

Supplementary Information

“A Diastereoselective Metal Catalyzed [2+2]Cycloaddition of bis-Enones”

JA010800P

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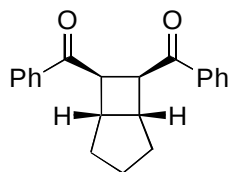
Austin, TX 78712

USA

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I. [2+2] Cycloaddition Products:



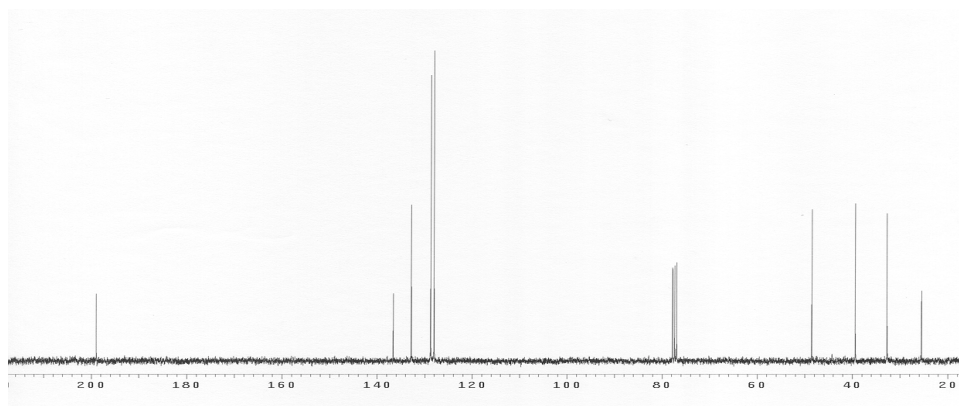
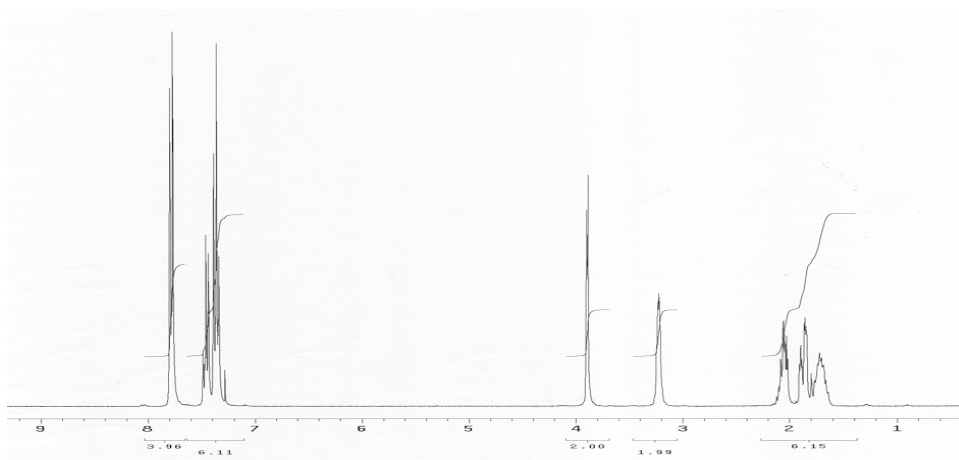
¹H NMR: (300 MHz, CDCl₃) 1.84 (m, 4H), 2.06 (m, 2H), 3.22 (m, 2H), 3.90 (d, *J* 4.2Hz, 2H), 7.42 (m, 6H), 7.78 (m, 4H).

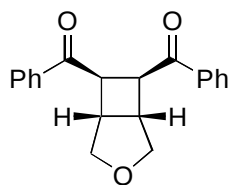
¹³C NMR: (75 MHz, CDCl₃) 25.5, 32.7, 39.3, 48.6, 128.1, 128.7, 136.6, 198.9.

HRMS: Calc'd for C₂₁H₂₁O₂: 305.1541, Found: 305.1538.

FTIR: (KBr) 3054, 2968, 1682, 1447, 1263, 743, 704 cm⁻¹.

m.p.: 155 ~157 °C





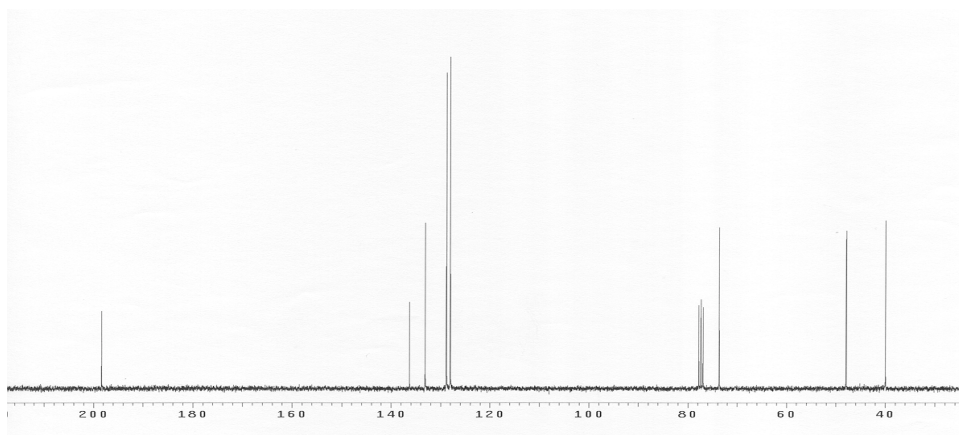
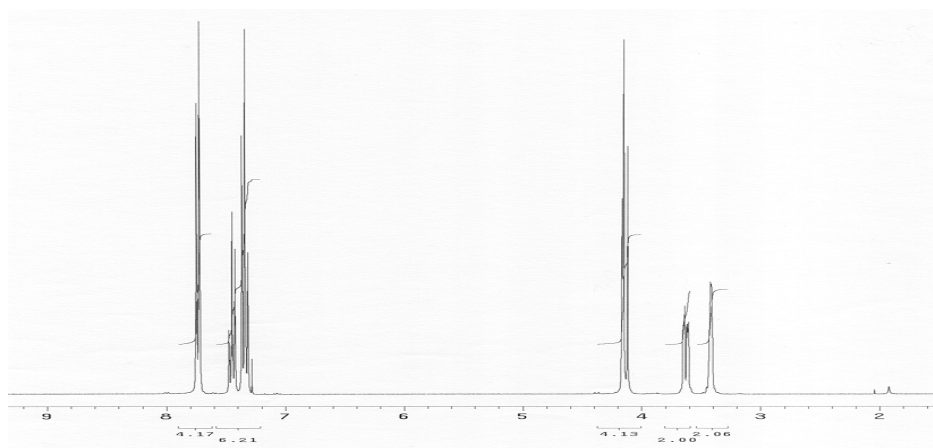
¹H NMR: (300 MHz, CDCl₃) 3.42 (m, 2H), 3.64 (m, 2H), 4.16 (m, 4H), 7.48 (m, 6H), 7.92 (m, 4H).

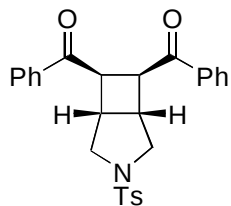
¹³C NMR: (75 MHz, CDCl₃) 39.9, 47.9, 73.7, 128.0, 128.8, 133.1, 136.2, 198.3.

HRMS: Calc'd for C₂₀H₁₉O₃: 307.1334, Found: 307.1336.

FTIR: (KBr) 3055, 2858, 1685, 1448, 1265, 738, 704 cm⁻¹.

m.p.: 163 ~ 165 °C





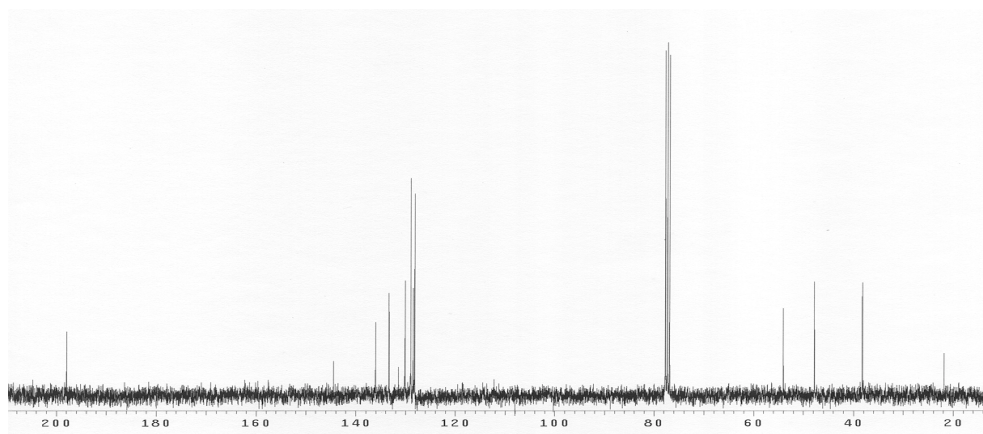
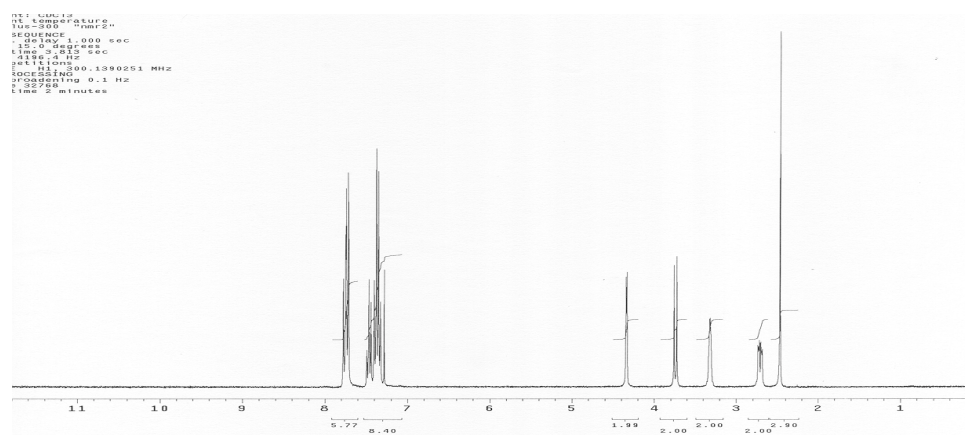
¹H NMR: (300 MHz, CDCl₃) 2.47 (s, 3H), 2.71 (m, 2H), 3.31 (m, 2H), 3.73 (d, *J* 1.8, 2H), 4.33 (d, *J* 4.2, 2H), 7.42 (m, 8H), 7.70 (m, 6H).

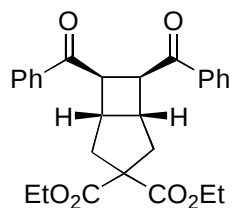
¹³C NMR: (75 MHz, CDCl₃) 21.8, 38.2, 47.7, 54.0, 128.1, 128.4, 128.8, 130.0, 131.3, 133.3, 136.0, 144.4, 197.9.

HRMS: Calc'd for C₂₇H₂₆NO₄S: 460.1582, Found: 460.1587.

FTIR: (KBr) 3056, 2985, 1685, 1346, 1265, 1163, 734, 704, 665 cm⁻¹.

m.p.: 170 ~172 °C





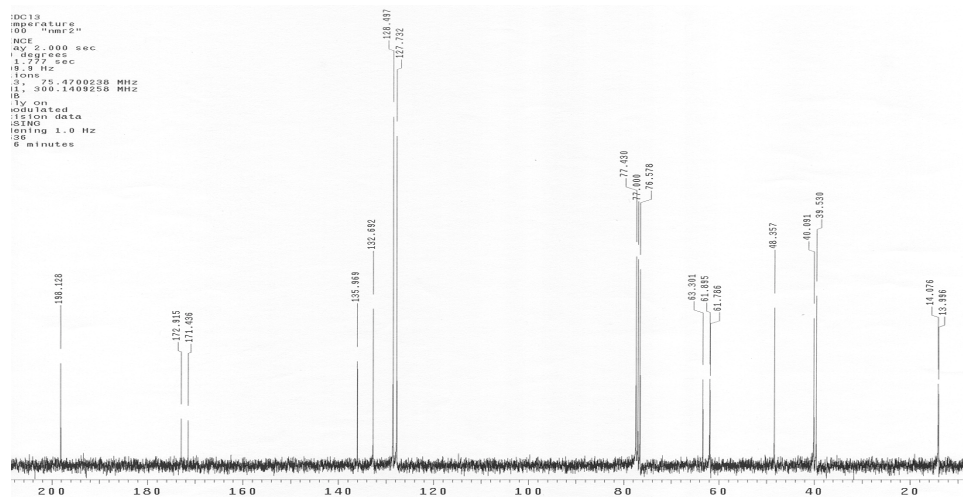
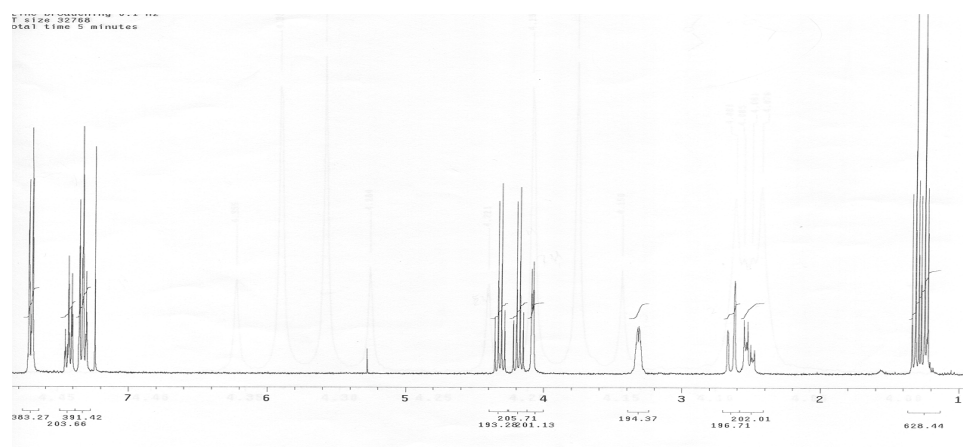
¹H NMR: (300 MHz, CDCl₃) 1.27 (m, 6H), 2.62 (m, 4H), 3.32 (m, 2H), 4.08 (d, *J* 3.9 Hz, 2H), 4.17 (q, *J* 6.9 Hz, 2H), 4.30 (q, *J* 6.3 Hz, 2H), 7.42 (m, 6H), 7.72 (m, 4H).

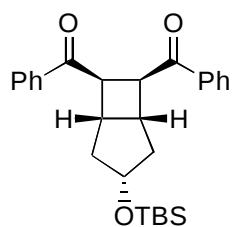
¹³C NMR: (75 MHz, CDCl₃) 13.9, 14.1, 39.5, 40.1, 48.3, 61.8, 61.9, 63.3, 127.7, 128.5, 132.7, 135.9, 171.4, 172.9, 198.1.

HRMS: Calc'd for C₂₇H₂₉O₆: 460.1582, Found: 460.1587.

FTIR: (KBr) 3054, 2985, 2306, 1724, 1686, 1597, 1581, 1448, 1272, 758, 697 cm⁻¹.

m.p.: 96 ~ 98 °C





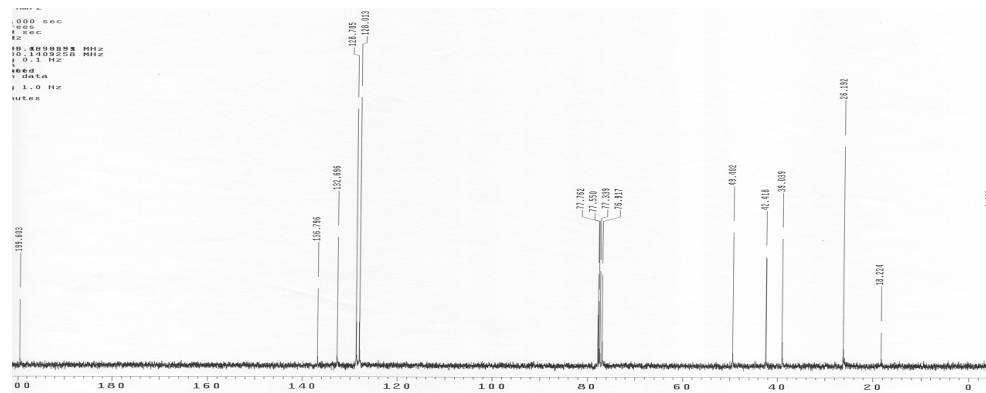
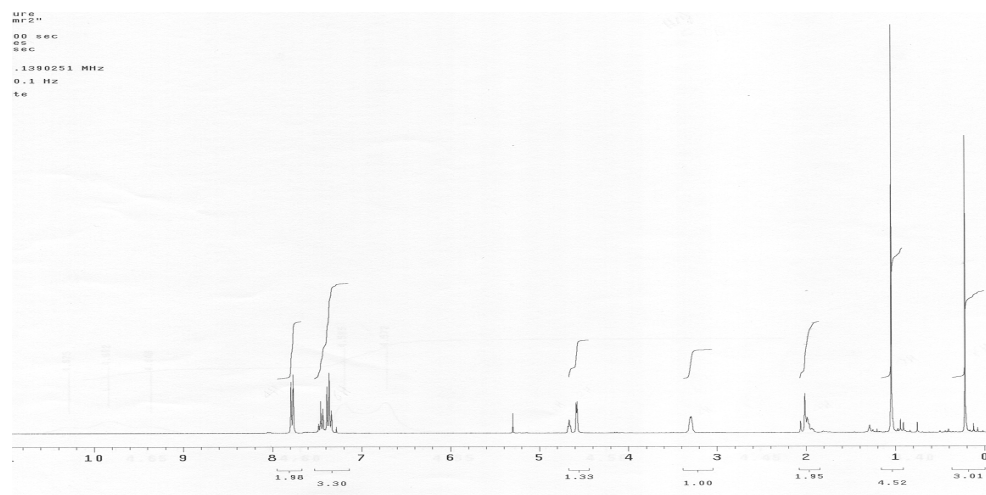
¹H NMR: (300 MHz, CDCl₃) 0.22 (s, 6H), 1.04 (s, 9H), 1.98 (m, 4H), 3.29 (d, *J* 2.7 Hz, 2H), 4.58 (d, *J* 3.9 Hz, 2H), 4.66 (m, 1H), 7.42 (m, 6H), 7.78 (m, 4H).

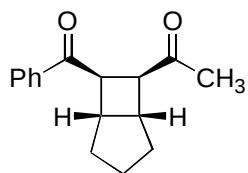
¹³C NMR: (75 MHz, CDCl₃) -4.5, 18.2, 26.2, 39.0, 42.4, 49.4, 128.0, 128.7, 132.7, 136.8, 199.6.

HRMS: Calc'd for C₂₇H₃₅O₃Si: 435.2355, Found: 435.2360.

FTIR: (KBr) 3054, 2954, 2929, 2856, 1684, 1448, 1264, 746, 704 cm⁻¹.

m.p.: 107 ~ 109 °C





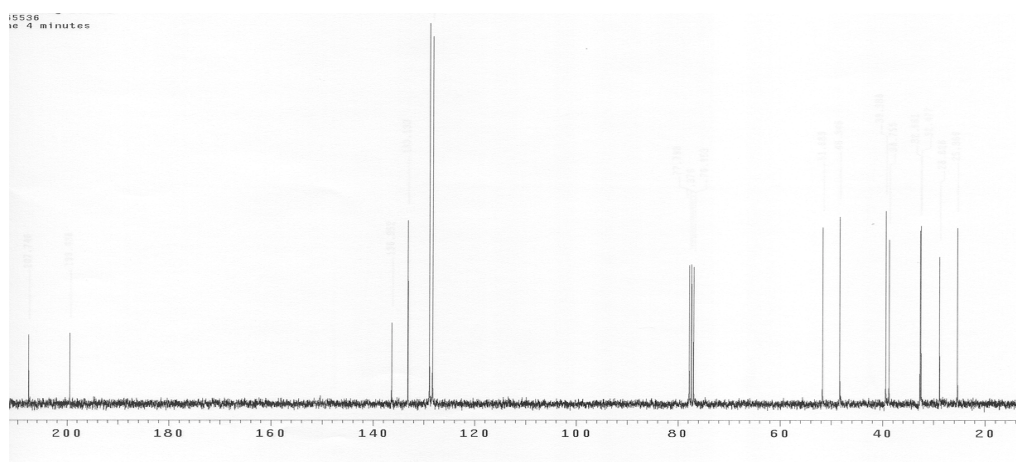
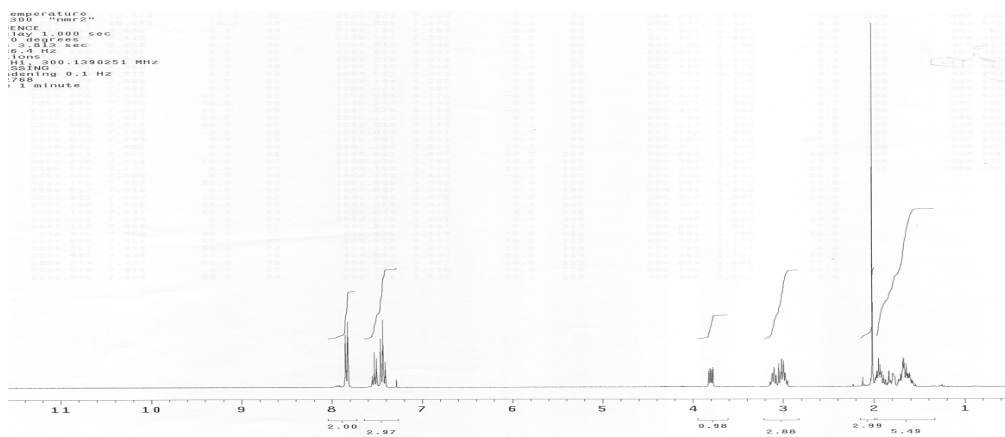
¹H NMR: (300 MHz, CDCl₃) 1.60 ~ 1.98 (m, 6H), 2.02 (s, 3H), 3.02 (m, 3H), 3.80 (dd, *J* 5.1, 9.6 Hz, 1H), 7.46 (m, 3H), 7.81 (m, 2H).

