Supporting Information

Title

**Stereoselective Iodine Atom Transfer [3+2] Cycloaddition Reaction with Alkenes
Using Unsymmetrical Allylated Active Methine Radicals**

Authored by

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The Contents of Supporting Information: Experimental procedures and characterization data for products **3a-3c**, **4b,4c**, and X-ray analytical data of **4c** are presented.

Experimental Section

Melting points were uncorrected. ^1H and ^{13}C NMR spectra were recorded on a 300 MHz spectrometer. In ^1H and ^{13}C NMR spectra, chemical shifts were expressed in (ppm) downfield from CHCl_3 (7.26 ppm) and CDCl_3 (77.0 ppm), respectively. Mass spectra were recorded by electron impact or chemical ionization. Column chromatography was performed on silica gel (75-150 μm). Medium-pressure liquid chromatography (MPLC) was performed on a 30 X 4 cm i. d. prepacked column (silica gel, 50 μm) with a UV detector.

General procedure of radical iodine atom transfer cycloaddition of 1 with alkyl-substituted alkenes. Under Ar atmosphere, to a solution of ethyl 1-diethylphosphonyl-2-(iodomethyl)cyclopropane-1-carboxylate **1** (196 mg, 0.5 mmol), 1-hexene (0.13 mL, 1.0 mmol) and $\text{Yb}(\text{OTf})_3$ (310 mg, 0.5 mmol) in CH_2Cl_2 (4 mL) was added Et_3B (0.5 mL, 1M hexane solution) at -15°C . 10 mL of dry air was subsequently introduced with a syringe. After being stirred for 6 h at $-15^\circ - 0^\circ\text{C}$, the mixture was poured into aqueous $\text{Na}_2\text{S}_2\text{O}_3$ solution and extracted with AcOEt. The AcOEt extracts were washed with brine, dried over MgSO_4 , and evaporated to dryness. Purification of the residue by column chromatography (AcOEt) gave a diastereomeric mixture of **3a**, which consisted of four isomers. Further purification by MPLC (0.5% MeOH in AcOEt) gave, in the order of elution, **3a** (131 mg, 55%), one of the diastereomer (9 mg, 4%) and a mixture of the rest two diastereomers (6 mg, 2.7%, diastereomer ratio = 1/1).

(1*R,3*S**,4*R**)-3-butyl-1-diethylphosphonyl-4-(iodomethyl)cyclopentane-1-carboxylic acid ethyl ester (3a).** **3a**: colorless oil; IR (neat) 1730 cm^{-1} ; ^1H -NMR (CDCl_3) : 4.10-4.25 (m, 6H), 3.26 (dd, $J = 5.3, 9.4\text{ Hz}$, 1H), 3.16 (t, $J = 9.4\text{ Hz}$, 1H), 2.67 (td, $J = 6.7, 13.8\text{ Hz}$, 1H), 2.57 (dd, $J = 3.8, 6.5\text{ Hz}$, 1H), 2.44 (m, 1H), 2.13 (ddd, $J = 6.7, 13.8, 17.3\text{ Hz}$, 1H), 1.93 (td, $J = 10.0, 23.4\text{ Hz}$, 1H), 1.92 (m, 1H), 1.22-1.43 (m, 15H), 0.90 (t, $J = 6.7\text{ Hz}$, 3H); ^{13}C -NMR (CDCl_3) : 171.7 (d, $J_{\text{C-P}} = 2.3\text{ Hz}$), 62.9 (d, $J_{\text{C-P}} = 6.3\text{ Hz}$), 62.8 (d, $J_{\text{C-P}} = 6.9\text{ Hz}$), 61.8, 53.1 (d, $J_{\text{C-P}} = 139\text{ Hz}$), 44.9 (d, $J_{\text{C-P}} = 9.2\text{ Hz}$), 42.2 (d, $J_{\text{C-P}} = 10.4\text{ Hz}$), 38.5 (d, $J_{\text{C-P}} = 1.2\text{ Hz}$), 36.8 (d, $J_{\text{C-P}} = 2.3\text{ Hz}$), 30.3, 28.7, 22.7, 16.5, 16.4, 14.0, 9.0; MS (m/z) 497 ($\text{M}^+ + \text{Na}$); Anal. Calcd for $\text{C}_{17}\text{H}_{32}\text{IO}_5\text{P}$: C, 43.05; H, 6.80. Found: C, 43.08; H, 6.99. diastereomer of **3a**: colorless oil; IR (neat) 1728 cm^{-1} ; ^1H -NMR (CDCl_3) : 4.10-4.26 (m, 6H), 3.39 (dd,

