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(±)-(4a*R*,5*R*,8*S*,8a*R*)-8-(*tert*-Butyldimethylsilyloxy)-2,5,8a-trimethyl-4a,5,8,8a-tetrahydronaphthalene-1,4-dione

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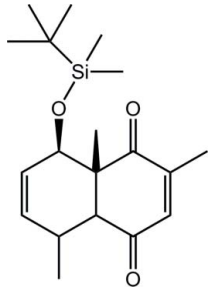
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.062; wR factor = 0.165; data-to-parameter ratio = 19.9.

In the title compound, $C_{19}H_{30}O_3Si$, both rings adopt a half-boat conformation. Overall, the molecule approximates a U-shape as the *cyclo*-2-ene-1,4-dione and butyldimethylsilyloxy substituents lie to the same side of the central cyclohexene ring; the methyl substituent lies to the other side of the molecule. In the crystal, linear supramolecular chains along the *b* axis are sustained by C—H···O interactions.

Related literature

For a general description of the synthesis of higher terpenoids using the Diels–Alder reaction, see: Brocksom *et al.* (2001). For the synthesis of a similar compound containing an N atom in place of the O atom, see: Vieira *et al.* (2007). For the synthesis, see: Finelli (2004). For additional conformational analysis, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{19}H_{30}O_3Si$
M_r = 334.52
Monoclinic, $P2/c$
a = 15.325 (2) Å
b = 7.1744 (9) Å
c = 17.965 (2) Å
β = 93.577 (9)°
V = 1971.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.15 × 0.10 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 MACH 3 diffractometer
4451 measured reflections
4305 independent reflections
1463 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
3 standard reflections every 30 min
intensity decay: 1.4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.165
S = 0.93
4305 reflections
216 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O2^{i}	0.98	2.55	3.524 (5)	171

Symmetry code: (i) $x, y+1, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006) and *MarvinSketch* (ChemAxon, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5287).

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Brocksom, T. J., Correa, A. G., Naves, R. M., Silva, F. Jr, Catani, V., Ceschi, M. A., Zukerman-Schpector, J., Toloi, A. P., Ferreira, M. L. & Brocksom, U. (2001). Diels–Alder Reactions in the Synthesis of Higher Terpenes, in *Organic Synthesis: Theory and Applications*, Vol. 5, edited by T. Hudlicky, pp. 390–87. London: JAI/Elsevier Science.
ChemAxon (2009). *MarvinSketch*. www.chemaxon.com.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Finelli, F. G. (2004). MSc thesis, Universidade Federal de São Carlos, Brazil.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Vieira, Y. W., Nakamura, J., Finelli, F. G., Brocksom, U. & Brocksom, T. J. (2007). *J. Braz. Chem. Soc.* **18**, 448–451.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.