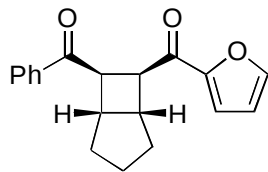
¹³C NMR: (75 MHz, CDCl₃) 25.4, 28.8, 32.5, 32.7, 38.7, 39.4, 48.3, 51.7, 128.2, 128.9, 133.1, 136.4, 199.4, 297.7.

HRMS: Calc'd for C₁₆H₁₉O₂: 243.1385, Found: 243.1387.

FTIR: (KBr) 3055, 2948, 2858, 1706, 1678, 1448, 1356, 1264, 1218, 739 cm⁻¹.

m.p.: 77 ~ 78 °C





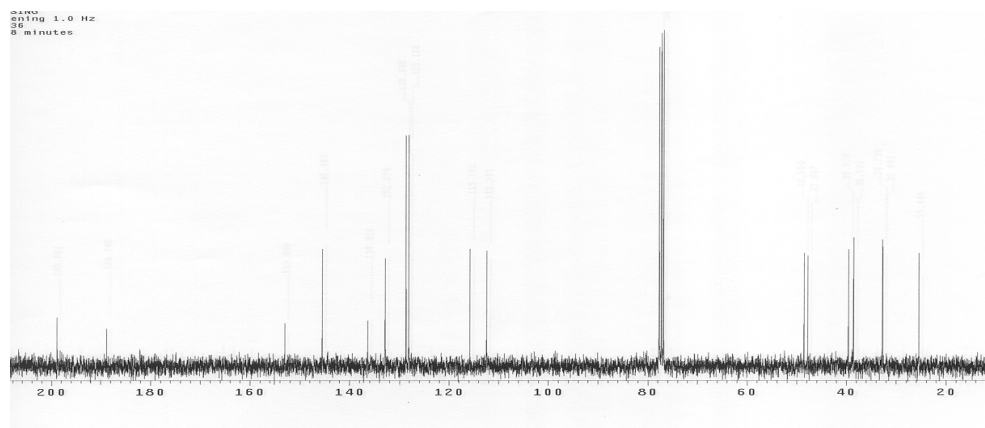
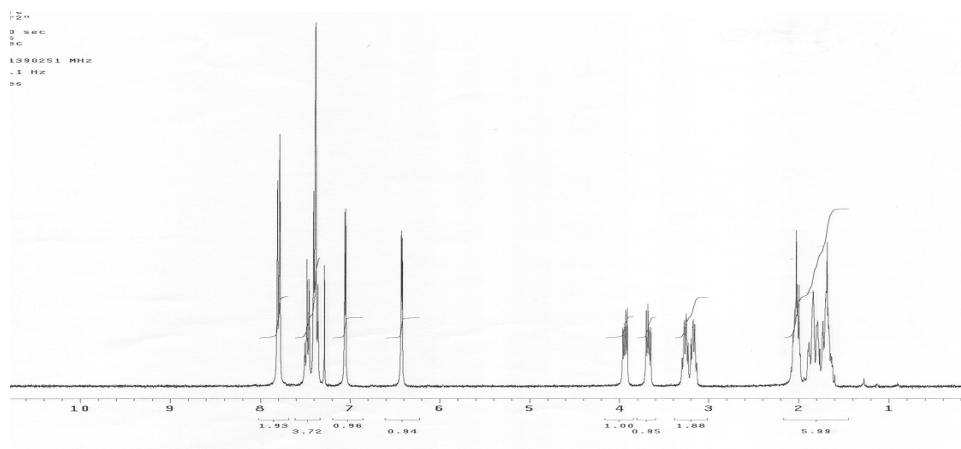
¹H NMR: (300 MHz, CDCl₃) 1.77 (m, 4H), 2.03 (m, 2H), 3.17 (m, 1H), 3.25 (m, 1H), 3.61 (dd, *J* 5.4, 9.9, 1H), 3.93 (dd, *J* 4.8, 9.9, 1H), 6.42 (dd, *J* 1.8, 3.6 Hz, 1H), 7.05 (dd, *J* 0.6, 3.6 Hz, 1H), 7.40 (m, 4H), 7.70 (m, 2H).

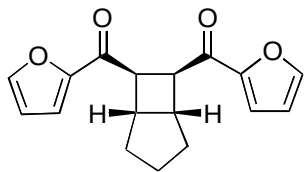
¹³C NMR: (75 MHz, CDCl₃) 25.4, 32.6, 32.7, 38.5, 39.6, 47.8, 48.5, 112.4, 115.7, 128.2, 128.7, 132.9, 136.3, 145.5, 153.0, 188.7, 198.6.

HRMS: Calc'd for C₁₉H₁₉O₃: 295.1334, Found: 295.1335.

FTIR: (KBr) 3054, 2950, 1682, 1467, 1265, 737, 704 cm⁻¹.

m.p.: 130 ~ 132 °C





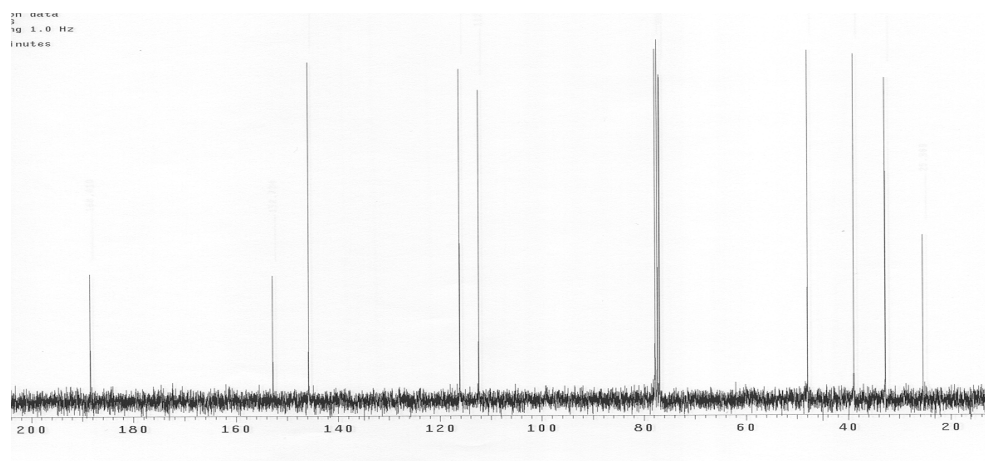
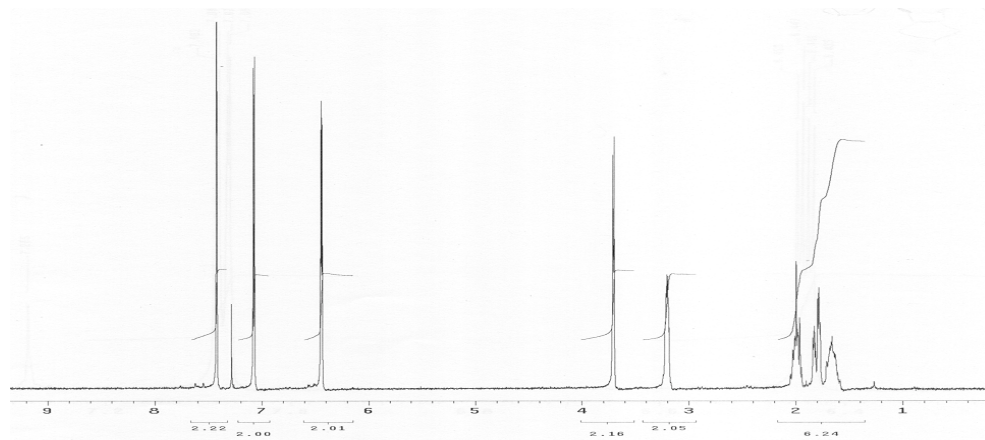
¹H NMR: (300 MHz, CDCl₃) 1.67 ~ 2.04 (m, 6H), 3.20 (m, 2H), 3.70 (d, *J* 4.2 Hz, 2H), 6.44 (dd, *J* 1.8, 3.6 Hz, 2H), 7.07 (dd, *J* 0.6, 3.6 Hz, 2H), 7.42 (dd, *J* 0.9, 1.5, 2H).

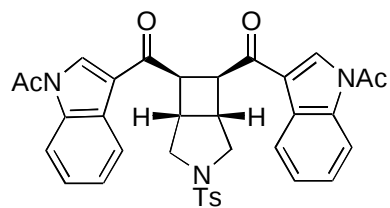
¹³C NMR: (75 MHz, CDCl₃) 25.4, 32.7, 38.9, 47.9, 112.4, 116.1, 145.7, 152.7, 188.7.

HRMS: Calc'd for C₁₇H₁₇O₄: 285.1126, Found: 285.1128.

FTIR: (KBr) 3054, 2953, 1678, 1570, 1468, 1265, 747, 704 cm⁻¹.

m.p.: 156 ~ 157 °C





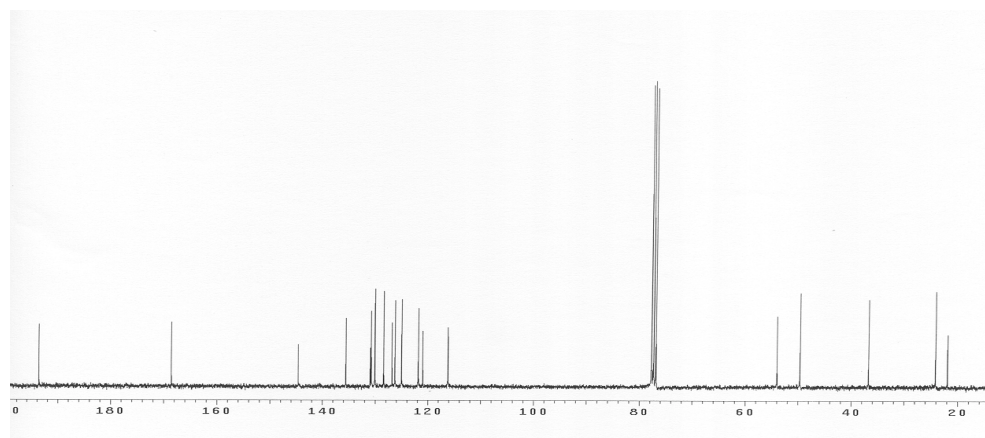
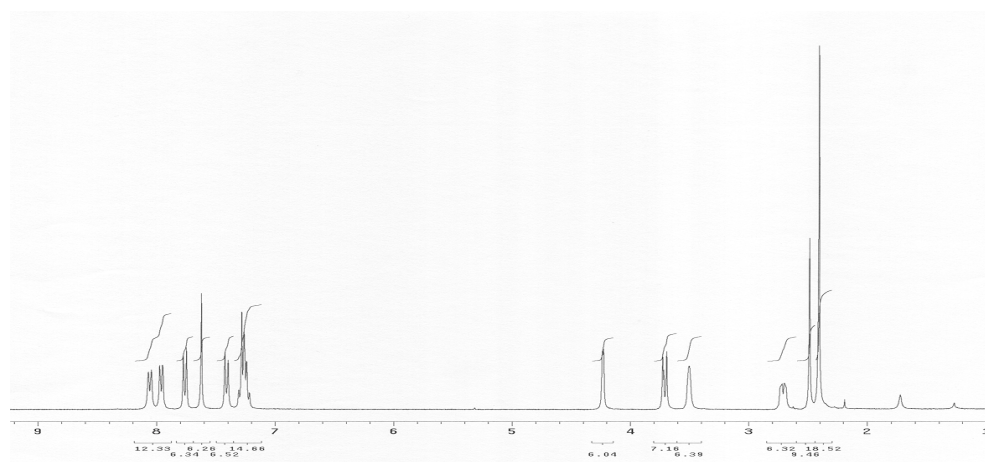
¹H NMR: (300 MHz, CDCl₃) 2.41 (s, 6H), 2.48 (s, 3H), 2.70 (m, 2H), 3.50 (s, 2H), 3.72 (d, *J* 3.0 Hz, 2H), 4.23 (d, *J* 2.8 Hz, 2H), 7.24 (m, 4H), 7.40 (d, *J* 7.9 Hz, 2H), 7.62 (s, 2H), 7.75 (d, *J* 8.4 Hz, 2H), 8.05 (d, *J* 7.9 Hz, 2H), 7.96 (d, *J* 6.9 Hz, 2H), 8.05 (d, *J* 7.9 Hz, 2H).

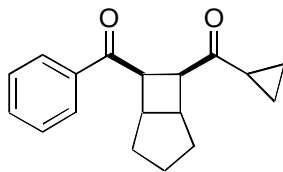
¹³C NMR: (75 MHz, CDCl₃) 21.8, 24.1, 36.7, 49.6, 53.9, 116.2, 121.0, 121.8, 125.0, 126.3, 126.8, 128.4, 130.1, 130.8, 131.0, 135.6, 144.6, 168.5, 193.6.

HRMS: Calc'd for C₃₅H₃₂N₃O₆S: 622.2011, Found: 622.2012.

FTIR: (KBr) 3052, 2984, 1723, 1670, 1545, 1447, 1262, 1213, 737, 703 cm⁻¹.

m.p.: 252 ~ 253 °C





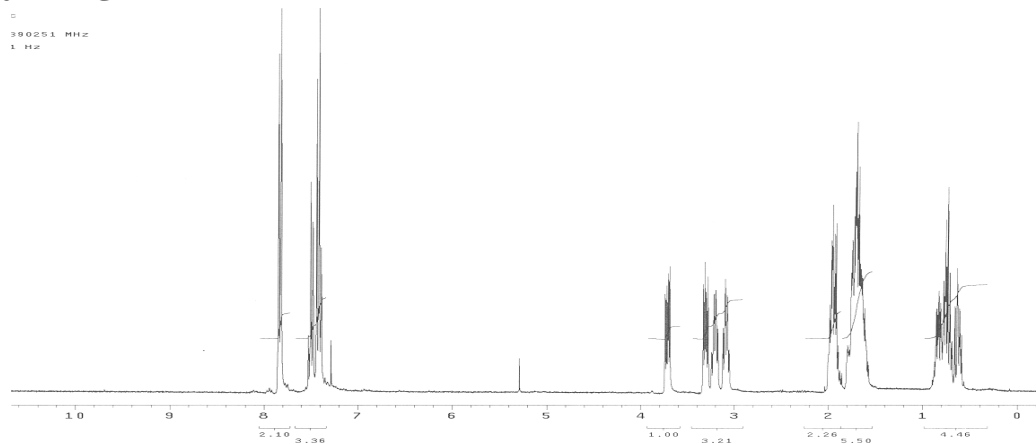
¹H NMR: (300 MHz, CDCl₃) 0.82 (m, 4H), 1.65 (m, 5H), 1.98 (m, 2H), 3.09 (dd, *J* 7.0, 12.7 Hz, 1H), 3.20 (dd, *J* 7.0, 12.7 Hz, 1H), 3.31 (dd, *J* 5.2, 9.7 Hz, 1H), 3.71 (dd, *J* 5.2, 9.7 Hz, 1H), 7.40 (m, 3H), 7.81 (m, 2H)

¹³C NMR: (75 MHz, CDCl₃) 10.9, 11.4, 19.8, 25.5, 32.6, 32.8, 38.3, 39.2, 47.2, 53.5, 128.2, 128.7, 132.8, 136.9, 199.5, 209.4

HRMS: Calc'd for C₁₈H₂₁O₂: 269.1541, Found: 269.1548

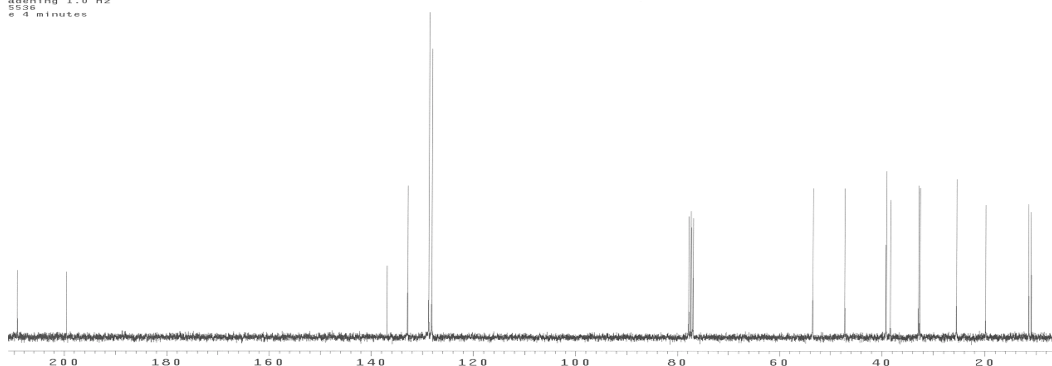
FTIR: (KBr) 2944, 1687, 1447, 1385, 1207, 911, 730, 693 cm⁻¹

m.p.: 70 ~72 °C



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-330 "nmr2"
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UENCE
day 2.000 sec
0.000 sec
0.100 sec
005.0 Hz
itions
C13. 25.4688888 MHz
H1. 300.1405258 MHz
ds
usly on
modulated
ecision data
ESSING 1.0 Hz
55.36
e 4 minutes
  
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Crystallographic Material for iso **1b**.

X-ray Experimental.

Table 1. Crystallographic Data for **1**.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of **1**.

Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of **1**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **1**.

Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of **1**.

Figure 1. View of **1** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary scale.

Figure 2. Unit cell packing diagram for **1**. The view is approximately down the **b** axis.

Table 6. Observed and calculated structure factor amplitudes for **1**. Values for F_o , F_c and $\sigma(F_o)$ have been multiplied by 10.

X-ray Experimental for $C_{21}H_{20}O_2$: Crystals grew as very large, colorless prisms by slow evaporation from ethyl acetate and hexane. The data crystal was cut from a larger crystal and had approximate dimensions; 0.30 x 0.19 x 0.16 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with $MoK\alpha$ radiation ($\lambda = 0.71073\text{\AA}$). A total of 469 frames of data were collected using ω -scans with a scan range of 1° and a counting time of 29 seconds per frame. The data were collected at -120°C using a Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR92² and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms were observed in a ΔF map and refined with isotropic displacement parameters. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0415*P)^2 + (0.9360*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.0982, with $R(F)$ equal to 0.0398 and a goodness of fit, S , = 1.072. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁴ The data were corrected for secondary extinction effects. The correction takes the form: $F_{\text{corr}} = kF_c/[1 + (6.0(6)\times 10^{-5}) * F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁵ All figures were generated using SHELXTL/PC.⁶ Tables of positional and thermal parameters, bond lengths and angles, figures and lists of observed and calculated structure factors are located in tables 1 through 6.

References

- 1) DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
- 2) SIR92. (1993). A program for crystal structure solution. Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. J. Appl. Cryst. 26, 343-350.
- 3) Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
- 4) $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w(|F_o|^4)\}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \Sigma(|F_o| - |F_c|) / \Sigma |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\Sigma w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
- 5) International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
- 6) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for 1.

Empirical formula	C ₂₁ H ₂₀ O ₂	
Formula weight	304.37	
Temperature	153(2) K	
Wavelength	0.71070 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.1952(2) Å	α = 71.637(1)°.
	b = 9.5194(2) Å	β = 85.462(1)°.
	c = 10.3566(2) Å	γ = 67.385(1)°.
Volume	793.31(3) Å ³	
Z	2	
Density (calculated)	1.274 Mg/m ³	
Absorption coefficient	0.080 mm ⁻¹	
F(000)	324	
Crystal size	0.30 x 0.19 x 0.16 mm	
Theta range for data collection	3.0 to 27.5°.	
Index ranges	-11 ≤ h ≤ 10, -12 ≤ k ≤ 11, -12 ≤ l ≤ 13	
Reflections collected	5529	
Independent reflections	3611 [R(int) = 0.0186]	
Completeness to theta = 27.53°	98.7 %	
Max. and min. transmission	0.9872 and 0.9763	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3611 / 0 / 289	
Goodness-of-fit on F ²	1.072	
Final R indices [I > 2σ(I)]	R1 = 0.0398, wR2 = 0.0896	
R indices (all data)	R1 = 0.0606, wR2 = 0.0982	
Extinction coefficient	6.0(6) × 10 ⁻⁵	
Largest diff. peak and hole	0.20 and -0.17 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
C1	3762(1)	3250(1)	1728(1)	27(1)
C2	4808(1)	3083(1)	2930(1)	28(1)
C3	6285(1)	2037(2)	2355(1)	31(1)
C4	7062(2)	301(2)	3226(2)	37(1)
C5	5921(2)	-461(2)	3083(1)	36(1)
C6	5351(2)	321(2)	1585(1)	35(1)
C7	5238(1)	2047(2)	1233(1)	30(1)
C8	2354(1)	2780(1)	2063(1)	27(1)
O9	1996(1)	2327(1)	3241(1)	33(1)
C10	1464(1)	2790(1)	910(1)	26(1)
C11	323(1)	2117(2)	1199(1)	33(1)
C12	-449(2)	2012(2)	160(1)	39(1)
C13	-108(2)	2600(2)	-1182(1)	40(1)
C14	992(2)	3302(2)	-1483(1)	39(1)
C15	1785(2)	3393(2)	-442(1)	33(1)
C16	4806(1)	4666(2)	2931(1)	31(1)
O17	5797(1)	5154(1)	2336(1)	53(1)
C18	3520(1)	5696(1)	3598(1)	27(1)
C19	3431(2)	7249(2)	3398(1)	33(1)
C20	2260(2)	8265(2)	3974(1)	37(1)
C21	1164(2)	7749(2)	4755(1)	35(1)
C22	1233(2)	6218(2)	4955(1)	34(1)
C23	2406(1)	5190(2)	4377(1)	29(1)

Table 3. Bond lengths [Å] and angles [°] for 1.