$J = 3.5, 10.0$ Hz, 1H), 3.12 (dd, $J = 7.7, 10.0$ Hz, 1H), 2.65 (m, 1H), 2.54 (ddd, $J = 8.2, 13.8, 18.4$ Hz, 1H), 2.13 (m, 1H), 1.96 (ddd, $J = 10.5, 13.2, 19.0$ Hz, 1H), 1.06-1.76 (m, 17H), 0.90 (t, $J = 6.7$ Hz, 3H); MS (m/z) 475 ($M^+ + H^+$); HRMS calcd for $C_{17}H_{33}IO_5P$ ($M^+ + H^+$) 475.1110, found 475.1120.

(Determination of stereochemistry of **3a** by NOE experiment)

cis- and *trans*-3,3-Diethyl-1-diethylphosphonyl-4-(iodomethyl)cyclopentane-1-carboxylic acid ethyl ester (**3b** and *trans*-**3b**). **3b** was prepared from **1** (196 mg, 0.5 mmol) and 2-ethyl-1-butene (0.12 mL, 1 mmol) in accordance with the general procedure for the reaction with alkyl-substituted alkenes. Purification of the residue by column chromatography (AcOEt) gave a mixture of **3b** and *trans*-**3b**. Further purification of the mixture by MPLC (0.5% MeOH in AcOEt) gave **3b** (less polar, 125 mg, 53%) and *trans*-**3b** (more polar, 18mg, 8%). **3b**: colorless oil; IR (neat) 1726 cm^{-1} ; 1H -NMR ($CDCl_3$) : 4.08-4.26 (m, 6H), 3.32 (dd, $J = 2.6, 9.4$ Hz, 1H), 3.11 (dd, $J = 9.4, 11.4$ Hz, 1H), 2.89 (m, 1H), 2.42 (dd, $J = 4.7, 14.1$ Hz, 1H), 2.05-2.25 (m, 3H), 1.23-1.37 (m, 13H), 0.84 (t, $J = 7.3$ Hz, 3H), 0.78 (t, $J = 7.3$ Hz, 3H); ^{13}C -NMR ($CDCl_3$) : 171.5, 63.0 (d, $J_{C-P} = 6.8$ Hz), 62.9 (d, $J_{C-P} = 6.8$ Hz), 61.8, 51.9 (d, $J_{C-P} = 135.2$ Hz), 50.3 (d, $J_{C-P} = 10.3$ Hz), 48.5 (d, $J_{C-P} = 9.2$ Hz), 41.7 (d, $J_{C-P} = 2.9$ Hz), 38.7 (d, $J_{C-P} = 2.3$ Hz), 29.4, 25.3, 16.7, 16.6, 14.3, 8.8, 8.7, 8.6; MS (m/z) 475 ($M^+ + H$); Anal. Calcd for $C_{17}H_{32}IO_5P$: C, 43.05; H, 6.80. Found: C, 43.29; H, 7.05. *trans*-**3b**: colorless oil; IR (neat) 1726 cm^{-1} ; 1H -NMR ($CDCl_3$) : 4.10-4.26 (m, 6H), 3.27 (dd, $J = 2.6, 9.7$ Hz, 1H), 2.92 (dd, $J = 9.7, 11.4$ Hz, 1H), 2.72-2.83 (m, 1H), 2.51 (dd, $J = 5.0, 14.1$ Hz, 1H), 2.16-2.36 (m, 2H), 2.07 (ddd, $J = 0.9, 14.1, 22.5$ Hz, 1H), 1.50 (m, 1H), 1.28-1.36 (m, 10H), 1.00-1.26 (m, 2H), 0.86 (t, $J = 7.3$ Hz, 3H), 0.79 (t, $J = 7.3$ Hz, 3H); ^{13}C -NMR ($CDCl_3$) : 172.0, 63.1 (d, $J_{C-P} = 7.4$ Hz), 63.0 (d, $J_{C-P} = 7.4$ Hz), 61.9, 50.3 (d, $J_{C-P} = 135.2$ Hz), 50.0 (d, $J_{C-P} = 2.9$ Hz), 48.4 (d, $J_{C-P} = 7.4$ Hz), 40.3 (d, $J_{C-P} = 4.0$ Hz), 38.3 (d,

