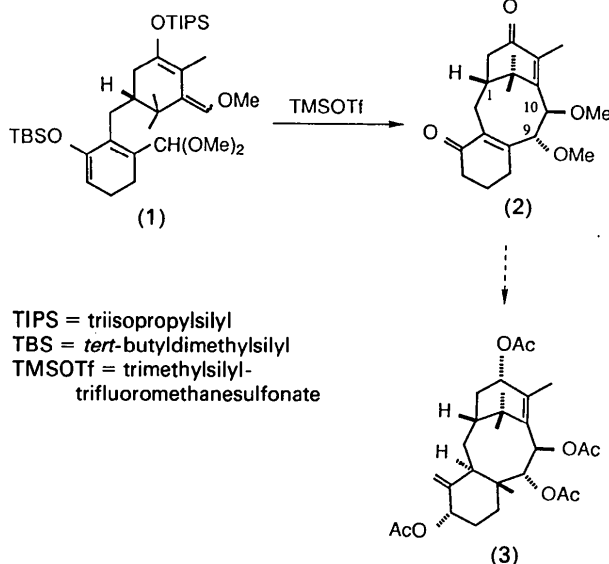
## Comment

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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framework and functional groups for the synthesis of taxusin (3). Although this synthesis has not yet been completed, work is in progress.



TIPS = triisopropylsilyl  
TBS = *tert*-butyldimethylsilyl  
TMSOTf = trimethylsilyl-trifluoromethanesulfonate

Triclinic  
 $P\bar{1}$   
 $a = 8.287 (1) \text{ \AA}$   
 $b = 15.228 (2) \text{ \AA}$   
 $c = 7.679 (1) \text{ \AA}$   
 $\alpha = 104.80 (2)^\circ$   
 $\beta = 109.75 (2)^\circ$   
 $\gamma = 93.23 (2)^\circ$   
 $V = 870.9 (7) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.27 \text{ Mg m}^{-3}$

Cell parameters from 21 reflections  
 $\theta = 10\text{--}15^\circ$   
 $\mu = 0.081 \text{ mm}^{-1}$   
 $T = 296 \text{ K}$   
Plate  
 $0.4 \times 0.35 \times 0.3 \text{ mm}$   
Colourless  
Crystal source: grown from methylene chloride/hexane

#### Data collection

Rigaku AFC-4 diffractometer  
 $\omega$ - $2\theta$  scans  
Absorption correction: none  
3062 measured reflections  
3062 independent reflections  
2111 observed reflections  
 $[I > 3.0\sigma(I)]$

$\theta_{\max} = 25^\circ$   
 $h = -9 \rightarrow 9$   
 $k = 0 \rightarrow 18$   
 $l = -9 \rightarrow 8$   
3 standard reflections monitored every 50 reflections  
intensity variation: 2.3%

#### Refinement

Refinement on  $F$   
 $R = 0.058$   
 $wR = 0.046$   
 $S = 5.15$   
2111 reflections  
329 parameters  
All H-atom parameters refined  
Calculated weights  
 $w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.03$   
 $\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.34 \text{ e \AA}^{-3}$   
Extinction correction: none  
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

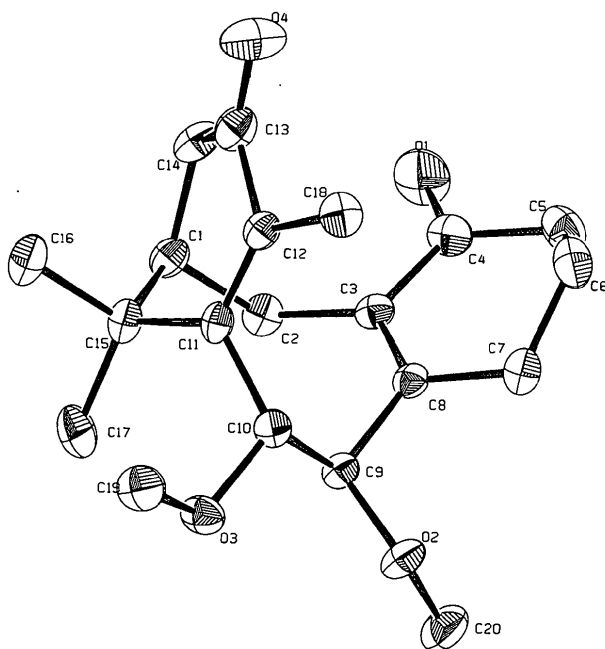


Fig. 1. View of  $\text{C}_{20}\text{H}_{28}\text{O}_4$  showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 30% probability.