C1-C8	1.5074(16)	C10-C11	1.3955(17)
C1-C2	1.5641(17)	C11-C12	1.3842(19)
C1-C7	1.5748(16)	C11-H11	0.980(14)
C1-H1	0.969(14)	C12-C13	1.385(2)
C2-C16	1.5071(17)	C12-H12	0.999(16)
C2-C3	1.5477(17)	C13-C14	1.380(2)
C2-H2	1.011(13)	C13-H13	0.985(15)
C3-C4	1.5195(18)	C14-C15	1.3903(19)
C3-C7	1.5617(17)	C14-H14	0.987(16)
C3-H3	0.969(13)	C15-H15	0.969(14)
C4-C5	1.5252(19)	C16-O17	1.2177(15)
C4-H4A	0.992(15)	C16-C18	1.4974(17)
C4-H4B	0.976(15)	C18-C23	1.3910(17)
C5-C6	1.5288(18)	C18-C19	1.3972(17)
C5-H5A	0.990(14)	C19-C20	1.3823(19)
C5-H5B	1.001(14)	C19-H19	0.990(14)
C6-C7	1.5301(18)	C20-C21	1.3825(19)
C6-H6A	1.007(14)	C20-H20	0.987(15)
C6-H6B	1.012(15)	C21-C22	1.3828(18)
C7-H7	0.992(13)	C21-H21	0.972(15)
C8-O9	1.2236(14)	C22-C23	1.3907(18)
C8-C10	1.4960(17)	C22-H22	1.001(14)
C10-C15	1.3905(17)	C23-H23	0.980(13)
C8-C1-C2	118.45(10)	C1-C2-H2	110.5(7)
C8-C1-C7	116.21(10)	C4-C3-C2	116.01(11)
C2-C1-C7	89.64(9)	C4-C3-C7	106.29(10)
C8-C1-H1	108.1(8)	C2-C3-C7	90.72(9)
C2-C1-H1	112.5(8)	C4-C3-H3	114.0(7)
C7-C1-H1	110.9(8)	C2-C3-H3	111.1(7)
C16-C2-C3	115.39(10)	C7-C3-H3	116.6(7)
C16-C2-C1	112.68(10)	C3-C4-C5	104.25(10)
C3-C2-C1	90.03(9)	C3-C4-H4A	108.6(8)
C16-C2-H2	111.8(7)	C5-C4-H4A	109.2(8)
C3-C2-H2	114.7(7)	C3-C4-H4B	112.2(8)

C5-C4-H4B	112.4(8)	C13-C14-H14	122.3(9)
H4A-C4-H4B	109.9(11)	C15-C14-H14	117.4(9)
C4-C5-C6	103.40(11)	C14-C15-C10	120.17(13)
C4-C5-H5A	110.0(8)	C14-C15-H15	119.5(8)
C6-C5-H5A	110.0(7)	C10-C15-H15	120.3(8)
C4-C5-H5B	113.1(8)	O17-C16-C18	119.50(11)
C6-C5-H5B	112.3(8)	O17-C16-C2	120.53(11)
H5A-C5-H5B	108.1(11)	C18-C16-C2	119.87(10)
C5-C6-C7	105.41(10)	C23-C18-C19	119.02(12)
C5-C6-H6A	109.2(8)	C23-C18-C16	123.23(11)
C7-C6-H6A	107.7(8)	C19-C18-C16	117.73(11)
C5-C6-H6B	112.5(8)	C20-C19-C18	120.51(12)
C7-C6-H6B	113.9(8)	C20-C19-H19	122.2(8)
H6A-C6-H6B	108.0(11)	C18-C19-H19	117.3(8)
C6-C7-C3	105.38(10)	C21-C20-C19	120.10(12)
C6-C7-C1	117.21(10)	C21-C20-H20	121.0(8)
C3-C7-C1	89.13(9)	C19-C20-H20	118.9(8)
C6-C7-H7	112.1(7)	C20-C21-C22	120.01(13)
C3-C7-H7	117.5(7)	C20-C21-H21	119.4(8)
C1-C7-H7	113.5(7)	C22-C21-H21	120.5(8)
O9-C8-C10	120.88(11)	C21-C22-C23	120.21(12)
O9-C8-C1	121.37(11)	C21-C22-H22	119.5(8)
C10-C8-C1	117.65(10)	C23-C22-H22	120.3(8)
C15-C10-C11	119.03(11)	C22-C23-C18	120.14(12)
C15-C10-C8	121.87(11)	C22-C23-H23	119.2(8)
C11-C10-C8	119.05(11)	C18-C23-H23	120.6(8)
C12-C11-C10	120.52(12)		
C12-C11-H11	120.2(8)		
C10-C11-H11	119.3(8)		
C13-C12-C11	119.98(14)		
C13-C12-H12	119.7(9)		
C11-C12-H12	120.4(9)		
C14-C13-C12	119.99(13)		
C14-C13-H13	120.8(8)		
C12-C13-H13	119.2(8)		
C13-C14-C15	120.27(13)		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	25(1)	26(1)	28(1)	-7(1)	4(1)	-10(1)
C2	25(1)	30(1)	31(1)	-10(1)	4(1)	-13(1)
C3	23(1)	36(1)	38(1)	-14(1)	5(1)	-12(1)
C4	31(1)	39(1)	37(1)	-16(1)	-4(1)	-6(1)
C5	36(1)	30(1)	36(1)	-11(1)	-1(1)	-6(1)
C6	31(1)	35(1)	37(1)	-18(1)	-1(1)	-6(1)
C7	26(1)	36(1)	25(1)	-9(1)	6(1)	-9(1)
C8	25(1)	23(1)	30(1)	-8(1)	5(1)	-8(1)
O9	35(1)	42(1)	29(1)	-12(1)	8(1)	-21(1)
C10	22(1)	26(1)	28(1)	-9(1)	3(1)	-6(1)
C11	29(1)	39(1)	32(1)	-11(1)	3(1)	-14(1)
C12	31(1)	50(1)	42(1)	-19(1)	1(1)	-17(1)
C13	30(1)	48(1)	39(1)	-19(1)	-6(1)	-5(1)
C14	35(1)	42(1)	28(1)	-9(1)	0(1)	-4(1)
C15	28(1)	31(1)	33(1)	-6(1)	3(1)	-8(1)
C16	29(1)	34(1)	35(1)	-10(1)	4(1)	-16(1)
O17	48(1)	49(1)	78(1)	-28(1)	31(1)	-33(1)
C18	27(1)	28(1)	26(1)	-7(1)	-3(1)	-12(1)
C19	36(1)	32(1)	35(1)	-10(1)	0(1)	-19(1)
C20	45(1)	28(1)	40(1)	-11(1)	-4(1)	-16(1)
C21	37(1)	32(1)	34(1)	-14(1)	-1(1)	-9(1)
C22	34(1)	35(1)	31(1)	-10(1)	5(1)	-14(1)
C23	32(1)	26(1)	29(1)	-7(1)	2(1)	-14(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 1.

	x	y	z	U(eq)
H1	3415(15)	4309(16)	1067(14)	35(3)
H2	4487(14)	2498(14)	3830(13)	28(3)
H3	7013(15)	2573(15)	2045(13)	31(3)
H4A	8074(17)	-199(17)	2830(14)	40(4)
H4B	7248(16)	201(16)	4172(15)	40(4)
H5A	5022(16)	-184(15)	3672(13)	34(3)
H5B	6424(16)	-1656(17)	3333(13)	38(4)
H6A	6178(16)	-217(16)	1011(14)	39(4)
H6B	4332(17)	228(16)	1401(14)	41(4)
H7	5464(14)	2480(15)	269(14)	32(3)
H11	92(15)	1693(16)	2150(14)	37(4)
H12	-1252(19)	1512(18)	371(15)	53(4)
H13	-673(17)	2527(17)	-1913(15)	44(4)
H14	1260(17)	3742(17)	-2426(16)	52(4)
H15	2559(16)	3883(16)	-665(13)	35(4)
H19	4230(17)	7582(17)	2831(14)	42(4)
H20	2205(16)	9366(18)	3796(14)	43(4)
H21	345(17)	8473(17)	5155(14)	42(4)
H22	429(16)	5858(16)	5515(14)	41(4)
H23	2429(15)	4115(16)	4515(13)	31(3)

Figure 1. View of **1** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary scale.

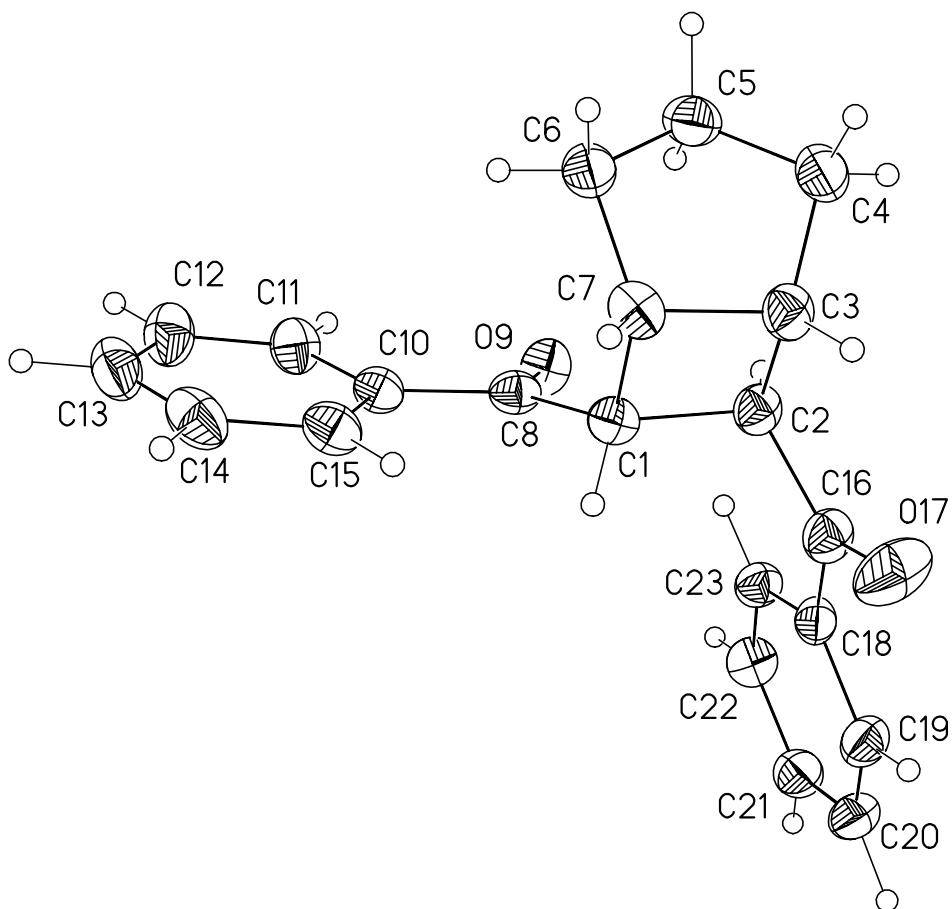


Figure 2. Unit cell packing diagram for **1**. The view is approximately down the **b** axis.

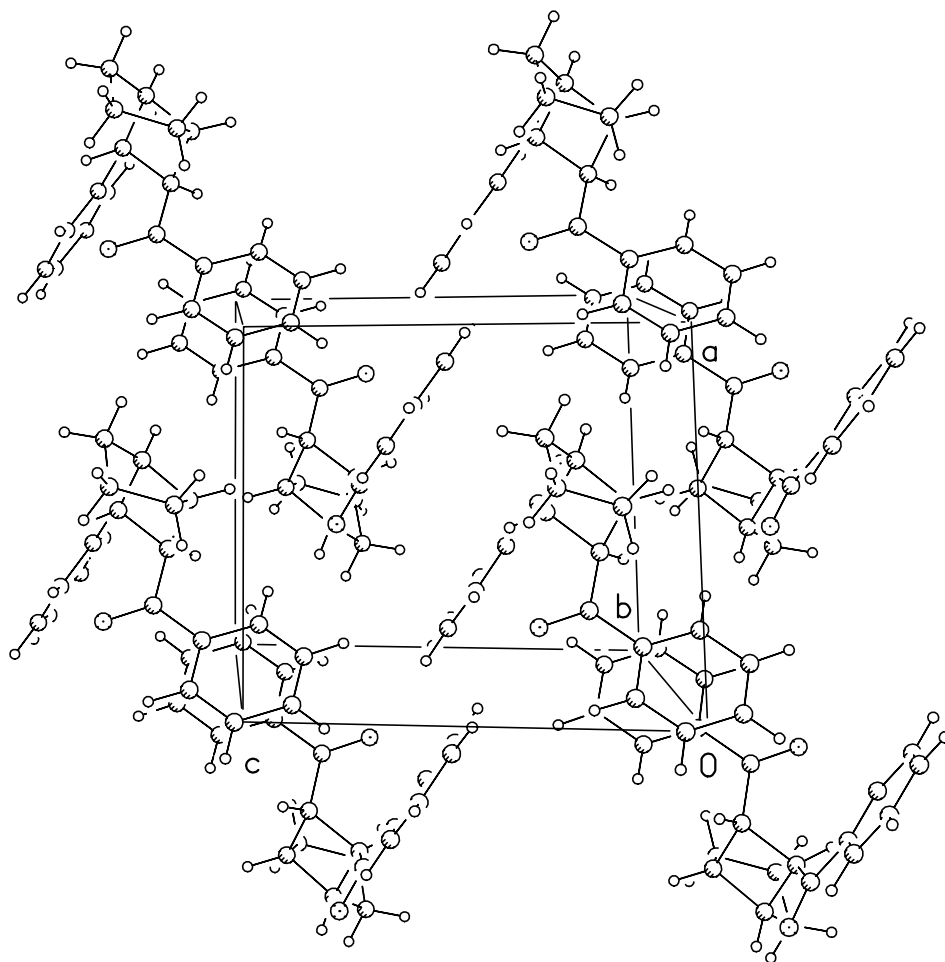


Table 6. Observed and calculated structure factors for 1

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h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	6	11	29	28	4	2	9	11	40	44	4	-1	2	12	54	54	3	1	5	12	21	26	4	5	8	12	90	89	5
3	6	11	43	38	3	3	9	11	10	0	10	0	2	12	11	14	11	2	5	12	35	41	4	-1	1	13	17	17	11
4	6	11	14	1	8	4	9	11	58	61	4	1	2	12	31	31	4	3	5	12	4	12	4	0	1	13	13	21	12
5	6	11	18	8	4	5	9	11	10	17	10	2	2	12	11	12	11	4	5	12	70	61	4	1	1	13	34	37	6
6	6	11	22	24	5	6	9	11	45	44	5	3	2	12	12	6	12	5	5	12	38	39	3	-2	2	13	12	9	11
7	6	11	20	28	5	-3	-1	12	24	29	7	5	2	12	59	61	6	6	5	12	0	0	1	-1	2	13	89	92	5
-2	7	11	60	59	4	-2	-1	12	0	4	1	-4	3	12	27	24	4	-2	6	12	0	2	1	0	2	13	49	44	4
-1	7	11	18	28	11	-1	-1	12	0	2	1	-3	3	12	18	8	8	-1	6	12	17	17	17	1	2	13	59	61	4
0	7	11	50	50	3	0	-1	12	44	42	4	-2	3	12	22	21	4	0	6	12	0	12	1	2	2	13	36	31	5
1	7	11	65	61	3	1	-1	12	25	25	8	-1	3	12	0	9	1	1	6	12	22	24	5	-2	3	13	33	35	7
2	7	11	10	10	9	-4	0	12	80	68	5	0	3	12	48	51	4	2	6	12	0	12	1	-1	3	13	29	33	6
3	7	11	81	74	3	-3	0	12	50	47	5	1	3	12	22	20	5	3	6	12	68	59	4	0	3	13	0	6	1
4	7	11	34	38	4	-2	0	12	100	99	4	2	3	12	28	34	4	4	6	12	30	31	5	1	3	13	0	10	1
5	7	11	28	24	4	-1	0	12	49	53	4	3	3	12	6	0	6	5	6	12	28	19	6	2	3	13	37	34	9
6	7	11	0	7	1	0	0	12	0	11	1	4	3	12	45	50	3	6	6	12	12	15	9	3	3	13	60	62	4
7	7	11	28	26	4	1	0	12	26	29	7	5	3	12	81	75	5	-1	7	12	0	8	1	-1	4	13	26	20	7
-1	8	11	0	21	1	2	0	12	15	4	10	-3	4	12	3	5	3	0	7	12	0	12	1	0	4	13	0	3	1
0	8	11	10	11	10	-4	1	12	28	28	10	-2	4	12	33	36	4	1	7	12	14	15	14	1	4	13	15	18	10
1	8	11	51	50	4	-3	1	12	20	27	11	-1	4	12	17	4	8	2	7	12	0	6	1	2	4	13	30	34	6
2	8	11	12	2	12	-2	1	12	21	22	11	0	4	12	26	27	3	3	7	12	31	27	3	3	4	13	53	52	4
3	8	11	33	33	3	-1	1	12	100	95	4	1	4	12	14	13	14	4	7	12	36	39	5	-1	5	13	7	5	7
4	8	11	76	79	4	0	1	12	24	8	8	4	4	12	12	4	11	5	7	12	19	2	10	0	6	13	12	17	12
5	8	11	9	6	8	1	1	12	31	29	4	5	4	12	20	17	5	0	8	12	36	41	6	1	6	13	41	40	4
6	8	11	10	12	9	2	1	12	7	2	6	-3	5	12	30	23	6	1	8	12	29	30	6						
7	8	11	18	0	17	-4	2	12	10	4	9	-2	5	12	36	32	5	2	8	12	13	4	12						
0	9	11	31	35	5	-3	2	12	49	42	4	-1	5	12	29	30	6	3	8	12	18	0	17						
1	9	11	0	10	1	-2	2	12	54	58	4	0	5	12	67	62	3	4	8	12	23	15	8						

Crystallographic Material for **1b**

X-ray Experimental.

Table 1. Crystallographic Data for 1.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of 1.

Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of 1.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of 1.

Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of 1.

Table 6. Observed and calculated structure factor amplitudes for 1. Values for F_o , F_c and $\sigma(F_o)$ have been multiplied by 10.

Figure 1. View of 1 showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Hydrogen atoms shown are drawn to an arbitrary scale.

Figure 2. Unit cell packing diagram for 1. The view is approximately down the **c** axis.

Table 6. Observed and calculated structure factor amplitudes 1. Values for F_o , F_c and $\sigma(F_o)$ have been multiplied by 10.

X-ray Experimental for C₂₁H₂₀O₂: Crystals grew as colorless by slow evaporation from ethylacetate and hexane. The data crystal was a long needle that had approximate dimensions; 0.45x0.08x0.08 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoK α radiation ($\lambda = 0.71073\text{\AA}$). A total of 366 frames of data were collected using ω -scans with a scan range of 1° and a counting time of 151 seconds per frame. The data were collected at –120 °C using a Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR92² and refined by full-matrix least-squares on F² with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.019*P)^2 + (0.7511*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.114, with R(F) equal to 0.0553 and a goodness of fit, S, = 1.008. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.⁴ The data were corrected for secondary extinction effects. The correction takes the form: $F_{\text{corr}} = kF_c/[1 + (8.4(12)\times 10^{-6}) * F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁵ All figures were generated using SHELXTL/PC.⁶ Tables of positional and thermal parameters, bond lengths and angles, figures and lists of observed and calculated structure factors are located in tables 1 through 6.

References

- 1) DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
- 2) SIR92. (1993). A program for crystal structure solution. Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. J. Appl. Cryst. 26, 343-350.
- 3) Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
- 4) $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w(|F_o|^4)\}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \Sigma(|F_o| - |F_c|) / \Sigma |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\Sigma w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
- 5) International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
- 6) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for 1.

Empirical formula	C ₂₁ H ₂₀ O ₂	
Formula weight	304.37	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 14.4375(5) Å	α = 90°.
	b = 10.6909(4) Å	β = 108.223(2)°.
	c = 10.8075(5) Å	γ = 90°.
Volume	1584.47(11) Å ³	
Z	4	
Density (calculated)	1.276 Mg/m ³	
Absorption coefficient	0.081 mm ⁻¹	
F(000)	648	
Crystal size	0.45 x 0.08 x 0.08 mm	
Theta range for data collection	3.0 to 27.5°.	
Index ranges	-18 ≤ h ≤ 18, -13 ≤ k ≤ 11, -14 ≤ l ≤ 13	
Reflections collected	5650	
Independent reflections	3606 [R(int) = 0.0413]	
Completeness to theta = 27.48°	99.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3606 / 0 / 209	
Goodness-of-fit on F ²	1.082	
Final R indices [I > 2σ(I)]	R1 = 0.0553, wR2 = 0.0999	
R indices (all data)	R1 = 0.0973, wR2 = 0.1140	
Extinction coefficient	8.4(12) × 10 ⁻⁶	
Largest diff. peak and hole	0.25 and -0.24 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
C1	1922(1)	2801(2)	923(2)	24(1)
C2	2975(1)	3018(2)	1870(2)	25(1)
C3	2649(1)	4270(2)	2323(2)	30(1)
C4	3093(2)	5446(2)	1944(2)	43(1)
C5	2602(2)	5554(2)	481(2)	45(1)
C6	1550(2)	5177(2)	285(2)	38(1)
C7	1624(1)	4140(2)	1273(2)	29(1)
C8	1301(1)	1880(2)	1343(2)	26(1)
O9	1569(1)	1420(1)	2432(1)	35(1)
C10	283(1)	1681(2)	453(2)	26(1)
C11	9(1)	1951(2)	-871(2)	31(1)
C12	-946(1)	1749(2)	-1652(2)	35(1)
C13	-1632(1)	1303(2)	-1109(2)	36(1)
C14	-1366(1)	1046(2)	205(2)	36(1)
C15	-410(1)	1215(2)	981(2)	31(1)
C16	3453(1)	2052(2)	2888(2)	26(1)
O17	3646(1)	2277(1)	4051(1)	34(1)
C18	3763(1)	828(2)	2454(2)	23(1)
C19	3726(1)	608(2)	1173(2)	28(1)
C20	4030(1)	-538(2)	826(2)	32(1)
C21	4358(1)	-1465(2)	1746(2)	33(1)
C22	4412(1)	-1250(2)	3032(2)	34(1)
C23	4124(1)	-102(2)	3388(2)	29(1)

Table 3. Bond lengths [Å] and angles [°] for 1.