$J_{C-P} = 3.5$ Hz), 28.0, 24.0, 16.7, 16.6, 14.3, 8.8, 8.4, 6.3; MS (m/z) 475 ($M^+ + H$); Anal. Calcd for $C_{17}H_{32}IO_5P$: C, 43.05; H, 6.80. Found: C, 43.42; H, 6.93.

General procedure of radical iodine atom transfer cycloaddition of **1 with ketene acetal.**

Under Ar atmosphere, to a solution of ethyl 1-diethylphosphonyl-2-(iodomethyl)cyclopropane-1-carboxylate **1** (196 mg, 0.5 mmol) and ketene acetal (114 mg, 1.0 mmol) in CH_2Cl_2 (4 mL) was added Et_3B (0.5 mL, 1M hexane solution) at $-15\text{ }^\circ C$. 10 mL of dry air was subsequently introduced with a syringe. After being stirred for 6 h at $-15\text{ }^\circ C$ to $0\text{ }^\circ C$, the mixture was poured into aqueous $Na_2S_2O_3$ solution and extracted with AcOEt. The AcOEt extracts were washed with brine, dried over $MgSO_4$, and evaporated to dryness. Purification of the residue by column chromatography (AcOEt) and subsequent MPLC (0.5% MeOH in AcOEt) gave **3c** (111 mg, 44%).

(2*S,4*R**)-2-Diethylphosphonyl-4-iodomethyl-6,11-dioxaspiro[4.6]undecane-2-carboxylic acid ethyl ester (**3c**).** **3c**: white solid; mp $43\text{ }^\circ C$; IR (neat) 1731 cm^{-1} ; 1H -NMR ($CDCl_3$) : 4.05-4.22 (m, 6H), 3.71 (m, 1H), 3.52-3.64 (m, 2H), 3.45 (m, 1H), 3.29 (dd, $J = 3.5, 9.7$ Hz, 1H), 3.07 (dd, $J = 9.7, 12.3$ Hz, 1H), 2.87 (ddd, $J = 7.3, 7.9, 14.4$ Hz, 1H), 2.73 (d, $J = 13.5$ Hz, 1H), 2.46 (m, 1H), 2.17 (ddd, $J = 3.8, 14.4, 23.1$ Hz, 1H), 2.14 (dd, $J = 13.5, 17.6$ Hz, 1H), 1.48-1.66 (m, 4H), 1.23-1.32 (m, 9H); ^{13}C -NMR ($CDCl_3$) : 170.9 (d, $J_{C-P} = 2.9$ Hz), 110.2 (d, $J_{C-P} = 15.4$ Hz), 64.7, 63.1 (d, $J_{C-P} = 6.8$ Hz), 63.0, 63.0 (d, $J_{C-P} = 6.8$ Hz), 61.7, 49.6 (d, $J_{C-P} = 136.8$ Hz), 48.3 (d, $J_{C-P} = 5.7$ Hz), 38.2 (d, $J_{C-P} = 2.9$ Hz), 35.9 (d, $J_{C-P} = 1.7$ Hz), 29.5, 29.0, 16.4, 16.3, 14.0, 8.8; MS (m/z) 527 ($M^+ + Na$); Anal. Calcd for $C_{17}H_{30}IO_7P$: C, 40.49; H, 6.00. Found: C, 40.71; H, 6.23.