## Experimental

### Crystal data

$\text{C}_{20}\text{H}_{28}\text{O}_4$   
 $M_r = 332.44$

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107 \text{ \AA}$

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
O1	1.0965 (4)	0.1198 (2)	0.3749 (4)	0.0784
O2	0.7130 (3)	0.1269 (2)	0.9044 (3)	0.0469
O3	0.5667 (3)	0.2815 (2)	0.8584 (3)	0.0458
O4	1.1523 (4)	0.4412 (2)	0.6975 (4)	0.0809
C1	0.7617 (5)	0.2687 (3)	0.3894 (5)	0.0415
C2	0.7814 (6)	0.1683 (3)	0.3909 (5)	0.0418
C3	0.9214 (5)	0.1521 (2)	0.5629 (5)	0.0352
C4	1.0802 (5)	0.1263 (2)	0.5295 (6)	0.0479
C5	1.2225 (6)	0.1058 (3)	0.6901 (7)	0.0572
C6	1.2245 (6)	0.1553 (3)	0.8880 (7)	0.0578
C7	1.0466 (5)	0.1366 (3)	0.8985 (6)	0.0483
C8	0.9013 (4)	0.1512 (2)	0.7313 (5)	0.0334
C9	0.7352 (5)	0.1686 (2)	0.7667 (5)	0.0350
C10	0.7340 (5)	0.2727 (2)	0.8493 (5)	0.0353
C11	0.7849 (5)	0.3243 (2)	0.7266 (5)	0.0343
C12	0.9494 (5)	0.3665 (2)	0.7909 (5)	0.0357
C13	1.0209 (6)	0.3860 (3)	0.6466 (6)	0.0495
C14	0.9334 (6)	0.3289 (3)	0.4378 (6)	0.0492
C15	0.6644 (5)	0.3160 (2)	0.5210 (5)	0.0417
C16	0.6434 (8)	0.4148 (3)	0.5023 (8)	0.0576
C17	0.4777 (6)	0.2654 (4)	0.4500 (7)	0.0561
C18	1.0784 (6)	0.3935 (3)	0.9958 (6)	0.0479
C19	0.5553 (7)	0.3747 (3)	0.9468 (8)	0.0571
C20	0.6584 (8)	0.0304 (3)	0.8276 (8)	0.0614

Table 2. Selected geometric parameters (Å, °)

O2—C9	1.420 (5)	C1—C15	1.560 (6)
O3—C10	1.423 (5)	C8—C9	1.515 (6)
C1—C2	1.550 (6)	C9—C10	1.552 (5)
C1—C14	1.524 (6)	C10—C11	1.514 (6)
C2—C1—C14	113.3 (4)	C8—C9—C10	111.8 (3)
C2—C1—C15	113.3 (4)	O3—C10—C9	106.9 (3)
C14—C1—C15	110.1 (3)	O3—C10—C11	115.3 (3)
O2—C9—C8	112.1 (3)	C9—C10—C11	109.1 (3)
O2—C9—C10	105.8 (3)		

Data collection: AFC-4 (Rigaku Corporation, 1974). Cell refinement: AFC-4. Data reduction: TEXSAN (Molecular Structure Corporation, 1985). Program(s) used to solve structure: TEXSAN. Program(s) used to refine structure: TEXSAN. Molecular graphics: TEXSAN.

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 71701 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1082]

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## 2,5-Dimethyl-4-nitroimidazole

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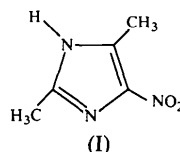
## Abstract

The molecules of the title compound, C<sub>5</sub>H<sub>7</sub>N<sub>3</sub>O<sub>2</sub>, are connected by bifurcated hydrogen bonds and form infinite chains along the *b* direction.

## Comment

Nitroimidazoles are known to be either effective radiosensitizers (Farrell, 1989) or antiprotozoic and an-

tibiotic drugs (Edwards, 1981). This investigation of 2,5-dimethyl-4-nitroimidazole (I) is part of an extensive structure–activity study. Perspective views showing the atomic numbering scheme and molecular packing are given in Figs. 1 and 2. The crystal contains



well ordered molecules of 2,5-dimethyl-4-nitroimidazole in a form also observed in crystals of 4-nitroimidazole (Segalas, Poitras & Beauchamp, 1992; De Bondt, Raglia, Blaton, Peeters & De Ranter, 1993) and 2-methyl-4-nitroimidazole (Kálmán, van Meurs & Toth, 1980). In all these compounds, unsubstituted as well as methyl and dimethyl substituted, the bond lengths and angles of the imidazole ring are in good agreement. The imidazole ring is planar. The maximum deviation from the weighted least-squares plane through non-H atoms is 0.004 (2) Å. As expected, C2—N3 [1.311 (3) Å] shows greater double-bond character than N1—C2 [1.363 (3) Å]. The dihedral angle between the five-membered ring and the nitro group is 177.0 (3)°, and differs from those in the aforementioned structures where the nitro group is coplanar with the ring. This deviation from planarity is caused by hydrogen bonds and molecular crowding in space. The molecules of the title compound, connected by the *b<sub>z</sub>* plane, are linked through bifurcated hydrogen bonds N1—H1···N3<sup>i</sup> [2.25 (3) Å, 167 (2)°] and N1—H1···O401<sup>i</sup> [2.57 (3) Å, 121 (2)°; symmetry code: (i) *x*,  $\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ] and form infinite chains along the *b* direction. In comparison to 4-nitroimidazole and 2-methyl-4-nitroimidazole, the weighted least-squares planes through the neighbouring imidazoles cross at an angle of 133.5 (1)° instead of being coplanar. This deviation from coplanarity is probably caused by steric hindrance due to the 2,5-dimethyl substituents.

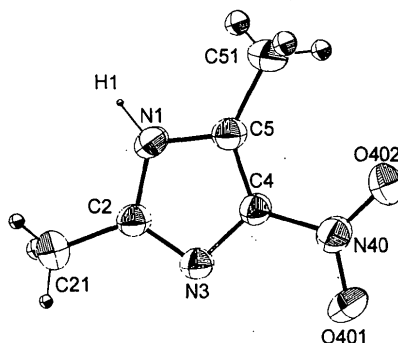


Fig. 1. A perspective view of the molecule with atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.