C1-C8	1.494(2)	C10-C11	1.391(3)
C1-C2	1.562(2)	C11-C12	1.389(3)
C1-C7	1.574(2)	C11-H11	0.96
C1-H1	0.96	C12-C13	1.385(3)
C2-C16	1.509(3)	C12-H12	0.96
C2-C3	1.549(2)	C13-C14	1.378(3)
C2-H2	0.96	C13-H13	0.96
C3-C4	1.524(3)	C14-C15	1.384(3)
C3-C7	1.564(3)	C14-H14	0.96
C3-H3	0.96	C15-H15	0.96
C4-C5	1.522(3)	C16-O17	1.224(2)
C4-H4A	0.96	C16-C18	1.504(2)
C4-H4B	0.96	C18-C19	1.388(3)
C5-C6	1.521(3)	C18-C23	1.396(3)
C5-H5A	0.96	C19-C20	1.392(2)
C5-H5B	0.96	C19-H19	0.96
C6-C7	1.519(3)	C20-C21	1.378(3)
C6-H6A	0.96	C20-H20	0.96
C6-H6B	0.96	C21-C22	1.387(3)
C7-H7	0.96	C21-H21	0.96
C8-O9	1.222(2)	C22-C23	1.389(3)
C8-C10	1.499(3)	C22-H22	0.96
C10-C15	1.390(3)	C23-H23	0.96
C8-C1-C2	116.86(16)	C4-C3-C2	115.67(16)
C8-C1-C7	106.89(14)	C4-C3-C7	105.13(16)
C2-C1-C7	89.49(13)	C2-C3-C7	90.36(14)
C8-C1-H1	113.6	C4-C3-H3	114.6
C2-C1-H1	114.2	C2-C3-H3	113.7
C7-C1-H1	113.1	C7-C3-H3	114.6
C16-C2-C3	118.45(16)	C5-C4-C3	104.05(17)
C16-C2-C1	120.39(15)	C5-C4-H4A	111.4
C3-C2-C1	90.30(13)	C3-C4-H4A	110.8
C16-C2-H2	108.8	C5-C4-H4B	110.2
C3-C2-H2	108.0	C3-C4-H4B	111.3
C1-C2-H2	109.4	H4A-C4-H4B	109.0

C6-C5-C4	104.18(18)	C14-C15-C10	120.5(2)
C6-C5-H5A	110.4	C14-C15-H15	120.1
C4-C5-H5A	110.3	C10-C15-H15	119.4
C6-C5-H5B	111.3	O17-C16-C18	119.78(17)
C4-C5-H5B	111.5	O17-C16-C2	121.32(17)
H5A-C5-H5B	109.0	C18-C16-C2	118.74(16)
C7-C6-C5	104.37(17)	C19-C18-C23	119.32(17)
C7-C6-H6A	110.6	C19-C18-C16	122.37(16)
C5-C6-H6A	111.4	C23-C18-C16	118.29(17)
C7-C6-H6B	111.1	C18-C19-C20	120.02(18)
C5-C6-H6B	110.4	C18-C19-H19	119.2
H6A-C6-H6B	108.9	C20-C19-H19	120.8
C6-C7-C3	106.69(16)	C21-C20-C19	120.34(19)
C6-C7-C1	117.30(17)	C21-C20-H20	120.1
C3-C7-C1	89.27(13)	C19-C20-H20	119.6
C6-C7-H7	113.8	C20-C21-C22	120.16(18)
C3-C7-H7	114.2	C20-C21-H21	119.7
C1-C7-H7	113.0	C22-C21-H21	120.1
O9-C8-C1	121.30(17)	C21-C22-C23	119.76(19)
O9-C8-C10	120.77(16)	C21-C22-H22	119.7
C1-C8-C10	117.37(16)	C23-C22-H22	120.5
C15-C10-C11	119.12(17)	C22-C23-C18	120.36(19)
C15-C10-C8	118.37(17)	C22-C23-H23	120.4
C11-C10-C8	122.51(17)	C18-C23-H23	119.2
C12-C11-C10	120.17(19)		
C12-C11-H11	120.3		
C10-C11-H11	119.6		
C13-C12-C11	120.1(2)		
C13-C12-H12	119.9		
C11-C12-H12	120.0		
C14-C13-C12	119.98(19)		
C14-C13-H13	120.0		
C12-C13-H13	120.0		
C13-C14-C15	120.15(19)		
C13-C14-H14	119.6		
C15-C14-H14	120.3		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	24(1)	23(1)	25(1)	-2(1)	8(1)	0(1)
C2	25(1)	23(1)	27(1)	-2(1)	9(1)	-2(1)
C3	34(1)	24(1)	31(1)	-5(1)	9(1)	-1(1)
C4	33(1)	25(1)	68(2)	-2(1)	11(1)	-1(1)
C5	56(1)	27(1)	62(2)	8(1)	32(1)	7(1)
C6	45(1)	26(1)	40(1)	-1(1)	7(1)	8(1)
C7	27(1)	24(1)	39(1)	-3(1)	13(1)	4(1)
C8	28(1)	21(1)	28(1)	-3(1)	9(1)	0(1)
O9	33(1)	40(1)	30(1)	5(1)	7(1)	-5(1)
C10	26(1)	19(1)	33(1)	-3(1)	10(1)	0(1)
C11	30(1)	28(1)	35(1)	-1(1)	9(1)	-1(1)
C12	33(1)	30(1)	38(1)	0(1)	3(1)	-2(1)
C13	27(1)	25(1)	50(2)	-4(1)	4(1)	-2(1)
C14	28(1)	31(1)	50(1)	-3(1)	14(1)	-5(1)
C15	31(1)	27(1)	38(1)	-2(1)	13(1)	-3(1)
C16	21(1)	27(1)	27(1)	-2(1)	6(1)	-3(1)
O17	37(1)	36(1)	28(1)	-4(1)	6(1)	2(1)
C18	18(1)	25(1)	27(1)	1(1)	6(1)	-3(1)
C19	27(1)	25(1)	30(1)	2(1)	8(1)	-1(1)
C20	31(1)	30(1)	37(1)	-4(1)	13(1)	1(1)
C21	25(1)	25(1)	48(1)	-2(1)	8(1)	4(1)
C22	27(1)	31(1)	40(1)	9(1)	6(1)	5(1)
C23	22(1)	35(1)	28(1)	3(1)	6(1)	2(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1.

	x	y	z	U(eq)
H1	1887	2720	24	29
H2	3405	3206	1374	30
H3	2639	4257	3207	36
H4A	3787	5362	2154	51
H4B	2952	6171	2379	51
H5A	2899	4990	26	54
H5B	2642	6390	178	54
H6A	1240	4877	-583	46
H6B	1186	5875	448	46
H7	1090	4122	1624	35
H11	483	2275	-1242	38
H12	-1128	1909	-2571	42
H13	-2295	1183	-1645	43
H14	-1847	746	578	43
H15	-220	1005	1890	38
H19	3481	1250	533	33
H20	4018	-676	-56	39
H21	4555	-2257	1494	40
H22	4635	-1902	3666	41
H23	4177	66	4280	35

Figure 1. View of 1 showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Hydrogen atoms shown are drawn to an arbitrary scale.

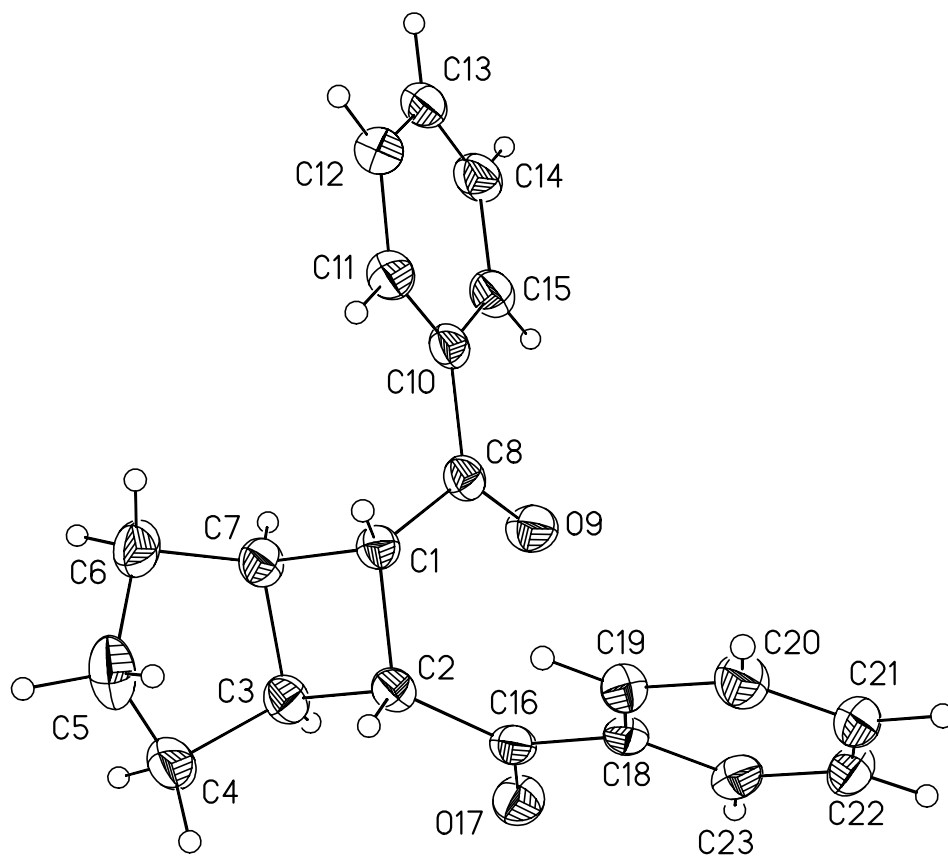


Figure 2. Unit cell packing diagram for 1. The view is approximately down the **c** axis.

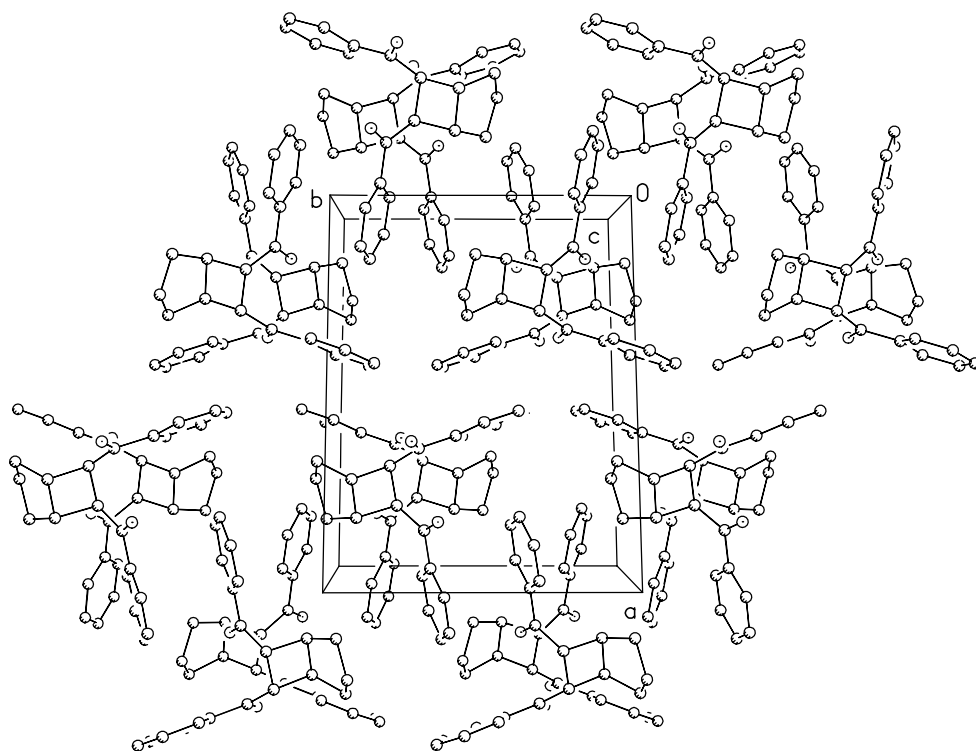


Table 6. Observed and calculated structure factors for 1

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h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
-12	2	12	32	31	31	-3	3	12	63	59	19	-7	5	12	0	5	1	-11	1	13	29	40	29	-9	3	13	46	9	27
-11	2	12	123	135	7	-2	3	12	87	72	11	-6	5	12	44	11	18	-10	1	13	27	51	27	-8	3	13	22	23	22
-10	2	12	23	23	23	-1	3	12	0	0	1	-5	5	12	54	30	23	-9	1	13	44	31	15	-7	3	13	0	14	1
-9	2	12	68	66	7	0	3	12	28	15	27	-4	5	12	86	86	15	-8	1	13	74	11	25	-6	3	13	82	50	10
-8	2	12	29	10	28	1	3	12	0	7	1	-3	5	12	0	33	1	-7	1	13	32	14	31	-5	3	13	53	47	32
-7	2	12	53	1	14	2	3	12	74	81	12	-2	5	12	0	21	1	-6	1	13	37	45	29	-4	3	13	59	43	16
-6	2	12	49	35	14	3	3	12	33	10	33	-1	5	12	44	14	16	-5	1	13	19	31	18	-3	3	13	0	14	1
-5	2	12	82	71	8	-12	4	12	0	5	1	0	5	12	0	6	1	-4	1	13	34	67	34	-2	3	13	36	25	28
-4	2	12	59	46	16	-11	4	12	0	8	1	1	5	12	0	23	1	-3	1	13	33	12	32	-1	3	13	27	56	26
-3	2	12	122	123	5	-10	4	12	89	91	7	-10	6	12	0	20	1	-2	1	13	81	52	13	0	3	13	35	30	35
-2	2	12	20	34	19	-9	4	12	0	13	1	-9	6	12	30	28	30	-1	1	13	49	30	15	-9	4	13	47	32	16
-1	2	12	63	66	10	-8	4	12	78	46	8	-8	6	12	22	0	21	0	1	13	0	8	1	-8	4	13	34	47	34
0	2	12	43	20	16	-7	4	12	45	53	12	-7	6	12	0	16	1	1	1	13	0	8	1	-7	4	13	50	26	20
1	2	12	52	51	12	-6	4	12	40	34	22	-6	6	12	61	42	12	-11	2	13	0	2	1	-5	4	13	35	5	34
2	2	12	16	39	15	-5	4	12	81	79	7	-5	6	12	0	2	1	-10	2	13	35	56	34	-4	4	13	29	17	28
3	2	12	0	32	1	-4	4	12	0	4	1	-4	6	12	33	6	32	-9	2	13	73	67	15	-3	4	13	50	42	15
-13	3	12	0	0	1	-3	4	12	17	21	16	-3	6	12	37	45	23	-8	2	13	58	16	20	-2	4	13	74	60	9
-12	3	12	62	60	13	-2	4	12	40	69	40	-2	6	12	38	37	38	-7	2	13	64	58	15	-7	5	13	0	22	1
-11	3	12	0	3	1	-1	4	12	42	36	16	-1	6	12	43	57	30	-6	2	13	56	64	16	-6	5	13	58	24	20
-10	3	12	78	64	8	0	4	12	0	10	1	0	6	12	39	2	39	-5	2	13	98	94	8	-5	5	13	51	12	44
-9	3	12	0	9	1	1	4	12	81	79	11	-7	7	12	89	87	8	-4	2	13	40	23	15	-4	5	13	58	67	18
-8	3	12	83	91	7	2	4	12	41	22	40	-6	7	12	33	29	33	-3	2	13	65	6	27	-6	0	14	8	34	8
-7	3	12	27	15	27	-11	5	12	37	43	26	-5	7	12	0	13	1	-2	2	13	52	47	21						
-6	3	12	49	56	13	-10	5	12	34	11	33	-4	7	12	0	4	1	-1	2	13	22	16	22						
-5	3	12	0	15	1	-9	5	12	52	21	11	-3	7	12	137	120	10	0	2	13	50	11	14						
-4	3	12	111	96	9	-8	5	12	46	34	12	-12	1	13	32	12	32	-10	3	13	32	28	32						

Crystallographic Material for **5b**

X-ray Experimental.

Table 1. Crystallographic Data for **5b**.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of **5b**.

Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of **5b**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **5b**.

Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of **5b**.

Figure 1. View of 1 showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Hydrogen atoms shown are drawn to an arbitrary scale.

Figure 2. Unit cell packing diagram for **5b**. The view is approximately down the **c** axis.

Figure 3. Unit cell packing diagram for **5b**. The view is approximately down the **a** axis. Molecules 2 are shown in wireframe form while molecules **5b** are displayed as ball-and-stick.

Figure 4. View of the fit by least-squares of selected atoms of molecule **5b** (dashed lines) onto the equivalent atoms of molecule 2. The seven atoms of the fused ring system were used in the fit.

Table 6. Observed and calculated structure factor amplitudes 1. Values for F_o , F_c and $\sigma(F_o)$ have been multiplied by 10.

X-ray Experimental for $C_{27}H_{34}SiO_3$: Crystals grew as long colorless lathes and needles by slow evaporation from ethylacetate and hexane. The data crystal was a long lathe that had approximate dimensions; 0.50 x 0.20 x 0.09 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with $MoK\alpha$ radiation ($\lambda = 0.71073\text{\AA}$). A total of 360 frames of data were collected using ω -scans with a scan range of 1.1° and a counting time of 168 seconds per frame. The data were collected at -120°C using a Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR92² and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms). There are two molecules in the asymmetric unit. Atoms of molecule 2 have a ' appended to the atom label. The molecules have only slight conformation differences (Figure 4.). The function, $\sum w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0244*P)^2 + (1.682*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.117, with $R(F)$ equal to 0.0579 and a goodness of fit, S , = 1.034. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁴ The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (5.1(5)\times 10^{-6}) * F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁵ All figures were generated using SHELXTL/PC.⁶ Tables of positional and thermal parameters, bond lengths and angles, figures and lists of observed and calculated structure factors are located in tables 1 through 6.

References

- 1) DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
- 2) SIR92. (1993). A program for crystal structure solution. Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. J. Appl. Cryst. 26, 343-350.
- 3) Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
- 4) $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w(|F_o|^4)\}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \Sigma(|F_o| - |F_c|) / \Sigma |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\Sigma w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
- 5) International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
- 6) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for 5b.

Empirical formula	C ₂₇ H ₃₄ O ₃ Si	
Formula weight	434.63	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.4365(1) Å	$\alpha = 92.475(1)^\circ$.
	b = 12.1163(2) Å	$\beta = 95.407(1)^\circ$.
	c = 20.7037(3) Å	$\gamma = 107.727(1)^\circ$.
Volume	2475.47(6) Å ³	
Z	4	
Density (calculated)	1.166 Mg/m ³	
Absorption coefficient	0.119 mm ⁻¹	
F(000)	936	
Crystal size	0.50 x 0.20 x 0.09 mm	
Theta range for data collection	2.94 to 27.50°.	
Index ranges	-13 ≤ h ≤ 13, -15 ≤ k ≤ 12, -26 ≤ l ≤ 26	
Reflections collected	16390	
Independent reflections	11097 [R(int) = 0.0280]	
Completeness to theta = 27.50°	97.4 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	11097 / 0 / 560	
Goodness-of-fit on F ²	1.034	
Final R indices [I > 2σ(I)]	R1 = 0.0579, wR2 = 0.1028	
R indices (all data)	R1 = 0.0958, wR2 = 0.1167	
Extinction coefficient	5.1(5) × 10 ⁻⁶	
Largest diff. peak and hole	0.28 and -0.34 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C1	4382(2)	6195(2)	6168(1)	27(1)
C2	4190(2)	7201(2)	6575(1)	30(1)
C3	4965(2)	7217(2)	7241(1)	27(1)
C4	4359(2)	6239(2)	7680(1)	24(1)
C5	5603(2)	5786(2)	7645(1)	23(1)
C6	6098(2)	6665(2)	7119(1)	26(1)
C7	5815(2)	6185(2)	6404(1)	30(1)
O8	3392(1)	5134(1)	6292(1)	28(1)
Si9	2536(1)	4120(1)	5716(1)	25(1)
C10	3655(2)	3340(2)	5403(1)	37(1)
C11	1858(2)	4815(2)	5033(1)	39(1)
C12	1140(2)	3125(2)	6122(1)	34(1)
C13	1707(3)	2865(2)	6786(1)	51(1)
C14	515(3)	1973(2)	5703(1)	55(1)
C15	34(3)	3696(3)	6216(1)	58(1)
C16	3989(2)	6578(2)	8331(1)	26(1)
O17	3882(2)	7539(1)	8448(1)	38(1)
C18	3652(2)	5670(2)	8812(1)	26(1)
C19	3551(2)	6007(2)	9453(1)	38(1)
C20	3246(3)	5182(2)	9907(1)	47(1)
C21	3031(2)	4025(2)	9732(1)	44(1)
C22	3114(2)	3680(2)	9096(1)	36(1)
C23	3428(2)	4498(2)	8639(1)	30(1)
C24	6637(2)	6098(2)	8238(1)	24(1)
O25	6549(1)	6760(1)	8682(1)	31(1)
C26	7816(2)	5640(2)	8259(1)	25(1)
C27	8806(2)	5993(2)	8796(1)	35(1)
C28	9873(2)	5543(2)	8857(1)	46(1)
C29	9969(2)	4743(2)	8387(1)	46(1)
C30	9020(2)	4405(2)	7848(1)	39(1)
C31	7941(2)	4854(2)	7780(1)	30(1)

C1'	5643(2)	1466(2)	8864(1)	30(1)
C2'	5791(2)	2582(2)	8523(1)	29(1)
C3'	4955(2)	2188(2)	7860(1)	26(1)
C4'	5520(2)	1533(2)	7357(1)	22(1)
C5'	4292(2)	417(2)	7377(1)	23(1)
C6'	3842(2)	1029(2)	7957(1)	27(1)
C7'	4193(2)	702(2)	8646(1)	32(1)
O8'	6593(2)	925(1)	8647(1)	33(1)
Si9'	7756(1)	621(1)	9139(1)	32(1)
C10'	8617(3)	1890(2)	9735(1)	55(1)
C11'	6920(3)	-663(2)	9575(1)	47(1)
C12'	8969(2)	337(2)	8586(1)	36(1)
C13'	8211(3)	-586(2)	8041(1)	47(1)
C14'	9694(3)	1448(2)	8273(2)	67(1)
C15'	10024(3)	-98(4)	8972(2)	83(1)
C16'	5849(2)	2070(2)	6722(1)	25(1)
O17'	5873(2)	3069(1)	6649(1)	33(1)
C18'	6255(2)	1376(2)	6208(1)	26(1)
C19'	6367(2)	1776(2)	5588(1)	34(1)
C20'	6707(2)	1139(2)	5098(1)	40(1)
C21'	6949(2)	105(2)	5219(1)	39(1)
C22'	6864(2)	-290(2)	5832(1)	41(1)
C23'	6512(2)	338(2)	6324(1)	32(1)
C24'	3214(2)	143(2)	6809(1)	23(1)
O25'	3296(1)	779(1)	6360(1)	32(1)
C26'	1985(2)	-895(2)	6818(1)	25(1)
C27'	966(2)	-1134(2)	6300(1)	33(1)
C28'	-159(2)	-2106(2)	6281(1)	41(1)
C29'	-282(2)	-2848(2)	6776(1)	40(1)
C30'	710(2)	-2606(2)	7299(1)	38(1)
C31'	1846(2)	-1636(2)	7324(1)	31(1)

Table 3. Bond lengths [Å] and angles [°] for 1.