(Determination of stereochemistry of **3c** by NOE experiment)

***cis*- and *trans*-3,3-Diethyl-4-iodomethyl-1-(phenylsulfonyl)cyclopentane-1-carboxylic acid methyl ester (4b and *trans*-4b).** **4b** and *trans*-**4b** were prepared from **2** (380 mg, 1.0 mmol) and 2-ethyl-1-butene (0.24 mL, 2 mmol) in accordance with the general procedure for the reaction with alkyl-substituted alkenes. Purification of the residue by column chromatography (hexane/AcOEt = 5) and subsequent MPLC (hexane/AcOEt = 5) gave **4b** (less polar, 330 mg, 71%) and *trans*-**4b** (more polar, 12.5 mg, 3%). **4b**: colorless oil; IR (neat) 1736 cm⁻¹; ¹H-NMR (CDCl₃) : 7.81 (d, *J* = 7.3 Hz, 2H), 7.68 (t, *J* = 7.3 Hz, 1H), 7.58 (t, *J* = 7.3 Hz, 2H), 3.65 (s, 3H), 3.34 (dd, *J* = 3.5, 9.7 Hz, 1H), 3.18 (dd, *J* = 9.7, 11.7 Hz, 1H), 2.84 (dd, *J* = 6.5, 14.1 Hz, 1H), 2.49 (d, *J* = 14.1 Hz, 1H), 2.41 (d, *J* = 14.1 Hz, 1H), 2.39-2.51 (m, 1H), 2.18 (m, 1H), 1.40 (q, *J* = 7.3 Hz, 2H), 1.24 (q, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.3 Hz, 3H), 0.73 (t, *J* = 7.3 Hz, 3H); ¹³C-NMR (CDCl₃) : 169.2, 136.6, 134.2, 129.7, 128.8, 76.8, 53.3, 49.7, 48.5, 40.5, 38.1, 29.1, 25.1, 8.4, 8.2, 7.7; MS (*m/z*) 465 (M⁺+H⁺); Anal. Calcd for C₁₈H₂₅ISO₄: C, 46.56; H, 5.43. Found: C, 46.83; H, 5.62. *trans*-**4b**: colorless oil; IR (neat) 1734 cm⁻¹; ¹H-NMR (CDCl₃) : 7.84 (d, *J* = 7.3 Hz, 2H), 7.69 (t, *J* = 7.3 Hz, 1H), 7.57 (t, *J* = 7.3 Hz, 2H), 3.68 (s, 3H), 3.24 (dd, *J* = 3.4, 9.4 Hz, 1H), 3.02 (dd, *J* = 7.0, 14.4 Hz, 1H), 2.86 (dd, *J* = 9.4, 11.4 Hz, 1H), 2.53 (d, *J* = 14.4 Hz, 1H), 2.42 (m, 1H), 2.39 (d, *J* = 14.4 Hz, 1H), 2.30 (dd, *J* = 11.4, 14.4 Hz, 1H), 1.54 (m, 1H), 1.36 (m, 1H), 1.18 (m, 1H), 0.99 (m, 1H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.75 (t, *J* = 7.3 Hz, 3H); MS (*m/z*) 465 (M⁺+H⁺); HRMS calcd for C₁₈H₂₅ISO₄(M+Na⁺): 487.0416, found 487.0430.

(2*S,4*R**)-2-Phenylsulfonyl-4-iodomethyl-6,11-dioxaspiro[4.6]undecane-2-carboxylic acid methyl ester (4c).** **4c** was prepared from **2** (114 mg, 0.3 mmol) and ketene acetal (68 mg, 0.6 mmol) in accordance with the general procedure for the reaction with ketene acetal. Purification of the residue by recrystallization (CCl₄/CHCl₃ = 1) gave **4c** (73 mg, 49%). **4c**: white solid; mp 98 °C; IR (KBr) 1738 cm⁻¹; ¹H-NMR (CDCl₃) : 7.81 (d, *J* = 7.3 Hz, 2H), 7.69 (t, *J* = 7.3 Hz, 1H), 7.56 (t, *J* = 7.3 Hz, 2H), 3.73 (m, 1H), 3.66 (s, 3H), 3.62 (m, 1H), 3.40-3.51 (m, 2H), 3.34 (dd, *J* = 3.5, 10.0 Hz, 1H), 3.11 (dd, *J* = 10.0, 12.3 Hz, 1H), 2.87 (dd, *J* = 8.3, 15.1 Hz, 1H), 2.72 (d, *J* = 13.5 Hz, 1H), 2.46-2.59 (m, 2H), 2.46 (d, *J* = 13.5 Hz, 1H), 1.52-1.64 (m, 4H); ¹³C-NMR (CDCl₃) : 168.3, 136.1, 134.3, 129.9, 128.9, 109.4, 74.5, 64.9, 63.1, 53.4, 48.5, 37.9, 35.3, 29.4, 28.9, 7.9; MS (*m/z*) 517 (M⁺+Na); Anal. Calcd for C₁₈H₂₃ISO₆: C, 43.73; H, 4.69. Found: C, 43.46; H, 4.74.