C1-O8	1.433(2)	C13-H13B	0.96
C1-C2	1.527(3)	C13-H13C	0.96
C1-C7	1.534(3)	C14-H14A	0.96
C1-H1	0.96	C14-H14B	0.96
C2-C3	1.528(3)	C14-H14C	0.96
C2-H2A	0.96	C15-H15A	0.96
C2-H2B	0.96	C15-H15B	0.96
C3-C4	1.542(3)	C15-H15C	0.96
C3-C6	1.560(3)	C16-O17	1.219(2)
C3-H3	0.96	C16-C18	1.500(3)
C4-C16	1.510(3)	C18-C23	1.392(3)
C4-C5	1.562(3)	C18-C19	1.396(3)
C4-H4	0.96	C19-C20	1.389(3)
C5-C24	1.508(3)	C19-H19	0.96
C5-C6	1.568(3)	C20-C21	1.376(4)
C5-H5	0.96	C20-H20	0.96
C6-C7	1.535(3)	C21-C22	1.382(3)
C6-H6	0.96	C21-H21	0.96
C7-H7A	0.96	C22-C23	1.388(3)
C7-H7B	0.96	C22-H22	0.96
O8-Si9	1.6531(14)	C23-H23	0.96
Si9-C10	1.855(2)	C24-O25	1.223(2)
Si9-C11	1.868(2)	C24-C26	1.493(3)
Si9-C12	1.879(2)	C26-C31	1.389(3)
C10-H10A	0.96	C26-C27	1.398(3)
C10-H10B	0.96	C27-C28	1.379(3)
C10-H10C	0.96	C27-H27	0.96
C11-H11A	0.96	C28-C29	1.375(4)
C11-H11B	0.96	C28-H28	0.96
C11-H11C	0.96	C29-C30	1.378(4)
C12-C13	1.528(3)	C29-H29	0.96
C12-C14	1.536(3)	C30-C31	1.390(3)
C12-C15	1.539(3)	C30-H30	0.96
C13-H13A	0.96	C31-H31	0.96

C1'-O8'	1.439(2)	C13'-H13E	0.96
C1'-C2'	1.525(3)	C13'-H13F	0.96
C1'-C7'	1.527(3)	C14'-H14D	0.96
C1'-H1'	0.96	C14'-H14E	0.96
C2'-C3'	1.529(3)	C14'-H14F	0.96
C2'-H2'A	0.96	C15'-H15D	0.96
C2'-H2'B	0.96	C15'-H15E	0.96
C3'-C4'	1.547(3)	C15'-H15F	0.96
C3'-C6'	1.560(3)	C16'-O17'	1.219(2)
C3'-H3'	0.96	C16'-C18'	1.498(3)
C4'-C16'	1.513(3)	C18'-C23'	1.390(3)
C4'-C5'	1.560(3)	C18'-C19'	1.393(3)
C4'-H4'	0.96	C19'-C20'	1.385(3)
C5'-C24'	1.500(3)	C19'-H19'	0.96
C5'-C6'	1.566(3)	C20'-C21'	1.380(3)
C5'-H5'	0.96	C20'-H20'	0.96
C6'-C7'	1.537(3)	C21'-C22'	1.376(3)
C6'-H6'	0.96	C21'-H21'	0.96
C7'-H7'A	0.96	C22'-C23'	1.388(3)
C7'-H7'B	0.96	C22'-H22'	0.96
O8'-Si9'	1.6478(15)	C23'-H23'	0.96
Si9'-C11'	1.855(3)	C24'-O25'	1.225(2)
Si9'-C10'	1.867(2)	C24'-C26'	1.501(3)
Si9'-C12'	1.880(2)	C26'-C27'	1.391(3)
C10'-H10D	0.96	C26'-C31'	1.398(3)
C10'-H10E	0.96	C27'-C28'	1.382(3)
C10'-H10F	0.96	C27'-H27'	0.96
C11'-H11D	0.96	C28'-C29'	1.382(3)
C11'-H11E	0.96	C28'-H28'	0.96
C11'-H11F	0.96	C29'-C30'	1.381(3)
C12'-C13'	1.528(3)	C29'-H29'	0.96
C12'-C14'	1.534(4)	C30'-C31'	1.387(3)
C12'-C15'	1.533(3)	C30'-H30'	0.96
C13'-H13D	0.96	C31'-H31'	0.96
O8-C1-C2	109.16(16)	C2-C1-C7	104.63(16)
O8-C1-C7	110.84(17)	O8-C1-H1	110.6

C2-C1-H1	110.9	C1-C7-H7B	110.0
C7-C1-H1	110.5	H7A-C7-H7B	108.6
C3-C2-C1	105.27(16)	C1-O8-Si9	123.63(12)
C3-C2-H2A	110.5	O8-Si9-C10	110.26(9)
C1-C2-H2A	110.4	O8-Si9-C11	109.26(9)
C3-C2-H2B	110.5	C10-Si9-C11	109.03(11)
C1-C2-H2B	111.3	O8-Si9-C12	104.90(9)
H2A-C2-H2B	108.8	C10-Si9-C12	111.50(11)
C2-C3-C4	117.76(17)	C11-Si9-C12	111.81(11)
C2-C3-C6	106.09(16)	Si9-C10-H10A	109.6
C4-C3-C6	89.74(15)	Si9-C10-H10B	109.2
C2-C3-H3	114.0	H10A-C10-H10B	109.5
C4-C3-H3	112.8	Si9-C10-H10C	109.6
C6-C3-H3	113.8	H10A-C10-H10C	109.5
C16-C4-C3	117.84(16)	H10B-C10-H10C	109.5
C16-C4-C5	120.21(16)	Si9-C11-H11A	109.4
C3-C4-C5	90.69(14)	Si9-C11-H11B	109.3
C16-C4-H4	108.9	H11A-C11-H11B	109.5
C3-C4-H4	108.8	Si9-C11-H11C	109.7
C5-C4-H4	109.0	H11A-C11-H11C	109.5
C24-C5-C4	115.78(16)	H11B-C11-H11C	109.5
C24-C5-C6	109.03(16)	C13-C12-C14	108.6(2)
C4-C5-C6	88.71(14)	C13-C12-C15	109.1(2)
C24-C5-H5	113.4	C14-C12-C15	109.3(2)
C4-C5-H5	113.9	C13-C12-Si9	109.84(15)
C6-C5-H5	113.6	C14-C12-Si9	109.89(16)
C7-C6-C3	106.26(16)	C15-C12-Si9	110.17(17)
C7-C6-C5	117.44(16)	C12-C13-H13A	109.4
C3-C6-C5	89.79(14)	C12-C13-H13B	109.4
C7-C6-H6	113.5	H13A-C13-H13B	109.5
C3-C6-H6	113.8	C12-C13-H13C	109.6
C5-C6-H6	113.4	H13A-C13-H13C	109.5
C6-C7-C1	106.38(16)	H13B-C13-H13C	109.5
C6-C7-H7A	110.9	C12-C14-H14A	110.2
C1-C7-H7A	111.3	C12-C14-H14B	108.6
C6-C7-H7B	109.7	H14A-C14-H14B	109.5

C12-C14-H14C	109.6	C28-C27-C26	120.6(2)
H14A-C14-H14C	109.5	C28-C27-H27	119.9
H14B-C14-H14C	109.5	C26-C27-H27	119.5
C12-C15-H15A	110.1	C29-C28-C27	119.8(2)
C12-C15-H15B	109.6	C29-C28-H28	120.1
H15A-C15-H15B	109.5	C27-C28-H28	120.0
C12-C15-H15C	108.6	C28-C29-C30	120.4(2)
H15A-C15-H15C	109.5	C28-C29-H29	119.6
H15B-C15-H15C	109.5	C30-C29-H29	119.9
O17-C16-C18	120.65(18)	C29-C30-C31	120.3(2)
O17-C16-C4	121.15(19)	C29-C30-H30	119.6
C18-C16-C4	118.05(17)	C31-C30-H30	120.2
C23-C18-C19	118.7(2)	C26-C31-C30	119.8(2)
C23-C18-C16	122.20(18)	C26-C31-H31	119.9
C19-C18-C16	119.07(19)	C30-C31-H31	120.3
C20-C19-C18	120.0(2)	O8'-C1'-C2'	109.04(16)
C20-C19-H19	120.6	O8'-C1'-C7'	110.40(17)
C18-C19-H19	119.4	C2'-C1'-C7'	104.82(17)
C21-C20-C19	120.8(2)	O8'-C1'-H1'	110.8
C21-C20-H20	119.8	C2'-C1'-H1'	110.6
C19-C20-H20	119.5	C7'-C1'-H1'	111.1
C20-C21-C22	119.6(2)	C3'-C2'-C1'	104.88(16)
C20-C21-H21	120.5	C3'-C2'-H2'A	110.7
C22-C21-H21	119.8	C1'-C2'-H2'A	110.4
C21-C22-C23	120.2(2)	C3'-C2'-H2'B	110.8
C21-C22-H22	120.3	C1'-C2'-H2'B	111.4
C23-C22-H22	119.6	H2'A-C2'-H2'B	108.7
C22-C23-C18	120.6(2)	C2'-C3'-C4'	117.48(17)
C22-C23-H23	120.1	C2'-C3'-C6'	105.85(16)
C18-C23-H23	119.3	C4'-C3'-C6'	89.79(14)
O25-C24-C26	120.20(18)	C2'-C3'-H3'	113.7
O25-C24-C5	120.57(17)	C4'-C3'-H3'	113.1
C26-C24-C5	119.15(17)	C6'-C3'-H3'	114.3
C31-C26-C27	119.09(19)	C16'-C4'-C3'	118.35(16)
C31-C26-C24	122.53(18)	C16'-C4'-C5'	121.10(16)
C27-C26-C24	118.35(18)	C3'-C4'-C5'	90.42(14)

C16'-C4'-H4'	108.5	H11D-C11'-H11E	109.5
C3'-C4'-H4'	108.0	Si9'-C11'-H11F	109.7
C5'-C4'-H4'	108.9	H11D-C11'-H11F	109.5
C24'-C5'-C4'	116.43(16)	H11E-C11'-H11F	109.5
C24'-C5'-C6'	109.78(15)	C13'-C12'-C14'	108.0(2)
C4'-C5'-C6'	89.09(14)	C13'-C12'-C15'	108.6(2)
C24'-C5'-H5'	113.0	C14'-C12'-C15'	109.2(2)
C4'-C5'-H5'	113.5	C13'-C12'-Si9'	110.36(16)
C6'-C5'-H5'	112.8	C14'-C12'-Si9'	110.55(17)
C7'-C6'-C3'	106.34(16)	C15'-C12'-Si9'	110.08(18)
C7'-C6'-C5'	117.27(17)	C12'-C13'-H13D	109.7
C3'-C6'-C5'	89.69(14)	C12'-C13'-H13E	109.3
C7'-C6'-H6'	114.0	H13D-C13'-H13E	109.5
C3'-C6'-H6'	113.8	C12'-C13'-H13F	109.4
C5'-C6'-H6'	113.1	H13D-C13'-H13F	109.5
C1'-C7'-C6'	105.86(17)	H13E-C13'-H13F	109.5
C1'-C7'-H7'A	111.6	C12'-C14'-H14D	109.8
C6'-C7'-H7'A	111.2	C12'-C14'-H14E	108.8
C1'-C7'-H7'B	109.6	H14D-C14'-H14E	109.5
C6'-C7'-H7'B	109.7	C12'-C14'-H14F	109.8
H7'A-C7'-H7'B	108.8	H14D-C14'-H14F	109.5
C1'-O8'-Si9'	123.83(12)	H14E-C14'-H14F	109.5
O8'-Si9'-C11'	108.58(11)	C12'-C15'-H15D	108.5
O8'-Si9'-C10'	109.75(10)	C12'-C15'-H15E	110.3
C11'-Si9'-C10'	109.87(13)	H15D-C15'-H15E	109.5
O8'-Si9'-C12'	104.50(9)	C12'-C15'-H15F	109.7
C11'-Si9'-C12'	112.96(11)	H15D-C15'-H15F	109.5
C10'-Si9'-C12'	111.01(12)	H15E-C15'-H15F	109.5
Si9'-C10'-H10D	110.0	O17'-C16'-C18'	120.82(18)
Si9'-C10'-H10E	109.2	O17'-C16'-C4'	120.82(18)
H10D-C10'-H10E	109.5	C18'-C16'-C4'	118.17(17)
Si9'-C10'-H10F	109.2	C23'-C18'-C19'	118.54(19)
H10D-C10'-H10F	109.5	C23'-C18'-C16'	122.34(18)
H10E-C10'-H10F	109.5	C19'-C18'-C16'	119.12(19)
Si9'-C11'-H11D	109.2	C20'-C19'-C18'	120.5(2)
Si9'-C11'-H11E	109.5	C20'-C19'-H19'	120.0

C18'-C19'-H19'	119.6
C21'-C20'-C19'	120.3(2)
C21'-C20'-H20'	119.8
C19'-C20'-H20'	119.8
C22'-C21'-C20'	119.8(2)
C22'-C21'-H21'	120.4
C20'-C21'-H21'	119.8
C21'-C22'-C23'	120.2(2)
C21'-C22'-H22'	120.0
C23'-C22'-H22'	119.8
C18'-C23'-C22'	120.6(2)
C18'-C23'-H23'	119.3
C22'-C23'-H23'	120.1
O25'-C24'-C5'	121.16(17)
O25'-C24'-C26'	120.26(18)
C5'-C24'-C26'	118.48(17)
C27'-C26'-C31'	119.24(19)
C27'-C26'-C24'	118.68(18)
C31'-C26'-C24'	122.07(18)
C28'-C27'-C26'	120.3(2)
C28'-C27'-H27'	120.2
C26'-C27'-H27'	119.5
C27'-C28'-C29'	120.3(2)
C27'-C28'-H28'	119.9
C29'-C28'-H28'	119.7
C28'-C29'-C30'	119.9(2)
C28'-C29'-H29'	119.8
C30'-C29'-H29'	120.3
C29'-C30'-C31'	120.4(2)
C29'-C30'-H30'	119.8
C31'-C30'-H30'	119.8
C30'-C31'-C26'	119.8(2)
C30'-C31'-H31'	120.8
C26'-C31'-H31'	119.4

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	30(1)	30(1)	20(1)	6(1)	2(1)	6(1)
C2	33(1)	28(1)	28(1)	8(1)	1(1)	9(1)
C3	30(1)	23(1)	25(1)	1(1)	1(1)	7(1)
C4	23(1)	26(1)	21(1)	2(1)	0(1)	8(1)
C5	24(1)	23(1)	23(1)	0(1)	2(1)	9(1)
C6	24(1)	28(1)	23(1)	2(1)	1(1)	4(1)
C7	28(1)	37(1)	24(1)	3(1)	5(1)	7(1)
O8	32(1)	28(1)	21(1)	2(1)	2(1)	3(1)
Si9	26(1)	28(1)	20(1)	1(1)	-1(1)	8(1)
C10	43(1)	44(1)	28(1)	2(1)	6(1)	20(1)
C11	43(1)	40(1)	33(1)	6(1)	-6(1)	13(1)
C12	31(1)	38(1)	28(1)	3(1)	2(1)	3(1)
C13	56(2)	53(2)	32(1)	17(1)	3(1)	-1(1)
C14	53(2)	47(2)	45(2)	1(1)	3(1)	-12(1)
C15	37(1)	80(2)	60(2)	11(2)	18(1)	18(1)
C16	24(1)	33(1)	23(1)	-1(1)	-2(1)	12(1)
O17	48(1)	39(1)	33(1)	-2(1)	3(1)	25(1)
C18	20(1)	37(1)	24(1)	2(1)	2(1)	11(1)
C19	36(1)	46(1)	28(1)	-4(1)	4(1)	7(1)
C20	47(2)	63(2)	23(1)	4(1)	8(1)	6(1)
C21	38(1)	56(2)	35(1)	17(1)	7(1)	8(1)
C22	31(1)	40(1)	39(1)	11(1)	7(1)	13(1)
C23	28(1)	37(1)	27(1)	4(1)	6(1)	14(1)
C24	25(1)	21(1)	23(1)	4(1)	1(1)	5(1)
O25	33(1)	35(1)	25(1)	-5(1)	-2(1)	14(1)
C26	24(1)	24(1)	27(1)	7(1)	4(1)	6(1)
C27	31(1)	40(1)	32(1)	4(1)	2(1)	11(1)
C28	30(1)	58(2)	51(2)	9(1)	-3(1)	17(1)

C29	27(1)	44(1)	73(2)	18(1)	10(1)	16(1)
C30	31(1)	31(1)	59(2)	4(1)	16(1)	12(1)
C31	27(1)	24(1)	38(1)	5(1)	7(1)	5(1)
C1'	34(1)	39(1)	19(1)	-1(1)	1(1)	14(1)
C2'	32(1)	32(1)	25(1)	-4(1)	3(1)	10(1)
C3'	30(1)	27(1)	24(1)	2(1)	3(1)	13(1)
C4'	24(1)	23(1)	20(1)	2(1)	1(1)	6(1)
C5'	27(1)	21(1)	21(1)	5(1)	3(1)	9(1)
C6'	25(1)	33(1)	23(1)	1(1)	3(1)	10(1)
C7'	37(1)	41(1)	19(1)	4(1)	7(1)	12(1)
O8'	38(1)	44(1)	21(1)	2(1)	-3(1)	22(1)
Si9'	39(1)	31(1)	23(1)	1(1)	-8(1)	12(1)
C10'	67(2)	45(2)	45(2)	-12(1)	-23(1)	19(1)
C11'	65(2)	49(2)	28(1)	10(1)	5(1)	17(1)
C12'	31(1)	38(1)	37(1)	6(1)	-4(1)	12(1)
C13'	51(2)	50(2)	39(1)	-3(1)	9(1)	12(1)
C14'	53(2)	50(2)	93(2)	11(2)	26(2)	1(1)
C15'	77(2)	137(3)	60(2)	4(2)	-13(2)	77(2)
C16'	22(1)	26(1)	22(1)	4(1)	-2(1)	1(1)
O17'	42(1)	25(1)	28(1)	5(1)	-1(1)	6(1)
C18'	21(1)	31(1)	21(1)	1(1)	2(1)	2(1)
C19'	29(1)	45(1)	25(1)	8(1)	3(1)	9(1)
C20'	34(1)	62(2)	22(1)	5(1)	6(1)	12(1)
C21'	33(1)	50(2)	30(1)	-8(1)	9(1)	5(1)
C22'	47(1)	35(1)	39(1)	0(1)	15(1)	10(1)
C23'	39(1)	31(1)	25(1)	3(1)	9(1)	7(1)
C24'	26(1)	23(1)	20(1)	1(1)	3(1)	9(1)
O25'	32(1)	33(1)	26(1)	9(1)	-2(1)	3(1)
C26'	25(1)	24(1)	25(1)	1(1)	5(1)	7(1)
C27'	31(1)	35(1)	29(1)	5(1)	2(1)	4(1)
C28'	31(1)	45(1)	36(1)	2(1)	-1(1)	-1(1)
C29'	31(1)	35(1)	46(2)	1(1)	10(1)	-4(1)
C30'	37(1)	33(1)	41(1)	12(1)	12(1)	4(1)
C31'	30(1)	30(1)	31(1)	6(1)	5(1)	7(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 1.

	x	y	z	U(eq)
H1	4307	6321	5713	33
H2A	3247	7073	6613	36
H2B	4545	7925	6385	36
H3	5294	7970	7473	32
H4	3577	5687	7441	28
H5	5368	4987	7484	28
H6	6999	7185	7234	31
H7A	6470	6649	6150	36
H7B	5856	5404	6374	36
H10A	4352	3867	5196	55
H10B	4061	3033	5760	55
H10C	3133	2714	5095	55
H11A	2598	5314	4837	58
H11B	1303	4223	4713	58
H11C	1326	5265	5195	58
H13A	988	2357	6990	76
H13B	2387	2496	6730	76
H13C	2100	3576	7055	76
H14A	-195	1460	5910	82
H14B	150	2135	5288	82
H14C	1201	1613	5643	82
H15A	-676	3190	6427	87
H15B	419	4417	6477	87
H15C	-331	3842	5797	87
H19	3702	6814	9577	46
H20	3171	5421	10346	56
H21	2832	3459	10048	53
H22	2956	2875	8968	43
H23	3494	4257	8198	36
H27	8722	6532	9130	42

H28	10558	5801	9222	55
H29	10700	4417	8436	55
H30	9111	3861	7519	47
H31	7278	4618	7405	36
H1'	5789	1628	9328	36
H2'A	6723	2961	8472	35
H2'B	5455	3110	8763	35
H3'	4610	2769	7676	31
H4'	6320	1407	7565	27
H5'	4550	-248	7499	27
H6'	2935	1063	7874	32
H7'A	3575	824	8936	38
H7'B	4152	-101	8631	38
H10D	7986	2023	10014	82
H10E	9347	1733	9994	82
H10F	8971	2567	9504	82
H11D	6473	-1309	9264	71
H11E	7587	-856	9860	71
H11F	6266	-491	9825	71
H13D	8839	-724	7762	71
H13E	7763	-1294	8227	71
H13F	7552	-322	7793	71
H14D	10312	1293	7993	101
H14E	9030	1704	8023	101
H14F	10184	2043	8606	101
H15D	10632	-234	8680	125
H15E	10525	472	9315	125
H15F	9579	-810	9155	125
H19'	6203	2496	5502	40
H20'	6779	1421	4672	48
H21'	7171	-337	4874	47
H22'	7036	-1007	5918	49
H23'	6459	64	6752	39

H27'	1050	-619	5956	40
H28'	-856	-2270	5921	49
H29'	-1056	-3531	6754	48
H30'	610	-3111	7649	45
H31'	2540	-1465	7686	37

Figure 1. View of molecule 1 of **5b** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity.