Comment

The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All diagrams and calculations were performed using maXus (Bruker Nonius, Delft & MacScience, Japan).

Experimental

Crystal data

$C_{18}H_{23}IO_6S$
 $C_{18}H_{23}IO_6S$
 $M_r = 494.344$

monoclinic

$P2_1/c$

$a = 10.05800 (10) \text{ \AA}$

$b = 11.66300 (10) \text{ \AA}$

$c = 17.0720 (2) \text{ \AA}$

$= 90.00^\circ$

$= 102.8270 (10)^\circ$

$= 90.00^\circ$

$V = 1952.68 (4) \text{ \AA}^3$

$Z = 4$

$D_x = 1.682 \text{ Mg m}^{-3}$

Density measured by: not measured
 fine-focus sealed tube

Mo K radiation

$= 0.71073$

Cell parameters from 4363

$= 0-27.08^\circ$

$\mu = 1.778 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Prism

$0.40 \times 0.40 \times 0.30 \text{ mm}$

Colourless

Crystal source: Local laboratory

Data collection

DIP Image plate

IP

Absorption correction: multi-scan

$T_{\min} = 0.554$, $T_{\max} = 0.593$

4363 measured reflections

4160 independent reflections

3800 observed reflections

Criterion: $>2\sigma(I)$

$R_{\text{int}} = 0.023$

$_{\max} = 27.08^\circ$

$h = -12 \quad 12$

$k = -14 \quad 0$

$l = -21 \quad 0$

Refinement

Refinement on F^2

fullmatrix least squares refinement

$R(\text{all}) = 0.0425$

$R(\text{gt}) = 0.0386$

$wR(\text{ref}) = 0.1440$

$wR(\text{gt}) = 0.1176$

$S(\text{ref}) = 1.193$

4160 reflections

237 parameters

0 restraints

H-atom parameters not refined

Calculated weights calc

$w_{\max} = 0.002$

$w_{\max} = 1.715 \text{e}\text{\AA}^3$

$w_{\min} = -2.024 \text{e}\text{\AA}^3$

Extinction correction: *SHELXL*

$F_c^* = k F_c [1 + 0.001 x F_c^2 / \sin(2\theta)]^{-1/4}$

Extinction coefficient = 0.0260 (18)

Atomic scattering factors from
International Tables Vol C Tables
4.2.6.8 and 6.1.1.4

Data collection: DIP Image plate

Cell refinement: Scalepack(HKL)

Data reduction: maXus (Mackay et al., 1999)

Program(s) used to refine structure: *SHELXL-97* (Sheldrick, 1997)

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

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

	x	y	z	U_{eq}	Occ
C1	-0.2327 (3)	-0.1120 (3)	0.85087 (19)	0.0156 (6)	1
C2	-0.1926 (3)	0.0054 (3)	0.82104 (18)	0.0147 (6)	1
C3	-0.0682 (3)	0.0005 (3)	0.78233 (19)	0.0168 (6)	1
C4	0.0500 (3)	0.0225 (3)	0.85385 (18)	0.0148 (6)	1
C5	-0.0006 (3)	0.1213 (2)	0.9009 (2)	0.0158 (6)	1
C6	-0.1517 (3)	0.0903 (3)	0.89427 (18)	0.0160 (6)	1
C7	0.0189 (3)	0.2360 (3)	0.8619 (2)	0.0202 (7)	1
C8	0.2949 (3)	0.0447 (3)	0.8847 (2)	0.0211 (7)	1
C9	0.3608 (3)	-0.0733 (3)	0.8883 (2)	0.0222 (7)	1
C10	0.2858 (3)	-0.1665 (3)	0.9247 (2)	0.0212 (7)	1
C11	0.1339 (3)	-0.1713 (3)	0.8860 (2)	0.0192 (6)	1
C12	-0.2474 (4)	-0.3112 (3)	0.8276 (2)	0.0288 (8)	1
C13	-0.3755 (3)	-0.0235 (3)	0.6678 (2)	0.0197 (7)	1
C14	-0.3093 (3)	-0.0048 (3)	0.6049 (2)	0.0206 (7)	1
C15	-0.3359 (4)	-0.0787 (3)	0.5390 (2)	0.0253 (7)	1
C16	-0.4266 (4)	-0.1694 (3)	0.5364 (3)	0.0297 (8)	1
C17	-0.4938 (4)	-0.1855 (3)	0.5978 (3)	0.0294 (8)	1
C18	-0.4689 (4)	-0.1135 (3)	0.6644 (2)	0.0257 (8)	1
I19	-0.05395 (2)	0.377739 (18)	0.922930 (14)	0.02523 (17)	1
O20	-0.2789 (3)	-0.12430 (19)	0.90972 (17)	0.0224 (6)	1
O21	-0.2121 (2)	-0.1974 (2)	0.80233	0.0194 (5)	1

O22	0.1669 (2)	0.0439 (2)	(14) 0.82545	0.0172 (5)	1
O23	0.0652 (2)	-0.07169 (19)	(13) 0.90834	0.0180 (5)	1
O24	-0.2966 (3)	0.1770 (2)	(14) 0.72786	0.0219 (5)	1
O25	-0.4531 (2)	0.0631 (2)	(15) 0.79124	0.0247 (5)	1
S26	-0.33989 (7)	0.06693 (7)	(15) 0.75241	0.0171 (2)	1
H3A	-0.0600	-0.0756	0.7581	0.020	1
H3B	-0.0733	0.0605	0.7407	0.020	1
H5	0.0499	0.1206	0.9583	0.019	1
H6A	-0.2090	0.1602	0.8849	0.019	1
H6B	-0.1639	0.0532	0.9443	0.019	1
H7A	-0.0311	0.2349	0.8049	0.024	1
H7B	0.1169	0.2473	0.8631	0.024	1
H8A	0.2784	0.0649	0.9380	0.025	1
H8B	0.3567	0.1030	0.8700	0.025	1
H9A	0.4555	-0.0678	0.9204	0.027	1
H9B	0.3653	-0.0966	0.8332	0.027	1
H10A	0.3274	-0.2418	0.9181	0.025	1
H10B	0.2977	-0.1519	0.9830	0.025	1
H11A	0.1209	-0.1743	0.8269	0.023	1
H11B	0.0938	-0.2416	0.9037	0.023	1
H12A	-0.2005	-0.3243	0.8836	0.043	1
H12B	-0.2193	-0.3696	0.7933	0.043	1
H12C	-0.3462	-0.3158	0.8227	0.043	1
H14	-0.2474	0.0573	0.6071	0.025	1
H15	-0.2919	-0.0671	0.4958	0.030	1
H16	-0.4423	-0.2207	0.4920	0.036	1
H17	-0.5577	-0.2463	0.5945	0.035	1
H18	-0.5146	-0.1251	0.7069	0.031	1

Table 2. Anisotropic displacement parameters ($\text{\AA}^2$)

	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	U_{33}
C1	0.0145 (14)	-0.0006 (11)	-0.0003 (11)	0.0173 (16)	-0.0013 (11)	0.0134 (14)
C2	0.0136 (13)	-0.0008 (11)	0.0008 (10)	0.0177 (15)	-0.0004 (11)	0.0117 (14)
C3	0.0150	0.0004	0.0073	0.0178	-0.0023	0.0193