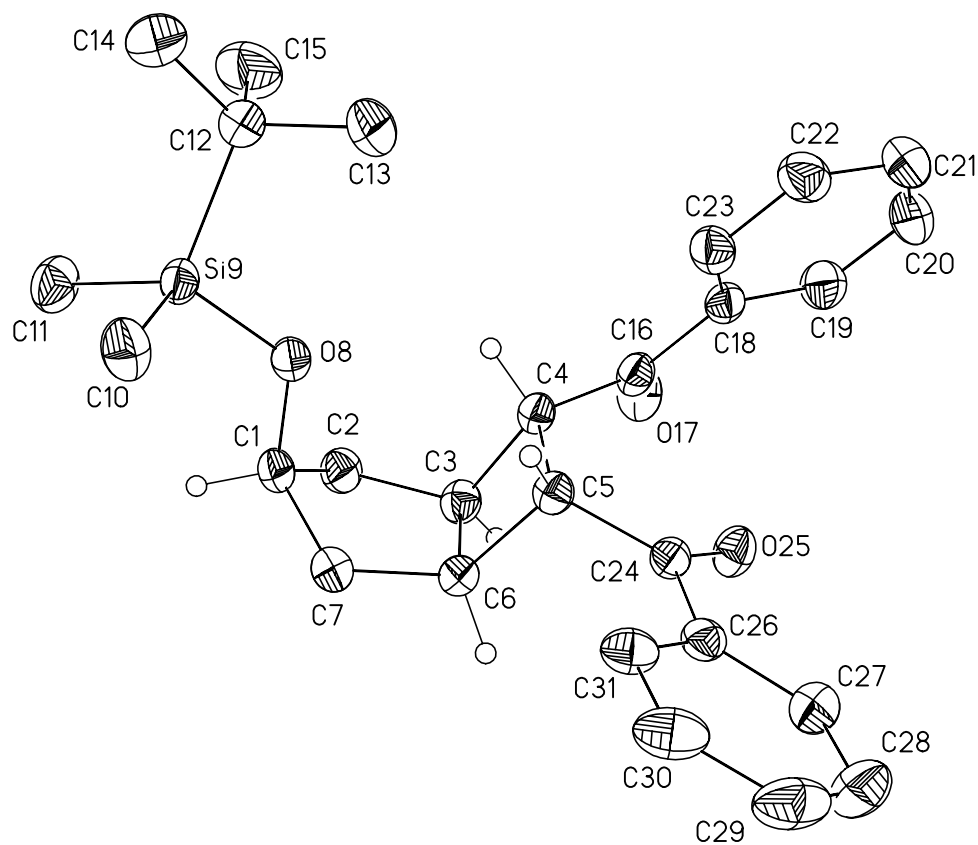


Figure 2. View of molecule 2 of **5b** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity.

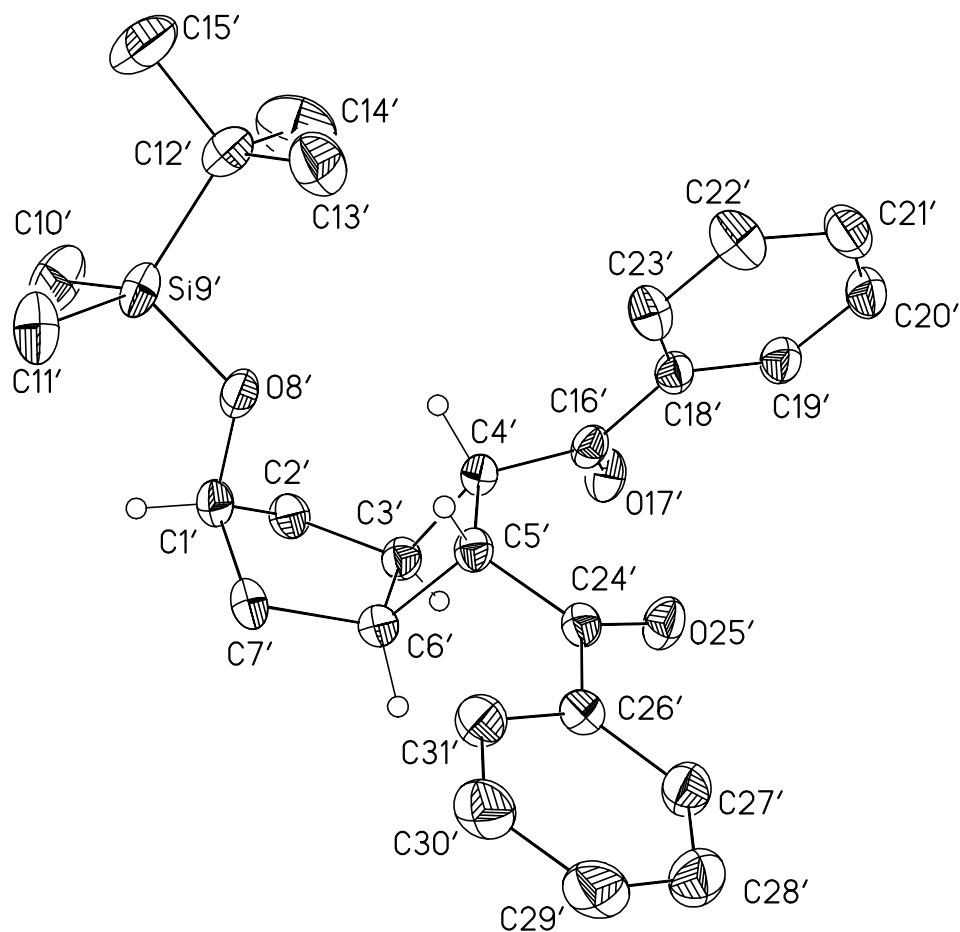


Figure 3. Unit cell packing diagram for **5b**. The view is approximately down the **a** axis. Molecules **2** are shown in wireframe form while molecules **5b** are displayed as ball-and-stick.

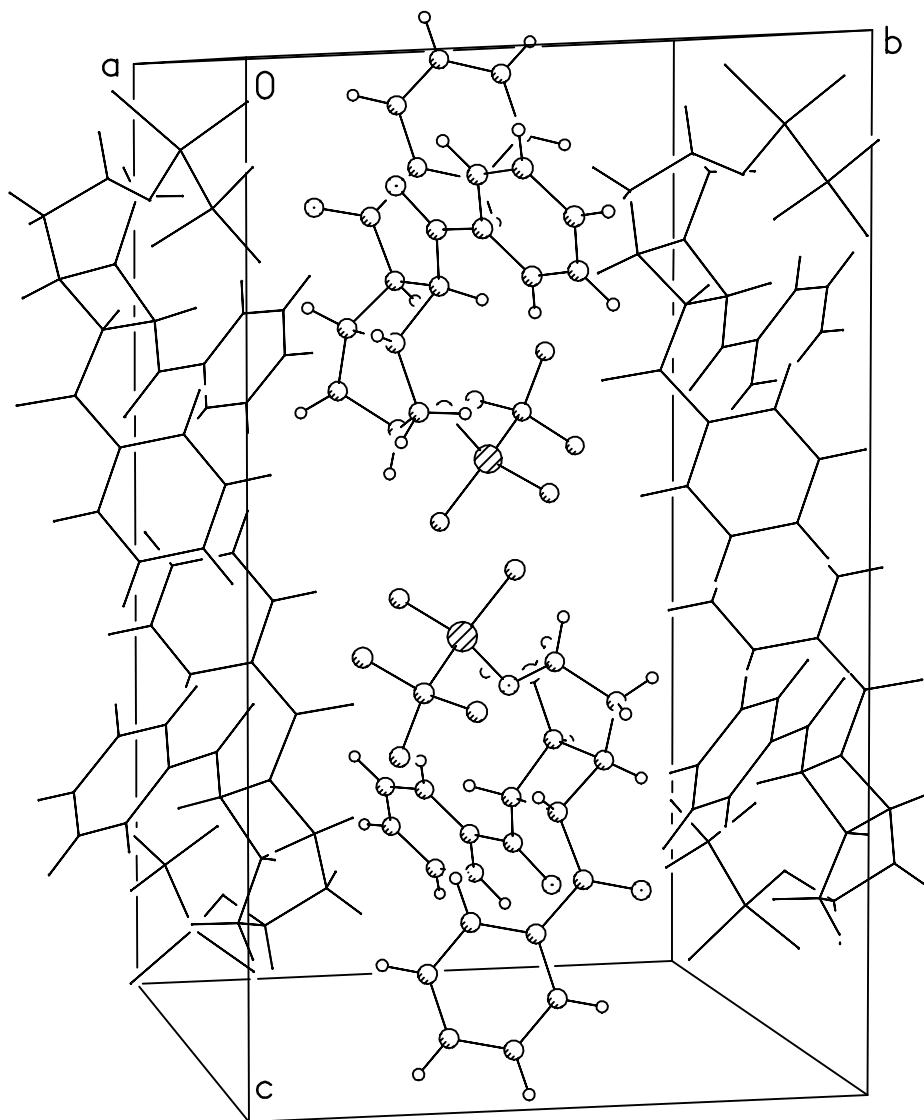


Figure 4. View of the fit by least-squares of selected atoms of molecule **5b** (dashed lines) onto the equivalent atoms of molecule 2. The seven atoms of the fused ring system were used in the fit.

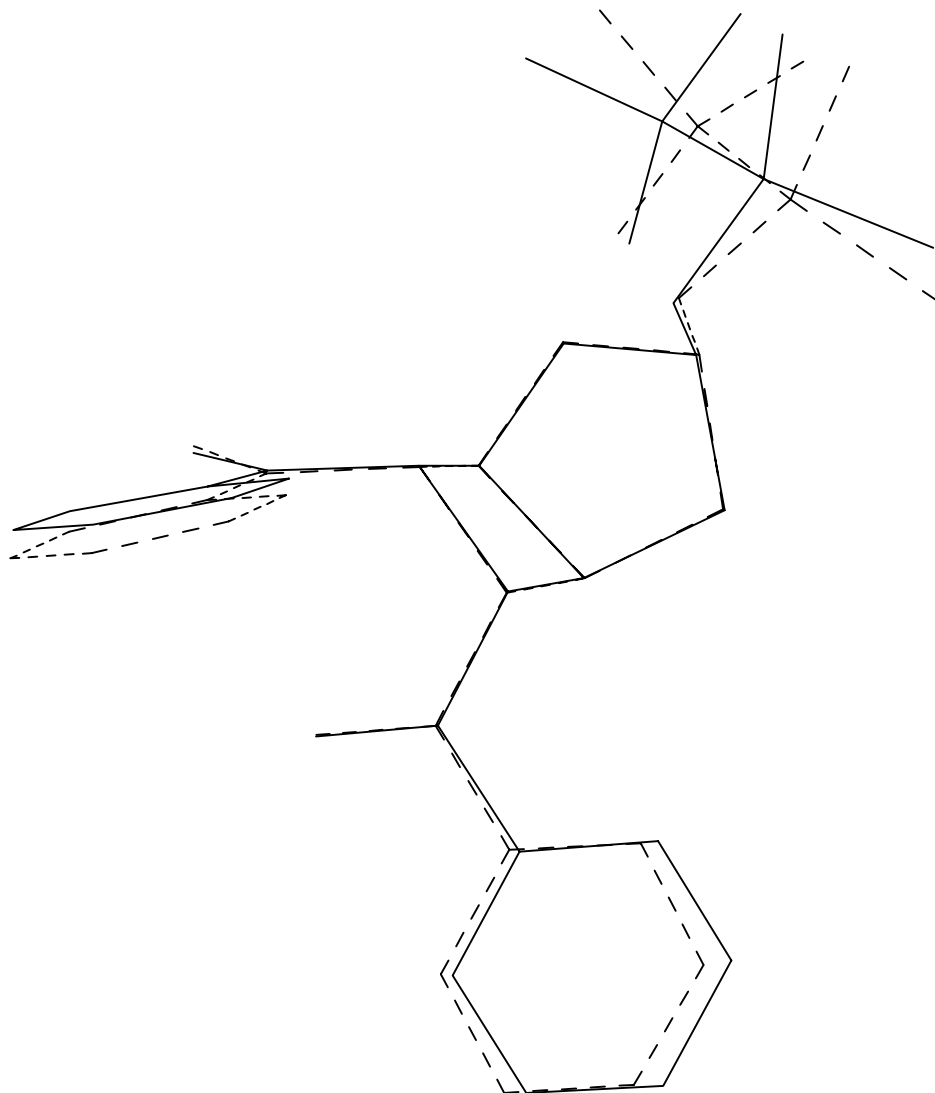


Table 6. Observed and calculated structure factors for 1

Page 1

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	0	0	64	58	8	-12	4	0	110	122	8	2	7	0	148	159	3	4	11	0	46	15	26	-4	-12	1	33	27	32
3	0	0	1428	1440	19	-11	4	0	69	63	17	3	7	0	171	175	3	5	11	0	57	38	24	-3	-12	1	37	39	23
4	0	0	668	665	7	-10	4	0	47	55	3	4	7	0	53	3	9	6	11	0	0	24	1	-2	-12	1	109	97	7
5	0	0	467	464	7	-9	4	0	111	116	3	5	7	0	49	69	11	-11	12	0	90	80	9	-1	-12	1	161	154	7
6	0	0	131	130	4	-8	4	0	56	54	7	6	7	0	133	124	5	-10	12	0	26	4	26	0	-12	1	97	117	8
7	0	0	71	73	3	-7	4	0	132	122	2	7	7	0	37	25	20	-9	12	0	27	35	27	1	-12	1	67	77	11
8	0	0	55	57	10	-6	4	0	121	122	1	8	7	0	76	64	8	-8	12	0	33	34	18	2	-12	1	279	278	6
9	0	0	387	401	4	-5	4	0	597	599	5	9	7	0	0	7	1	-7	12	0	48	43	17	3	-12	1	283	273	4
10	0	0	82	63	5	-4	4	0	392	385	4	-13	8	0	96	108	14	-6	12	0	76	89	5	4	-12	1	103	100	5
11	0	0	42	33	14	-3	4	0	323	321	3	-12	8	0	23	12	23	-5	12	0	160	167	3	5	-12	1	236	242	11
12	0	0	146	152	7	-2	4	0	232	227	2	-11	8	0	53	39	13	-4	12	0	142	134	5	6	-12	1	88	85	5
-13	1	0	115	111	11	-1	4	0	527	526	5	-10	8	0	180	179	5	-3	12	0	52	43	10	7	-12	1	36	32	18
-12	1	0	76	76	9	0	4	0	182	187	4	-9	8	0	75	75	3	-2	12	0	279	276	5	8	-12	1	47	44	11
-11	1	0	44	45	34	1	4	0	87	90	8	-8	8	0	101	111	3	-1	12	0	28	27	28	9	-12	1	23	34	23
-10	1	0	93	94	6	2	4	0	458	447	7	-7	8	0	114	111	6	0	12	0	58	50	12	10	-12	1	81	80	16
-9	1	0	150	149	6	3	4	0	395	418	5	-6	8	0	52	43	11	1	12	0	43	45	19	11	-12	1	0	4	1
-8	1	0	150	149	3	4	4	0	52	38	6	-5	8	0	36	20	7	2	12	0	138	124	6	-6	-11	1	53	80	20
-7	1	0	50	40	5	5	4	0	367	363	4	-4	8	0	27	21	12	3	12	0	50	78	18	-5	-11	1	76	60	11
-6	1	0	44	46	4	6	4	0	222	223	3	-3	8	0	181	182	2	4	12	0	65	48	21	-4	-11	1	117	120	6
-5	1	0	239	230	3	7	4	0	169	169	3	-2	8	0	284	288	2	5	12	0	45	60	45	-3	-11	1	71	40	13
-4	1	0	558	564	7	8	4	0	88	105	5	-1	8	0	27	10	11	-10	13	0	22	19	21	-2	-11	1	132	134	6
-3	1	0	778	797	9	9	4	0	39	28	10	0	8	0	177	175	2	-9	13	0	119	135	13	-1	-11	1	202	202	5
-2	1	0	266	270	4	10	4	0	163	171	6	1	8	0	201	202	6	-8	13	0	104	108	5	0	-11	1	0	8	1
1	1	0	109	105	1	11	4	0	103	95	13	2	8	0	60	48	6	-7	13	0	98	105	6	1	-11	1	193	190	4
2	1	0	541	553	6	-13	5	0	0	17	1	3	8	0	335	335	4	-6	13	0	44	40	11	2	-11	1	201	199	6
3	1	0	599	611	6	-12	5	0	172	119	21	4	8	0	56	58	10	-5	13	0	104	113	5	3	-11	1	151	150	9
4	1	0	568	566	5	-11	5	0	0	49	1	5	8	0	137	157	4	-4	13	0	0	10	1	4	-11	1	24	27	24
5	1	0	34	17	4	-10	5	0	71	81	2	6	8	0	154	138	6	-3	13	0	47	20	22	5	-11	1	107	120	9
6	1	0	143	150	2	-9	5	0	21	14	21	7	8	0	75	86	12	-2	13	0	69	28	16	6	-11	1	22	14	21
7	1	0	195	208	2	-8	5	0	284	290	2	8	8	0	93	100	10	-1	13	0	59	13	20	7	-11	1	57	51	9
8	1	0	206	202	3	-7	5	0	572	583	5	-12	9	0	98	82	17	0	13	0	36	10	35	8	-11	1	66	42	34
9	1	0	169	167	2	-6	5	0	373	384	3	-11	9	0	110	92	10	1	13	0	65	76	10	9	-11	1	46	62	12
10	1	0	55	50	10	-5	5	0	68	66	3	-10	9	0	91	94	10	2	13	0	0	49	1	10	-11	1	102	116	10
11	1	0	109	123	8	-4	5	0	63	76	3	-9	9	0	188	189	3	3	13	0	0	2	1	11	-11	1	95	18	18
12	1	0	74	39	25	-3	5	0	0	10	1	-8	9	0	184	189	2	-9	14	0	80	25	17	12	-11	1	51	51	40
-13	2	0	36	11	30	-2	5	0	160	166	2	-7	9	0	27	13	26	-8	14	0	67	75	13	-7	-10	1	0	52	1
-12	2	0	16	40	15	-1	5	0	121	122	4	-6	9	0	152	144	13	-7	14	0	57	22	15	-6	-10	1	53	59	13
-11	2	0	65	64	16	0	5	0	38	49	6	-5	9	0	247	246	2	-6	14	0	53	47	12	-5	-10	1	23	6	23
-10	2	0	110	117	4	1	5	0	45	59	5	-4	9	0	230	230	6	-5	14	0	55	43	14	-4	-10	1	53	45	21
-9	2	0	235	236	3	2	5	0	197	180	2	-3	9	0	102	107	3	-4	14	0	253	259	4	-3	-10	1	136	131	6
-8	2	0	110	109	3	3	5	0	37	43	7	-2	9	0	189	187	3	-3	14	0	102	107	8	-2	-10	1	151	151	5
-7	2	0	96	85	1	4	5	0	368	366	7	-1	9	0	181	177	3	-2	14	0	0	3	1	-1	-10	1	7	18	7
-6	2	0	206	215	3	5	5	0	289	282	4	0	9	0	218	223	3	-1	14	0	0	23	1	0	-10	1	322	329	4
-5	2	0	80	97	2	6	5	0	49	21	12	1	9	0	250	238	3	0	14	0	82	41	15	1	-10	1	136	132	9
-4	2	0	151	161	1	7	5	0	0	3	1	2	9	0	44	23	18	1	14	0	0	2	1	2	-10	1	144	122	102
-3	2	0	765	774	7	8	5	0	87	86	5	3	9	0	235	238	3	2	14	0	13	18	12	3	-10	1	52	14	8
-2	2	0	1439	1411	27	9	5	0	43	3	16	4	9	0	59	45	12	-7	15	0	78	74	12	4	-10	1	179	173	4
-1	2	0	939	963	10	10	5	0	127	113	6	5	9	0	59	50	10	-6	15	0	86	57	13	5	-10	1	52	52	8
0	2	0	653	680	7	-13	6	0	101	105	16	6	9	0	0	0	1	-5	15	0	113	88	10	6	-10	1	254	255	3
1	2	0	1274	1276	16	-12	6	0	68	81	18	7	9	0	70	62	11	-4	15	0	71	60	18	7	-10	1	43	29	8
2	2	0	1006	1000	9	-11	6	0	48	36	47	8	9	0	101	90	8	-3	15	0	43	2	43	8	-10	1	70	73	5
3	2	0	215	215	3	-10	6	0	88	91	5	-12	10	0	65	87	27	-2	15	0	58	15	57	9	-10	1	23	37	23
4	2	0	227	232	2	-9	6	0	156	159	2	-11	10	0	96	108	9	-1	15	0	0	27	1	10	-10	1	47	35	47
5	2	0	199	199	2	-8	6	0	32	27	7	-10	10	0	0	46	1	1	-15	1	54	65	33	11	-10	1	106	110	15
6	2	0	270	270	2	-7	6	0	251	241	5	-9	10	0	91	82	4	2	-15	1	63	56	21	12	-10	1	113	129	9
7	2	0	152	154	2	-6	6	0	145	149	6	-8	10	0	84	86	4	3	-15	1	27	11	27	-8	-9	1	118	108	9
8	2	0	253	248	4	-5	6	0	74	74	2	-7	10	0	35	34	10	4	-15	1	63	13	20	-7	-9	1	69	21	19
9	2	0	436	433	6	-4	6	0	73	69	3	-6	10	0	163	164	2	5	-15	1	90	60	9	-6	-9	1	122	122	6
10	2	0	25	47	25	-3	6	0	274	285	3	-5	10	0	30	13	20	6	-15	1	72	54	21	-5	-9	1	33	24	32
11	2	0	59	37																									