	(13)	(11)	(11)	(15)	(12)	(15)
C4	0.0162	-0.0008	0.0057	0.0137	0.0009	0.0154
	(13)	(11)	(11)	(14)	(11)	(14)
C5	0.0144	0.0000	0.0070	0.0149	-0.0017	0.0195
	(14)	(10)	(12)	(16)	(11)	(15)
C6	0.0157	0.0016	0.0032	0.0187	-0.0005	0.0137
	(14)	(12)	(11)	(15)	(12)	(14)
C7	0.0232	-0.0017	0.0091	0.0147	-0.0027	0.0245
	(15)	(12)	(12)	(15)	(12)	(16)
C8	0.0121	-0.0040	0.0028	0.0238	-0.0060	0.0267
	(13)	(12)	(12)	(17)	(13)	(17)
C9	0.0134	0.0029	0.0058	0.0269	-0.0030	0.0270
	(14)	(13)	(12)	(19)	(14)	(17)
C10	0.0194	0.0058	0.0076	0.0212	-0.0013	0.0242
	(15)	(13)	(13)	(17)	(13)	(16)
C11	0.0201	0.0028	0.0085	0.0162	0.0007	0.0231
	(15)	(12)	(12)	(15)	(12)	(16)
C12	0.043 (2)	-0.0058	0.0158	0.0152	-0.0004	0.0317
		(15)	(16)	(16)	(14)	(19)
C13	0.0156	0.0041	0.0030	0.0212	0.0046	0.0219
	(14)	(12)	(12)	(16)	(12)	(16)
C14	0.0174	-0.0003	0.0032	0.0200	-0.0001	0.0237
	(14)	(12)	(12)	(16)	(13)	(17)
C15	0.0207	0.0025	0.0051	0.032 (2)	-0.0026	0.0233
	(15)	(15)	(13)		(15)	(17)
C16	0.0242	0.0028	0.0016	0.0249	-0.0088	0.038 (2)
	(17)	(15)	(15)	(19)	(16)	
C17	0.0226	-0.0061	-0.0013	0.0268	-0.0037	0.035 (2)
	(17)	(14)	(15)	(18)	(16)	
C18	0.0205	-0.0035	0.0032	0.028 (2)	0.0026	0.0276
	(17)	(13)	(14)		(14)	(19)
I19	0.0280	0.00066	0.01283	0.0164	-0.00339	0.0340
	(2)	(7)	(13)	(2)	(8)	(2)
O20	0.0270	-0.0017	0.0134	0.0206	0.0003	0.0231
	(13)	(9)	(10)	(13)	(9)	(13)
O21	0.0259	-0.0013	0.0050	0.0154	-0.0017	0.0170
	(11)	(9)	(9)	(11)	(9)	(11)
O22	0.0140	0.0006	0.0034	0.0236	0.0008	0.0141
	(10)	(9)	(8)	(12)	(9)	(10)
O23	0.0190	0.0043	0.0081	0.0173	0.0018	0.0195
	(10)	(9)	(8)	(12)	(9)	(11)
O24	0.0270	0.0010	0.0030	0.0163	0.0019	0.0213
	(12)	(9)	(9)	(11)	(9)	(12)

O25	0.0176 (11)	0.0061 (10)	0.0110 (9)	0.0313 (14)	0.0019 (10)	0.0281 (13)
S26	0.0153 (4)	0.0030 (3)	0.0057 (3)	0.0175 (4)	0.0015 (3)	0.0194 (4)

Table 3 . *Geometric parameters* (Å, °)