Table 6. Observed and calculated structure factors for 1

Page 2

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
3	-8	1	208	212	2	-5	-4	1	28	14	17	8	-1	1	95	93	2	-4	3	1	481	496	4	9	6	1	30	33	30
4	-8	1	0	16	1	-4	-4	1	72	82	3	9	-1	1	358	351	4	-3	3	1	748	753	6	10	6	1	56	77	12
5	-8	1	178	182	2	-3	-4	1	413	399	4	10	-1	1	28	3	27	-2	3	1	38	43	7	-13	7	1	101	115	11
6	-8	1	219	222	2	-2	-4	1	506	480	5	11	-1	1	110	106	6	-1	3	1	271	264	2	-12	7	1	87	71	47
7	-8	1	76	78	9	-1	-4	1	236	231	3	12	-1	1	0	13	1	0	3	1	176	168	2	-11	7	1	30	20	30
8	-8	1	0	2	1	0	-4	1	156	155	5	13	-1	1	35	37	35	1	3	1	130	146	2	-10	7	1	24	46	24
9	-8	1	221	206	8	1	-4	1	297	300	6	-12	0	1	73	25	28	2	3	1	273	279	5	-9	7	1	254	265	3
10	-8	1	74	80	6	2	-4	1	279	292	3	-11	0	1	28	17	28	3	3	1	321	314	3	-8	7	1	64	69	3
11	-8	1	64	78	23	3	-4	1	223	232	2	-10	0	1	213	214	3	4	3	1	275	281	3	-7	7	1	67	75	5
12	-8	1	99	18	42	4	-4	1	435	433	4	-9	0	1	169	164	3	5	3	1	344	340	4	-6	7	1	52	44	4
13	-8	1	91	92	12	5	-4	1	51	36	3	-8	0	1	19	35	19	6	3	1	24	22	24	-5	7	1	203	201	2
-9	-7	1	166	152	6	6	-4	1	291	280	5	-7	0	1	65	75	2	7	3	1	367	368	4	-4	7	1	253	248	2
-8	-7	1	45	14	13	7	-4	1	54	51	3	-6	0	1	163	169	2	8	3	1	94	101	5	-3	7	1	150	147	2
-7	-7	1	70	69	10	8	-4	1	357	367	3	-5	0	1	159	159	2	9	3	1	26	23	11	-2	7	1	51	73	8
-6	-7	1	64	67	14	9	-4	1	91	97	4	-4	0	1	120	124	2	10	3	1	56	11	15	-1	7	1	20	0	19
-5	-7	1	130	130	5	10	-4	1	58	52	4	-3	0	1	65	67	1	11	3	1	78	75	10	0	7	1	122	97	2
-4	-7	1	23	4	23	11	-4	1	78	62	18	-2	0	1	92	94	4	-13	4	1	72	68	15	1	7	1	157	155	4
-3	-7	1	396	401	3	12	-4	1	95	82	26	2	0	1	179	185	4	-12	4	1	125	103	10	2	7	1	779	782	6
-2	-7	1	152	158	3	13	-4	1	39	20	38	3	0	1	77	80	6	-11	4	1	0	35	1	3	7	1	203	194	2
-1	-7	1	89	58	6	-11	-3	1	75	62	34	4	0	1	74	72	3	-10	4	1	113	127	3	4	7	1	141	144	3
0	-7	1	419	424	4	-10	-3	1	86	75	8	5	0	1	76	70	17	-9	4	1	130	138	1	5	7	1	51	52	6
1	-7	1	74	93	4	-9	-3	1	98	101	4	6	0	1	31	31	5	-8	4	1	365	356	3	6	7	1	30	28	15
2	-7	1	0	41	1	-8	-3	1	235	231	3	7	0	1	72	78	7	-7	4	1	18	16	11	7	7	1	39	0	14
3	-7	1	132	127	4	-7	-3	1	223	225	3	8	0	1	63	62	14	-6	4	1	251	267	2	8	7	1	131	130	7
4	-7	1	57	46	7	-6	-3	1	196	198	3	9	0	1	72	72	10	-5	4	1	643	629	5	9	7	1	38	56	21
5	-7	1	11	11	10	-5	-3	1	7	24	7	10	0	1	86	87	6	-4	4	1	187	177	1	-13	8	1	0	50	1
6	-7	1	539	530	5	-4	-3	1	23	1	23	11	0	1	142	136	8	-3	4	1	300	295	2	-12	8	1	64	27	24
7	-7	1	110	113	2	-3	-3	1	914	902	8	12	0	1	97	98	13	-2	4	1	341	342	3	-11	8	1	130	121	9
8	-7	1	37	55	11	-2	-3	1	218	220	2	-13	1	1	91	61	36	-1	4	1	51	63	3	-10	8	1	107	109	6
9	-7	1	43	43	22	-1	-3	1	84	85	2	-12	1	1	74	68	17	0	4	1	42	50	4	-9	8	1	102	107	3
10	-7	1	117	126	5	0	-3	1	348	329	4	-11	1	1	97	96	7	1	4	1	42	51	8	-8	8	1	91	85	2
11	-7	1	0	39	1	1	-3	1	87	15	4	-10	1	1	177	172	2	2	4	1	235	235	2	-7	8	1	279	280	3
12	-7	1	62	13	47	2	-3	1	82	90	6	-9	1	1	55	59	5	3	4	1	31	38	8	-6	8	1	211	200	5
13	-7	1	82	84	14	3	-3	1	57	55	2	-8	1	1	40	47	19	4	4	1	87	73	3	-5	8	1	195	202	4
-10	-6	1	118	117	6	4	-3	1	277	276	2	-7	1	1	256	255	3	5	4	1	0	1	1	-4	8	1	87	80	4
-9	-6	1	59	50	16	5	-3	1	291	280	3	-6	1	1	546	543	5	6	4	1	102	107	4	-3	8	1	77	85	4
-8	-6	1	10	30	10	6	-3	1	232	223	2	-5	1	1	37	38	11	7	4	1	72	86	4	-2	8	1	51	60	7
-7	-6	1	49	58	13	7	-3	1	173	174	2	-4	1	1	692	697	6	8	4	1	87	84	4	-1	8	1	217	224	2
-6	-6	1	82	84	8	8	-3	1	67	52	10	-3	1	1	976	1002	13	9	4	1	149	156	5	0	8	1	197	188	2
-5	-6	1	111	120	4	9	-3	1	154	160	8	-2	1	1	485	514	5	10	4	1	57	23	15	1	8	1	200	196	5
-4	-6	1	75	61	5	10	-3	1	95	92	12	1	1	1	190	192	2	11	4	1	72	40	13	2	8	1	61	62	5
-3	-6	1	37	2	9	11	-3	1	150	160	10	2	1	1	667	674	9	-13	5	1	83	97	25	3	8	1	28	17	13
-2	-6	1	87	94	3	12	-3	1	45	31	22	3	1	1	403	414	8	-12	5	1	59	85	15	4	8	1	100	92	4
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0	-6	1	291	287	3	-11	-2	1	160	159	6	5	1	1	487	493	9	-10	5	1	113	125	3	6	8	1	136	130	5
1	-6	1	125	120	5	-10	-2	1	223	225	5	6	1	1	332	339	3	-9	5	1	0	2	1	7	8	1	23	16	23
2	-6	1	406	381	9	-9	-2	1	163	158	3	7	1	1	47	42	7	-8	5	1	56	44	4	8	8	1	39	5	29
3	-6	1	350	323	7	-8	-2	1	59	46	6	8	1	1	222	231	3	-7	5	1	96	96	9	-12	9	1	62	30	26
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6	-6	1	402	395	5	-5	-2	1	118	129	2	11	1	1	48	45	33	-4	5	1	592	573	5	-9	9	1	197	198	3
7	-6	1	540	554	6	-4	-2	1	346	358	4	12	1	1	64	49	23	-3	5	1	145	158	4	-8	9	1	51	39	7
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10	-6	1	37	35	9	-1	-2	1	1525	1515	27	-11	2	1	18	9	17	0	5	1	110	105	2	-5	9	1	159	160	3
11	-6	1	116	114	11	0	-2	1	560	579	7	-10	2	1	317	318	3	1	5	1	203	212	2	-4	9	1	192	180	2
12	-6	1	101	93	11	1	-2	1	1546	1522	17	-9	2	1	260	254	3	2	5	1	280	270	3	-3	9	1	89	91	3
13	-6	1	65	5	40	2	-2	1	1172	1188	15	-8	2	1	0	16	1	3	5	1	492	495	5	-2	9	1	188	186	3
-10	-5	1	82	49	10	3	-2	1	78	90	3	-7	2	1	396	398	3	4	5	1	339	345	3	-1	9	1	99	100	3
-9	-5	1	80	74	7	4	-2	1	537	535	5	-6	2	1	76	87	1	5	5	1	0								

Table 6. Observed and calculated structure factors for 1

Page 3

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
7	10	1	53	54	30	9	-14	2	73	36	16	9	-9	2	48	55	9	3	-5	2	197	192	2	-12	-1	2	117	99	12
-12	11	1	37	9	36	-3	-13	2	97	66	10	10	-9	2	39	21	26	4	-5	2	593	573	5	-11	-1	2	79	45	14
-11	11	1	0	0	1	-2	-13	2	103	107	9	11	-9	2	94	72	11	5	-5	2	404	412	3	-10	-1	2	0	27	1
-10	11	1	39	51	38	-1	-13	2	97	80	8	12	-9	2	102	73	14	6	-5	2	146	129	2	-9	-1	2	0	1	1
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-8	11	1	134	126	8	1	-13	2	0	23	1	-8	-8	2	95	81	7	8	-5	2	104	99	3	-7	-1	2	0	23	1
-7	11	1	196	197	3	2	-13	2	132	129	5	-7	-8	2	61	49	13	9	-5	2	293	298	3	-6	-1	2	92	88	3
-6	11	1	25	5	25	3	-13	2	88	86	10	-6	-8	2	103	110	7	10	-5	2	52	42	4	-5	-1	2	233	224	2
-5	11	1	167	163	3	4	-13	2	108	105	6	-5	-8	2	78	83	10	11	-5	2	31	72	31	-4	-1	2	315	307	4
-4	11	1	0	21	1	5	-13	2	127	125	5	-4	-8	2	111	100	4	12	-5	2	53	36	26	-3	-1	2	197	199	2
-3	11	1	53	31	11	6	-13	2	79	65	8	-3	-8	2	253	251	4	13	-5	2	55	82	38	-2	-1	2	118	99	2
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0	11	1	42	62	14	9	-13	2	0	14	1	0	-8	2	143	136	3	-9	-4	2	59	64	7	2	-1	2	321	322	5
1	11	1	0	30	1	10	-13	2	0	32	1	1	-8	2	175	184	3	-8	-4	2	80	79	5	3	-1	2	493	491	7
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1	12	1	43	16	19	-6	-11	2	0	31	1	-4	-7	2	246	241	3	10	-4	2	59	52	9	-4	0	2	111	112	1
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h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
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0	-7	9	16	35	15	-4	-3	9	470	472	4	-11	1	9	51	36	14	7	4	9	31	17	19	7	8	9	0	32	1
1	-7	9	358	370	5	-3	-3	9	318	300	3	-10	1	9	90	80	5	8	4	9	41	1	34	-12	9	9	25	9	24
2	-7	9	93	83	4	-2	-3	9	8	46	7	-9	1	9	151	149	4	9	4	9	37	34	24	-11	9	9	175	180	6
3	-7	9	217	220	3	-1	-3	9	379	356	4	-8	1	9	123	118	4	10	4	9	13	29	12	-10	9	9	0	13	1
4	-7	9	263	265	4	0	-3	9	108	87	11	-7	1	9	194	188	2	-12	5	9	89	105	10	-9	9	9	16	46	16
5	-7	9	76	87	8	1	-3	9	144	145	2	-6	1	9	375	365	4	-11	5	9	96	100	9	-8	9	9	63	64	5
6	-7	9	156	148	5	2	-3	9	223	227	4	-5	1	9	244	250	3	-10	5	9	81	99	7	-7	9	9	69	76	4
7	-7	9	329	331	4	3	-3	9	46	59	5	-4	1	9	410	408	5	-9	5	9	128	125	3	-6	9	9	168	180	5
8	-7	9	38	21	12	4	-3	9	99	97	3	-3	1	9	296	292	4	-8	5	9	32	37	8	-5	9	9	149	148	2
9	-7	9	84	92	13	5	-3	9	433	429	5	-2	1	9	135	127	4	-7	5	9	34	19	12	-4	9	9	135	136	3
10	-7	9	97	96	19	6	-3	9	330	327	3	-1	1	9	122	104	9	-6	5	9	42	44	7	-3	9	9	66	70	6
11	-7	9	140	82	21	7	-3	9	77	73	8	0	1	9	279	278	4	-5	5	9	67	75	2	-2	9	9	95	90	8
12	-7	9	126	88	12	8	-3	9	251	264	6	1	1	9	135	124	3	-4	5	9	31	25	6	-1	9	9	91	90	6
-9	-6	9	52	44	19	9	-3	9	178	182	10	2	1	9	187	189	2	-3	5	9	125	114	4	0	9	9	171	178	4
-8	-6	9	72	81	7	10	-3	9	0	50	1	3	1	9	144	151	2	-2	5	9	214	204	3	1	9	9	250	246	4
-7	-6	9	53	47	8	11	-3	9	56	22	29	4	1	9	518	531	6	-1	5	9	72	98	4	2	9	9	165	178	3
-6	-6	9	50	40	8	12	-3	9	46	6	46	5	1	9	414	415	5	0	5	9	155	141	5	3	9	9	0	21	1
-5	-6	9	0	0	1	-11	-2	9	39	10	38	6	1	9	172	167	6	1	5	9	55	61	5	4	9	9	67	40	12
-4	-6	9	223	231	2	-10	-2	9	76	86	6	7	1	9	199	216	6	2	5	9	0	13	1	5	9	9	33	28	32
-3	-6	9	135	148	4	-9	-2	9	13	6	12	8	1	9	46	6	18	3	5	9	34	30	11	6	9	9	67	85	13
-2	-6	9	19	37	19	-8	-2	9	261	259	3	9	1	9	67	75	8	4	5	9	189	190	2	-11	10	9	125	130	6
-1	-6	9	171	170	4	-7	-2	9	298	287	4	10	1	9	162	171	5	5	5	9	376	374	4	-10	10	9	102	109	7
0	-6	9	42	47	10	-6	-2	9	155	150	2	11	1	9	100	83	11	6	5	9	110	119	3	-9	10	9	0	5	1
1	-6	9	142	142	2	-5	-2	9	142	137	2	-11	2	9	173	172	6	7	5	9	135	132	5	-8	10	9	107	110	5
2	-6	9	22	27	21	-4	-2	9	230	239	2	-10	2	9	200	195	3	8	5	9	66	67	11	-7	10	9	22	11	21
3	-6	9	88	87	9	-3	-2	9	169	158	5	-9	2	9	31	43	6	9	5	9	66	83	13	-6	10	9	26	24	25
4	-6	9	54	41	8	-2	-2	9	65	51	3	-8	2	9	40	41	5	-12	6	9	162	52	42	-5	10	9	154	148	2
5	-6	9	62	62	6	-1	-2	9	363	363	3	-7	2	9	58	55	12	-11	6	9	86	65	10	-4	10	9	41	37	41
6	-6	9	66	24	65	0	-2	9	307	303	5	-6	2	9	39	32	5	-10	6	9	75	72	12	-3					

Table 6. Observed and calculated structure factors for 1

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h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
1	6	10	288	294	3	-7	11	10	102	107	7	-5	-10	11	85	40	12	2	-6	11	52	76	28	0	-2	11	126	117	4
2	6	10	168	162	5	-6	11	10	70	69	7	-4	-10	11	36	65	35	3	-6	11	229	226	12	1	-2	11	14	26	14
3	6	10	405	404	5	-5	11	10	37	33	12	-3	-10	11	60	77	21	4	-6	11	56	69	9	2	-2	11	45	56	11
4	6	10	7	26	7	-4	11	10	133	131	6	-2	-10	11	64	52	14	5	-6	11	109	112	8	3	-2	11	0	8	1
5	6	10	86	93	4	-3	11	10	88	82	7	-1	-10	11	34	32	34	6	-6	11	255	263	4	4	-2	11	175	178	4
6	6	10	76	74	8	-2	11	10	23	43	23	0	-10	11	237	228	4	7	-6	11	103	102	9	5	-2	11	93	93	3
7	6	10	169	176	6	-1	11	10	50	59	15	1	-10	11	57	62	12	8	-6	11	73	58	4	6	-2	11	72	65	5
8	6	10	39	72	38	0	11	10	100	101	7	2	-10	11	43	40	13	9	-6	11	60	73	25	7	-2	11	75	84	40
-12	7	10	22	0	21	1	11	10	145	143	6	3	-10	11	261	265	4	10	-6	11	71	78	13	8	-2	11	103	101	6
-11	7	10	118	62	26	2	11	10	50	27	23	4	-10	11	172	179	11	11	-6	11	36	15	35	9	-2	11	165	163	9
-10	7	10	145	123	9	3	11	10	120	129	9	5	-10	11	84	92	13	-10	-5	11	39	47	39	10	-2	11	39	3	39
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-8	7	10	41	20	8	-10	12	10	90	90	13	7	-10	11	75	40	11	-8	-5	11	27	4	27	-11	-1	11	200	101	50
-7	7	10	148	160	2	-9	12	10	41	49	41	8	-10	11	141	145	11	-7	-5	11	31	19	31	-10	-1	11	199	193	6
-6	7	10	28	19	8	-8	12	10	37	13	21	9	-10	11	132	127	11	-6	-5	11	79	90	9	-9	-1	11	0	4	1
-5	7	10	222	220	4	-7	12	10	0	6	1	10	-10	11	29	72	29	-5	-5	11	362	356	6	-8	-1	11	34	29	8
-4	7	10	385	379	3	-6	12	10	34	31	27	-7	-9	11	216	208	5	-4	-5	11	253	247	2	-7	-1	11	215	213	4
-3	7	10	69	74	7	-5	12	10	116	122	5	-6	-9	11	45	54	17	-3	-5	11	136	134	7	-6	-1	11	227	230	2
-2	7	10	523	522	4	-4	12	10	0	6	1	-5	-9	11	105	104	7	-2	-5	11	58	48	9	-5	-1	11	202	202	2
-1	7	10	142	135	3	-3	12	10	45	28	16	-4	-9	11	136	159	6	-1	-5	11	448	467	4	-4	-1	11	218	227	3
0	7	10	201	204	2	-2	12	10	94	88	8	-3	-9	11	80	69	11	0	-5	11	160	159	6	-3	-1	11	191	189	4
1	7	10	257	259	3	-1	12	10	162	159	7	-2	-9	11	45	26	19	1	-5	11	130	139	3	-2	-1	11	90	77	2
2	7	10	122	126	5	0	12	10	34	8	33	-1	-9	11	239	249	4	2	-5	11	93	108	4	-1	-1	11	266	272	2
3	7	10	0	26	1	1	12	10	74	59	11	0	-9	11	327	322	7	3	-5	11	252	250	4	0	-1	11	98	103	5
4	7	10	37	39	11	2	12	10	98	98	17	1	-9	11	147	147	6	4	-5	11	83	65	6	1	-1	11	207	204	2
5	7	10	101	87	6	-7	13	10	117	103	12	2	-9	11	314	319	5	5	-5	11	226	210	5	2	-1	11	113	115	2
6	7	10	101	69	11	-6	13	10	41	14	16	3	-9	11	95	100	7	6	-5	11	172	159	7	3	-1	11	266	271	7
7	7	10	46	31	17	-5	13	10	15	10	14	4	-9	11	219	235	6	7	-5	11	201	190	3	4	-1	11	249	267	3
-12	8	10	70	81	19	-4	13	10	28	53	28	5	-9	11	113	131	9	8	-5	11	19	4	19	5	-1	11	312	319	6
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-10	8	10	70	72	13	-2	13	10	106	105	7	7	-9	11	22	14	21	10	-5	11	98	75	19	7	-1	11	293	285	9
-8	8	10	192	204	2	-1	13	10	80	64	11	8	-9	11	7	53	7	11	-5	11	142	114	16	8	-1	11	93	110	12
-7	8	10	0	10	1	0	13	10	99	94	13	9	-9	11	38	16	38	-10	-4	11	59	36	17	9	-1	11	118	99	13
-6	8	10	292	285	4	-6	14	10	16	4	16	10	-9	11	120	95	14	-9	-4	11	31	49	31	10	-1	11	103	98	12
-5	8	10	113	117	9	-5	14	10	71	47	10	11	-9	11	78	56	23	-8	-4	11	66	61	9	11	-1	11	100	107	11
-4	8	10	153	144	4	-4	14	10	7	22	7	-8	-8	11	63	58	11	-7	-4	11	89	90	5	-11	0	11	53	45	20
-3	8	10	88	78	5	-3	14	10	38	30	38	-7	-8	11	59	51	10	-6	-4	11	108	103	3	-10	0	11	69	61	11
-2	8	10	89	78	9	-2	14	10	56	35	28	-6	-8	11	24	7	24	-5	-4	11	62	48	8	-9	0	11	51	50	7
-1	8	10	70	66	5	0	-14	11	0	66	1	-5	-8	11	144	138	5	-4	-4	11	44	37	7	-8	0	11	181	182	2
0	8	10	0	3	1	-1	-14	11	47	43	47	-4	-8	11	93	104	7	-3	-4	11	191	186	2	-7	0	11	234	225	3
1	8	10	105	104	5	2	-14	11	55	24	54	-3	-8	11	0	17	1	-2	-4	11	84	76	3	-6	0	11	47	52	5
2	8	10	308	306	4	3	-14	11	31	33	31	-2	-8	11	0	3	1	-1	-4	11	168	170	2	-5	0	11	154	162	2
3	8	10	149	155	3	4	-14	11	45	25	45	-1	-8	11	114	122	5	0	-4	11	256	244	3	-4	0	11	176	172	3
4	8	10	77	68	8	5	-14	11	28	49	28	0	-8	11	73	83	9	1	-4	11	109	109	3	-3	0	11	0	7	1
5	8	10	162	150	5	6	-14	11	122	97	12	1	-8	11	225	226	3	2	-4	11	94	97	3	-2	0	11	153	155	2
6	8	10	43	9	32	-2	-13	11	49	33	49	2	-8	11	219	221	3	3	-4	11	142	137	3	-1	0	11	220	231	3
7	8	10	0	29	1	-1	-13	11	34	25	33	3	-8	11	44	50	16	4	-4	11	92	68	6	0	0	11	38	17	27
-12	9	10	39	33	39	0	-13	11	40	18	40	4	-8	11	30	20	29	5	-4	11	179	177	3	1	0	11	65	54	7
-11	9	10	82	50	15	-1	-13	11	29	64	29	5	-8	11	148	147	5	6	-4	11	52	49	7	2	0	11	72	68	15
-10	9	10	126	120	9	2	-13	11	58	28	25	6	-8	11	194	186	3	7	-4	11	45	51	7	3	0	11	113	120	2
-9	9	10	30	10	30	3	-13	11	18	32	17	7	-8	11	63	34	62	8	-4	11	71	88	7	4	0	11	136	145	2
-8	9	10	92	92	11	4	-13	11	49	15	18	8	-8	11	224	183	14	9	-4	11	124	111	8	5	0	11	42	57	24
-7	9	10	135	134	2	5	-13	11	69	57	15	9	-8	11	116	100	15	10	-4	11	122	113	10	6	0	11	41	13	28
-6	9	10	4	4	4	6	-13	11	43	8	42	10	-8	11	72	49	16	11	-4	11	155	121	25	7	0	11	52	49	21
-5	9	10	85	89	4	7	-13	11	47	13	36	11	-8	11	0	5	1	-10	-3	11	33	5	32	8	0	11	73	32	15
-4	9	10	216	213	9	8	-13	11	42	25	29	-9	-7	11	103	70	14	-9	-3	11	24	24	24	9	0	11	34	42	34
-3	9	10	27	41	26	-4	-12	11	53	8	42	-8	-7	11	124	113	8	-8	-3	11	236	239	10	10	0	11	66	54	26
-2	9	10	81	90	5	-3	-12	11	0	3	1	-7	-7	11	49	54	15	-7	-3	11	159	158	3	11	0	11	25	4	24
-1	9	10	75	69																									

Table 6. Observed and calculated structure factors for 1

h k l 10Fo 10Fc 10s					h k l 10Fo 10Fc 10s					h k l 10Fo 10Fc 10s					h k l 10Fo 10Fc 10s					h k l 10Fo 10Fc 10s				
1 -4 17 206 192 5	9 0 17 0 26 1	-3 5 17 122 118 4	2 -11 18 35 37 35	2 -5 18 52 75 15																				
2 -4 17 324 321 8	-11 1 17 96 78 28	-2 5 17 27 27 6	3 -11 18 89 93 11	3 -5 18 45 23 22																				
3 -4 17 154 162 8	-10 1 17 36 28 35	-1 5 17 49 52 17	4 -11 18 66 71 18	4 -5 18 101 112 6																				
4 -4 17 199 195 5	-9 1 17 95 84 14	0 5 17 266 267 3	5 -11 18 0 37 1	5 -5 18 31 20 31																				
5 -4 17 125 130 4	-8 1 17 167 172 8	1 5 17 88 91 3	6 -11 18 49 41 27	6 -5 18 124 112 7																				
6 -4 17 115 114 5	-7 1 17 38 50 11	2 5 17 148 151 7	-3 -10 18 106 107 9	7 -5 18 80 88 13																				
7 -4 17 83 51 15	-6 1 17 129 128 12	3 5 17 37 13 16	-2 -10 18 49 75 14	8 -5 18 58 43 22																				
8 -4 17 51 79 38	-5 1 17 237 242 6	4 5 17 31 4 30	-1 -10 18 34 35 34	9 -5 18 93 64 17																				
9 -4 17 191 61 41	-4 1 17 104 109 3	5 5 17 39 21 39	0 -10 18 139 138 7	-9 -4 18 109 95 12																				
-9 -3 17 141 151 16	-3 1 17 27 3 26	6 5 17 0 15 1	1 -10 18 189 196 6	-8 -4 18 0 2 1																				
-8 -3 17 19 19 19	-2 1 17 150 148 6	-10 6 17 44 72 43	2 -10 18 56 58 15	-7 -4 18 74 79 8																				
-7 -3 17 74 75 7	-1 1 17 381 378 6	-9 6 17 49 65 48	3 -10 18 33 52 32	-6 -4 18 47 43 17																				
-6 -3 17 0 29 1	0 1 17 204 202 3	-8 6 17 0 2 1	4 -10 18 43 8 36	-5 -4 18 37 37 36																				
-5 -3 17 39 50 31	1 1 17 274 272 5	-7 6 17 134 122 4	5 -10 18 46 26 31	-4 -4 18 50 63 17																				
-4 -3 17 69 74 7	2 1 17 38 29 37	-6 6 17 165 183 9	6 -10 18 98 69 23	-3 -4 18 50 52 9																				
-3 -3 17 18 24 17	3 1 17 180 172 5	-5 6 17 78 84 6	7 -10 18 52 37 39	-2 -4 18 74 70 16																				
-2 -3 17 57 55 9	4 1 17 120 122 3	-4 6 17 55 45 16	-5 -9 18 132 125 16	-1 -4 18 159 159 5																				
-1 -3 17 100 85 9	5 1 17 113 133 4	-3 6 17 0 11 1	-4 -9 18 26 46 26	0 -4 18 74 85 7																				
0 -3 17 176 177 3	6 1 17 64 65 16	-2 6 17 46 36 30	-3 -9 18 0 13 1	1 -4 18 241 245 6																				
1 -3 17 74 82 7	7 1 17 112 113 7	-1 6 17 182 171 8	-2 -9 18 53 33 17	2 -4 18 34 35 34																				
2 -3 17 382 393 7	8 1 17 62 76 18	0 6 17 125 123 4	-1 -9 18 55 28 18	3 -4 18 118 126 7																				
3 -3 17 310 306 8	-11 2 17 0 0 1	1 6 17 132 128 11	0 -9 18 28 2 28	4 -4 18 0 14 1																				
4 -3 17 342 330 9	-10 2 17 51 56 34	2 6 17 132 121 4	1 -9 18 0 3 1	5 -4 18 143 136 4																				
5 -3 17 196 183 4	-9 2 17 51 43 29	3 6 17 121 106 7	2 -9 18 33 31 32	6 -4 18 152 141 5																				
6 -3 17 47 33 10	-8 2 17 103 86 8	4 6 17 0 32 1	3 -9 18 30 4 30	7 -4 18 122 143 12																				
7 -3 17 0 15 1	-7 2 17 37 20 20	5 6 17 82 22 17	4 -9 18 73 85 11	8 -4 18 117 137 14																				
8 -3 17 44 82 43	-6 2 17 202 202 5	-10 7 17 29 40 29	5 -9 18 0 11 1	9 -4 18 72 11 21																				
9 -3 17 119 123 12	-5 2 17 119 129 9	-9 7 17 0 29 1	6 -9 18 84 7 19	-9 -3 18 0 3 1																				
-10 -2 17 0 16 1	-4 2 17 237 235 2	-8 7 17 90 56 12	7 -9 18 15 59 14	-8 -3 18 192 203 8																				
-9 -2 17 114 123 10	-3 2 17 68 61 23	-7 7 17 69 60 16	-6 -8 18 58 22 40	-7 -3 18 59 65 11																				
-8 -2 17 214 202 8	-2 2 17 256 246 3	-6 7 17 26 5 25	-5 -8 18 72 10 14	-6 -3 18 238 238 6																				
-7 -2 17 42 21 41	-1 2 17 95 87 3	-5 7 17 9 14 9	-4 -8 18 112 109 6	-5 -3 18 76 76 24																				
-6 -2 17 117 120 5	0 2 17 160 143 6	-4 7 17 120 118 8	-3 -8 18 34 18 23	-4 -3 18 37 37 13																				
-5 -2 17 122 123 2	1 2 17 93 96 4	-3 7 17 180 172 4	-2 -8 18 147 164 6	-3 -3 18 179 183 3																				
-4 -2 17 50 49 49	2 2 17 45 21 13	-2 7 17 0 3 1	-1 -8 18 40 0 22	-2 -3 18 197 200 4																				
-3 -2 17 40 43 8	3 2 17 272 274 4	-1 7 17 59 47 11	0 -8 18 243 235 5	-1 -3 18 35 43 21																				
-2 -2 17 201 204 3	4 2 17 203 198 3	0 7 17 0 8 1	1 -8 18 100 104 7	0 -3 18 120 124 10																				
-1 -2 17 291 292 4	5 2 17 34 16 13	1 7 17 101 75 8	2 -8 18 151 137 7	1 -3 18 37 52 11																				
0 -2 17 36 41 17	6 2 17 138 120 137	2 7 17 40 27 40	3 -8 18 28 32 28	2 -3 18 285 287 7																				
1 -2 17 407 409 4	7 2 17 15 20 15	3 7 17 124 90 10	4 -8 18 36 2 35	3 -3 18 43 23 42																				
2 -2 17 51 76 8	8 2 17 0 12 1	4 7 17 43 21 43	5 -8 18 77 73 21	4 -3 18 10 31 10																				
3 -2 17 226 233 6	-11 3 17 0 39 1	5 7 17 135 114 29	6 -8 18 86 67 10	5 -3 18 61 68 12																				
4 -2 17 205 208 5	-10 3 17 0 27 1	-10 8 17 0 35 1	7 -8 18 74 61 24	6 -3 18 62 27 22																				
5 -2 17 77 66 5	-9 3 17 91 11 19	-9 8 17 33 14 32	8 -8 18 47 9 46	7 -3 18 77 4 25																				
6 -2 17 0 14 1	-8 3 17 127 132 11	-8 8 17 97 98 17	-7 -7 18 83 73 18	8 -3 18 114 74 11																				
7 -2 17 124 124 10	-7 3 17 0 49 1	-7 8 17 131 101 15	-6 -7 18 23 10 23	9 -3 18 0 4 1																				
8 -2 17 0 7 1	-6 3 17 86 92 5	-6 8 17 89 86 6	-5 -7 18 63 68 13	-9 -2 18 0 2 1																				
9 -2 17 66 67 17	-5 3 17 30 23 18	-5 8 17 27 14 27	-4 -7 18 88 86 7	-8 -2 18 72 70 12																				
-10 -1 17 60 48 13	-4 3 17 0 9 1	-4 8 17 123 106 6	-3 -7 18 89 86 7	-7 -2 18 45 0 45																				
-9 -1 17 107 93 18	-3 3 17 145 157 3	-3 8 17 5 6 5	-2 -7 18 80 92 10	-6 -2 18 61 59 6																				
-8 -1 17 43 50 28	-2 3 17 48 44 10	-2 8 17 167 173 4	-1 -7 18 28 39 28	-5 -2 18 75 91 23																				
-7 -1 17 192 194 5	-1 3 17 35 5 9	-1 8 17 81 59 6	0 -7 18 188 190 5	-4 -2 18 97 96 4																				
-6 -1 17 98 100 5	0 3 17 255 265 3	0 8 17 208 190 4	1 -7 18 60 10 12	-3 -2 18 35 29 34																				
-5 -1 17 208 225 4	1 3 17 415 425 4	1 8 17 26 26 25	2 -7 18 31 12 31	-2 -2 18 139 145 3																				
-4 -1 17 238 250 3	2 3 17 237 243 3	2 8 17 54 38 12	3 -7 18 84 84 10	-1 -2 18 0 22 1																				
-3 -1 17 270 270 3	3 3 17 220 216 3	3 8 17 142 150 8	4 -7 18 0 12 1	0 -2 18 455 469 5																				
-2 -1 17 181 183 3	4 3 17 69 71 11	4 8 17 126 120 15	5 -7 18 23 30 23	1 -2 18 197 195 6																				
-1 -1 17 264 258 3	5 3 17 44 31 12	-8 9 17 88 13 43	6 -7 18 24 7 24	2 -2 18 141 144 6																				
0 -1 17 447 448 4	6 3 17 15 25 14	-7 9 17 47 54 18	7 -7 18 111 89 15	3 -2 18 48 35 13																				
1 -1 17 70 73 6	7 3 17 81 68 23	-6 9 17 37 14 20	8 -7 18 97 46 18	4 -2 18 0 30 1																				
2 -1 17 76 47 21	-11 4 17 47 10 37	-5 9 17 73 88 9	-7 -6 18 115 95 9	5 -2 18 65 14 21																				
3 -1 17 222 222 4	-10 4 17 66 50 12	-4 9 17 105 95 5	-6 -6 18 94 95 14	6 -2 18 147 149 6																				
4 -1 17 120 115 4	-9 4 17 134 84 29	-3 9 17 92 78 5	-5 -6 18 29 6 29	7 -2 18 57 47 44																				
5 -1 17 152 169 4	-8 4 17 57 57 16	-2 9 17 44 51 23	-4 -6 18 39 28 26	8 -2 18 69 82 22																				
6 -1 17 15 90 14	-7 4 17 88 90 23	-1 9 17 0 8 1	-3 -6 18 77 65 11	9 -2 18 103 44 33																				
7 -1 17 56 76 41	-6 4 17 150 161 4	0 9 17 65 49 10	-2 -6 18 78 70 16	-10 -1 18 138 129 16																				
8 -1 17 106 52 23	-5 4 17 66 65 8	1 9 17 33 21 33	-1 -6 18 125 142 6	-9 -1 18 184 170 27																				
9 -1 17 149 149 11	-4 4 17 95 102 3	2 9 17 77 98 14	0 -6 18 242 241 6	-8 -1 18 124 108 13																				
-10 0 17 102 96 12	-3 4 17 129 136 3	-7 10 17 92 70 26	1 -6 18 59 60 11	-7 -1 18 149 143 7																				
-9 0 17 51 45 17	-2 4 17 87 81 5	-6 10 17 149 140 10	2 -6 18 38 42 38	-6 -1 18 32 3 31																				
-8 0 17 0 0 1	-1 4 17 0 14 1	-5 10 17 0 0 1	3 -6 18 69 73 14	-5 -1 18 123 122 7																				
-7 0 17 99 87 5	0 4 17 72 79 3	-4 10 17 61 61 11	4 -6 18 25 37 25	-4 -1 18 0 25 1																				
-6 0 17 62 44 7	1 4 17 72 60 7	-3 10 17 83 71 8	5 -6 18 140 140 6	-3 -1 18 305 301 4																				
-5 0 17 227 227 4	2 4 17 73 63 5	-2 10 17 109 95 7	6 -6 18 61 59 8	-2 -1 18 168 172 3																				
-4 0 17 40 31 27	3 4 17 248 245 3	-1 10 17 103 106 9	7 -6 18 145 153 11	-1 -1 18 228 218 3																				
-3 0 17 232 224 2	4 4 17 41 28 10	0 10 17 57 8 23	8 -6 18 156 152 10	0 -1 18 118 119 3																				
-2 0 17 33 34 16	5 4 17 156 163 10	1 10 17 111 112 11	9 -6 18 177 195 9	1 -1 18 127 124 7																				
-1 0 17 139 127 9	6 4 17 10 18 10	-5 11 17 27 5 27	-8 -5 18 57 35 26	2 -1 18 261 262 9																				
0 0 17 395 398 5	7 4 17 72 79 20	-4 11 17 43 63 22	-7 -5 18 58 64 13	3 -1 18 189 197 4																				
1 0 17 66 45 11	-11 5 17 104 82 13	-3 11 17 78 53 15	-6 -5 18 123 130 8	4 -1 18 54 54 9																				
2 0 17 124 122 4	-10 5 17 88 48 26	-2 11 17 0 29 1	-5 -5 18 116 109 6	5 -1 18 43 28 11																				
3 0 17 15 8 14	-9 5 17 55 76 33	-1 11 17 41 34 40	-4 -5 18 111 103 8	6 -1 18 99 107 11																				
4 0 17 40 27 13	-8 5 17 13 26 12	3 -12 18 79 101 22	-3 -5 18 112 122 9	7 -1 18 21 40 20																				
5 0 17 72 67 6	-7 5 17 78 62 9	-2 -11 18 63 12 29	-2 -5 18 38 44 14	8 -1 18 90 65 23																				
6 0 17 0 12 1	-6 5 17 34 27 33	-1 -11 18 16 3 16	-1 -5 18 42 60 11	-10 0 18 0 27 1																				
7 0 17 0 20 1	-5 5 17 79 80 6	0 -11 18 0 45 1	0 -5 18 70 61 70	-9 0 18 87 60 14																				
8 0 17 47 22 46	-4 5 17 125 129 3	1 -11 18 86 28 20	1 -5 18 0 18 1	-8 0 18 62 60 13																				

Table 6. Observed and calculated structure factors for 1

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
1	4	23	138	136	12	-2	-3	24	39	4	39	-5	2	24	52	26	51	2	-4	25	47	11	46	2	2	25	181	174	14
2	4	23	52	48	51	-1	-3	24	0	0	1	-4	2	24	37	13	37	3	-4	25	37	18	37	-3	3	25	116	87	22
3	4	23	147	152	12	0	-3	24	101	97	21	-3	2	24	24	33	24	-3	-3	25	76	88	23	-2	3	25	108	111	15
-5	5	23	102	98	12	1	-3	24	91	93	11	-2	2	24	0	7	1	-2	-3	25	118	125	9	-1	3	25	55	74	30
-4	5	23	138	136	15	2	-3	24	31	29	31	-1	2	24	51	49	30	-1	-3	25	127	124	15	0	3	25	73	45	20
-3	5	23	68	66	14	3	-3	24	117	109	10	0	2	24	44	9	17	0	-3	25	63	21	20	1	3	25	44	27	23
-2	5	23	135	129	9	4	-3	24	96	92	15	1	2	24	16	5	16	1	-3	25	101	105	12	-3	4	25	166	148	11
-1	5	23	27	12	26	-5	-2	24	0	57	1	2	2	24	52	47	16	2	-3	25	37	53	36	-2	4	25	151	140	9
0	5	23	92	94	7	-4	-2	24	22	58	21	3	2	24	94	90	9	3	-3	25	23	30	23	-1	4	25	41	56	40
1	5	23	63	39	13	-3	-2	24	0	7	1	-5	3	24	44	51	21	-4	-2	25	113	134	13	0	4	25	106	110	9
2	5	23	84	75	27	-2	-2	24	59	3	25	-4	3	24	64	61	41	-3	-2	25	120	91	15	-2	5	25	0	47	1
-4	6	23	99	84	24	-1	-2	24	43	46	28	-3	3	24	105	102	12	-2	-2	25	233	226	11	0	-4	26	93	24	15
-3	6	23	118	65	27	0	-2	24	88	82	19	-2	3	24	100	86	11	-1	-2	25	34	22	33	1	-4	26	0	19	1
-2	6	23	140	143	12	1	-2	24	32	16	31	-1	3	24	91	61	21	0	-2	25	128	126	10	-2	-3	26	0	2	1
-1	6	23	77	93	20	2	-2	24	157	158	9	0	3	24	121	122	7	1	-2	25	77	42	28	-1	-3	26	72	7	22
0	6	23	67	27	25	3	-2	24	42	25	42	1	3	24	56	26	46	2	-2	25	57	73	26	0	-3	26	50	33	39
1	6	23	64	65	13	4	-2	24	0	9	1	2	3	24	96	89	12	3	-2	25	49	28	48	2	-3	26	150	144	8
-2	7	23	62	66	26	-5	-1	24	112	75	19	-5	4	24	0	71	1	-4	-1	25	103	116	20	-2	-2	26	0	15	1
-1	7	23	41	29	26	-4	-1	24	44	39	43	-4	4	24	90	68	12	-3	-1	25	32	11	31	-1	-2	26	13	20	12
0	-7	24	140	122	9	-3	-1	24	87	56	13	-3	4	24	101	123	9	-2	-1	25	237	219	9	0	-2	26	61	57	30
1	-7	24	79	90	14	-2	-1	24	65	83	11	-2	4	24	75	64	25	-1	-1	25	82	78	12	1	-2	26	37	20	36
2	-7	24	0	9	1	-1	-1	24	0	24	1	-1	4	24	50	26	16	0	-1	25	65	78	13	2	-2	26	83	84	12
3	-7	24	124	107	34	0	-1	24	82	83	20	0	4	24	58	33	17	1	-1	25	0	24	1	-3	-1	26	58	35	29
-1	-6	24	83	65	37	1	-1	24	110	113	9	1	4	24	54	7	23	2	-1	25	135	133	9	-2	-1	26	69	34	61
0	-6	24	0	6	1	2	-1	24	39	55	38	2	4	24	64	53	19	3	-1	25	170	135	16	-1	-1	26	39	25	39
1	-6	24	72	67	18	3	-1	24	75	61	9	-4	5	24	140	134	19	-4	0	25	39	19	39	0	-1	26	46	12	45
2	-6	24	45	39	45	4	-1	24	192	30	37	-3	5	24	94	44	27	-3	0	25	110	74	22	1	-1	26	165	159	10
3	-6	24	186	188	10	-5	0	24	75	70	10	-2	5	24	89	80	9	-2	0	25	0	27	1	2	-1	26	85	81	12
-3	-5	24	49	59	48	-4	0	24	25	6	25	-1	5	24	132	130	10	-1	0	25	165	157	8	-3	0	26	151	158	30
-2	-5	24	0	16	1	-3	0	24	33	12	32	0	5	24	0	9	1	0	0	25	0	2	1	-2	0	26	113	106	13
-1	-5	24	0	32	1	-2	0	24	41	5	40	-3	6	24	0	9	1	1	0	25	118	118	9	-1	0	26	75	49	14
0	-5	24	207	206	8	-1	0	24	75	48	18	-2	6	24	0	7	1	2	0	25	34	34	33	0	0	26	65	22	22
1	-5	24	157	154	6	0	0	24	98	98	16	-1	6	24	57	54	15	-4	1	25	66	50	24	1	0	26	112	103	12
2	-5	24	53	63	23	1	0	24	51	18	22	0	-6	25	97	96	23	-3	1	25	0	23	1	-3	1	26	131	154	12
3	-5	24	71	27	17	2	0	24	94	91	6	1	-6	25	59	70	24	-2	1	25	166	161	10	-2	1	26	0	25	1
-3	-4	24	53	32	36	3	0	24	147	151	6	2	-6	25	41	64	40	-1	1	25	39	26	22	-1	1	26	110	118	9
-2	-4	24	71	40	71	-5	1	24	46	1	46	-1	-5	25	128	90	14	0	1	25	82	79	8	0	1	26	128	129	10
-1	-4	24	58	28	21	-4	1	24	188	202	11	0	-5	25	95	89	16	1	1	25	86	68	19	1	1	26	80	75	29
0	-4	24	0	20	1	-3	1	24	105	80	13	1	-5	25	148	130	12	2	1	25	117	100	19	-2	2	26	44	12	43
1	-4	24	98	106	14	-2	1	24	30	15	30	2	-5	25	75	35	15	-4	2	25	38	66	38	-1	2	26	61	49	29
2	-4	24	127	124	11	-1	1	24	99	110	6	3	-5	25	81	72	13	-3	2	25	55	45	26	-2	3	26	26	7	26
3	-4	24	78	116	17	0	1	24	70	66	8	-2	-4	25	156	152	9	-2	2	25	194	197	11	-1	3	26	49	63	29
4	-4	24	222	208	9	1	1	24	79	83	9	-1	-4	25	109	113	12	-1	2	25	65	14	26						
-4	-3	24	68	32	33	2	1	24	37	26	36	0	-4	25	63	87	21	0	2	25	152	149	6						
-3	-3	24	0	24	1	4	1	24	90	68	17	1	-4	25	108	84	11	1	2	25	94	71	14						