C1—O20	1.206 (4)	O24—S26	1.447 (3)
C1—O21	1.340 (4)	O25—S26	1.440 (2)
C1—C2	1.545 (4)	C3—H3A	0.9900
C2—C3	1.541 (4)	C3—H3B	0.9900
C2—C6	1.577 (4)	C5—H5	1.0000
C2—S26	1.820 (3)	C6—H6A	0.9900
C3—C4	1.525 (4)	C6—H6B	0.9900
C4—O22	1.391 (3)	C7—H7A	0.9900
C4—O23	1.426 (4)	C7—H7B	0.9900
C4—C5	1.553 (4)	C8—H8A	0.9900
C5—C7	1.527 (4)	C8—H8B	0.9900
C5—C6	1.541 (4)	C9—H9A	0.9900
C7—H19	2.167 (3)	C9—H9B	0.9900
C8—O22	1.450 (4)	C10—H10A	0.9900
C8—C9	1.523 (5)	C10—H10B	0.9900
C9—C10	1.531 (5)	C11—H11A	0.9900
C10—C11	1.525 (4)	C11—H11B	0.9900
C11—O23	1.445 (4)	C12—H12A	0.9800
C12—O21	1.463 (4)	C12—H12B	0.9800
C13—C14	1.400 (5)	C12—H12C	0.9800
C13—C18	1.401 (5)	C14—H14	0.9500
C13—S26	1.760 (4)	C15—H15	0.9500
C14—C15	1.396 (5)	C16—H16	0.9500
C15—C16	1.391 (6)	C17—H17	0.9500
C16—C17	1.381 (6)	C18—H18	0.9500
C17—C18	1.391 (6)		
O20—C1—O21	124.6 (3)	C4—C3—C2	102.3 (2)
O20—C1—C2	123.8 (3)	O22—C4—O23	112.9 (2)
O21—C1—C2	111.5 (3)	O22—C4—C3	108.7 (2)
C3—C2—C1	113.9 (3)	O23—C4—C3	110.2 (2)
C3—C2—C6	105.6 (2)	O22—C4—C5	116.5 (3)
C1—C2—C6	109.7 (2)	O23—C4—C5	103.6 (2)
C3—C2—S26	111.2 (2)	C3—C4—C5	104.6 (2)
C1—C2—S26	109.1 (2)	C7—C5—C6	113.2 (3)
C6—C2—S26	107.1 (2)	C7—C5—C4	109.7 (3)

C6—C5—C4	102.7 (2)	O22—C8—H8A	109.8
C5—C6—C2	106.5 (2)	C9—C8—H8A	109.8
C5—C7—I19	111.6 (2)	O22—C8—H8B	109.8
O22—C8—C9	109.5 (3)	C9—C8—H8B	109.8
C8—C9—C10	114.1 (3)	H8A—C8—H8B	108.2
C11—C10—C9	112.8 (3)	C8—C9—H9A	108.7
O23—C11—C10	110.4 (3)	C10—C9—H9A	108.7
C14—C13—C18	120.9 (3)	C8—C9—H9B	108.7
C14—C13—S26	119.4 (3)	C10—C9—H9B	108.7
C18—C13—S26	119.7 (3)	H9A—C9—H9B	107.6
C15—C14—C13	118.9 (3)	C11—C10—H10A	109.0
C16—C15—C14	120.2 (3)	C9—C10—H10A	109.0
C17—C16—C15	120.4 (4)	C11—C10—H10B	109.0
C16—C17—C18	120.6 (3)	C9—C10—H10B	109.0
C17—C18—C13	118.9 (4)	H10A—C10—H10B	107.8
C1—O21—C12	114.2 (3)	B	
C4—O22—C8	116.5 (2)	O23—C11—H11A	109.6
C4—O23—C11	115.8 (2)	C10—C11—H11A	109.6
O25—S26—O24	118.72 (16)	O23—C11—H11B	109.6
O25—S26—C13	108.76 (16)	C10—C11—H11B	109.6
O24—S26—C13	108.28 (15)	H11A—C11—H11B	108.1
O25—S26—C2	107.76 (15)	B	
O24—S26—C2	106.39 (15)	O21—C12—H12A	109.5
C13—S26—C2	106.25 (15)	O21—C12—H12B	109.5
C4—C3—H3A	111.3	H12A—C12—H12B	109.5
C2—C3—H3A	111.3	B	
C4—C3—H3B	111.3	O21—C12—H12C	109.5
C2—C3—H3B	111.3	H12A—C12—H12C	109.5
H3A—C3—H3B	109.2	C	
C7—C5—H5	110.4	H12B—C12—H12C	109.5
C6—C5—H5	110.4	C	
C4—C5—H5	110.4	C15—C14—H14	120.6
C5—C6—H6A	110.4	C13—C14—H14	120.6
C2—C6—H6A	110.4	C16—C15—H15	119.9
C5—C6—H6B	110.4	C14—C15—H15	119.9
C2—C6—H6B	110.4	C17—C16—H16	119.8
H6A—C6—H6B	108.6	C15—C16—H16	119.8
C5—C7—H7A	109.3	C16—C17—H17	119.7
I19—C7—H7A	109.3	C18—C17—H17	119.7
C5—C7—H7B	109.3	C17—C18—H18	120.6
I19—C7—H7B	109.3	C13—C18—H18	120.6
H7A—C7—H7B	108.0		

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