



ORTEP drawing

The Ortep plot is drawn with 50% probability ellipsoids for the non-hydrogen atoms. The hydrogen atoms are drawn with an artificial radius.

Hart 1731

The data collection crystal was a clear, colorless rectangular plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated an orthorhombic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure an octant of reciprocal space with a redundancy factor of 4.8, which means that 90% of these reflections were measured at least 4.8 times. Phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.048.

The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

For each methyl group, the hydrogen atoms were added at calculated positions using a riding model with $U(H) = 1.5 * U_{eq}(\text{bonded carbon atom})$. The torsion angle, which defines the orientation of the methyl group about the C-C, O-C, or Si-C bond, was refined. The hydrogen atom bonded to O(1) was refined isotropically. The rest of the hydrogen atoms were included in the model at calculated positions using a riding model with $U(H) = 1.2 * U_{eq}(\text{bonded atom})$.

The final refinement cycle was based on 3789 intensities and 191 variables and resulted in agreement factors of $R1(F) = 0.056$ and $wR2(F^2) = 0.099$. For the subset of data with $I > 2 * \sigma(I)$, the $R1(F)$ value is 0.037 for 2894 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.29 and -0.22 e/Å³. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

The O(1)-H bond is involved in an intermolecular hydrogen bonding network with the O(2) atom (see Table 7).

References

- (1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.
- (2) SHELXS-97: Sheldrick, G. M., Acta Cryst., (2008), A64, 112-122.
- (3) SHELXL-97: Sheldrick, G. M., Acta Cryst., (2008), A64, 112-122.
- (4) WinGX-Version 1.70.01: Farrugia, L. J., J. Appl. Cryst., (1999), 32, 837-838.
- (5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 1. Crystallographic details for Hart 1731

Formula	C ₁₆ H ₂₆ O ₃ Si
Formula weight	294.46
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	Pbca
Unit cell dimensions	a = 11.1345(1) Å b = 18.9292(2) Å c = 15.6921(2) Å
Volume	3307.38(6) Å ³
Z	8
Density (calculated)	1.183 Mg/m ³
Absorption coefficient	0.147 mm ⁻¹
F(000)	1280
Crystal size	0.08 x 0.27 x 0.35 mm ³
Theta range for data collection	2.15 to 27.48°
Index ranges	-14 ≤ h ≤ 14, -24 ≤ k ≤ 24, -20 ≤ l ≤ 20
Reflections collected	41862
Independent reflections	3789 [R(int) = 0.048]
Completeness to theta = 27.48°	100.0 %
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3789 / 0 / 191
Goodness-of-fit on F ²	1.044
Final R indices [I > 2σ(I)]	R ₁ = 0.0370, wR ₂ = 0.0919
R indices (all data)	R ₁ = 0.0561, wR ₂ = 0.0993
Largest diff. peak and hole	0.291 and -0.219 e/Å ³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Hart 1731. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
C(1)	10991(1)	3311(1)	-174(1)	19(1)
C(2)	9868(1)	3443(1)	361(1)	21(1)
C(3)	10256(1)	4031(1)	976(1)	21(1)
C(4)	11273(1)	4337(1)	689(1)	18(1)
C(5)	11637(1)	4026(1)	-168(1)	18(1)
C(6)	12898(1)	3780(1)	-399(1)	19(1)
C(7)	12898(1)	3604(1)	-1354(1)	23(1)
C(8)	11786(1)	3193(1)	-1628(1)	25(1)
C(9)	10899(1)	3054(1)	-1081(1)	24(1)
C(10)	11905(1)	2858(1)	321(1)	22(1)
C(11)	11946(1)	4953(1)	1043(1)	27(1)
C(12)	15001(1)	4036(1)	-271(1)	31(1)
C(13)	11779(2)	2968(1)	-2546(1)	36(1)
C(14)	9440(2)	3550(1)	2766(1)	32(1)
C(15)	7679(1)	4196(1)	1521(1)	32(1)
C(16)	9570(1)	5118(1)	2392(1)	27(1)
O(1)	9465(1)	2840(1)	829(1)	27(1)
O(2)	13070(1)	3129(1)	77(1)	21(1)
O(3)	13780(1)	4266(1)	-159(1)	24(1)
Si	9264(1)	4228(1)	1914(1)	21(1)

Table 3. Bond lengths [Å] and angles [°] for Hart 1731.

C(1)-C(9)	1.5080(19)
C(1)-C(2)	1.527(2)
C(1)-C(5)	1.5328(19)
C(1)-C(10)	1.5399(19)
C(2)-O(1)	1.4284(17)
C(2)-C(3)	1.5349(19)
C(2)-H(2)	1.0000
C(3)-C(4)	1.350(2)
C(3)-Si	1.8773(14)
C(4)-C(11)	1.4925(19)
C(4)-C(5)	1.5240(18)
C(5)-C(6)	1.5224(18)
C(5)-H(5)	1.0000
C(6)-O(3)	1.3969(16)
C(6)-O(2)	1.4531(16)
C(6)-C(7)	1.5364(19)
C(7)-C(8)	1.525(2)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(9)	1.334(2)
C(8)-C(13)	1.503(2)
C(9)-H(9)	0.9500
C(10)-O(2)	1.4476(16)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-O(3)	1.4387(17)
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800

C(13)-H(13C)	0.9800
C(14)-Si	1.8642(16)
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-Si	1.8702(15)
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-Si	1.8746(15)
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
O(1)-H(1O1)	0.83(2)
C(9)-C(1)-C(2)	121.07(12)
C(9)-C(1)-C(5)	108.79(11)
C(2)-C(1)-C(5)	103.66(11)
C(9)-C(1)-C(10)	109.95(12)
C(2)-C(1)-C(10)	110.82(11)
C(5)-C(1)-C(10)	100.31(11)
O(1)-C(2)-C(1)	114.07(11)
O(1)-C(2)-C(3)	110.14(11)
C(1)-C(2)-C(3)	103.51(11)
O(1)-C(2)-H(2)	109.6
C(1)-C(2)-H(2)	109.6
C(3)-C(2)-H(2)	109.6
C(4)-C(3)-C(2)	109.78(12)
C(4)-C(3)-Si	132.02(11)
C(2)-C(3)-Si	118.11(10)
C(3)-C(4)-C(11)	129.27(13)
C(3)-C(4)-C(5)	110.55(12)
C(11)-C(4)-C(5)	119.81(12)
C(6)-C(5)-C(4)	124.95(11)
C(6)-C(5)-C(1)	99.31(11)
C(4)-C(5)-C(1)	102.84(11)

C(6)-C(5)-H(5)	109.4
C(4)-C(5)-H(5)	109.4
C(1)-C(5)-H(5)	109.4
O(3)-C(6)-O(2)	109.10(10)
O(3)-C(6)-C(5)	112.54(11)
O(2)-C(6)-C(5)	105.00(10)
O(3)-C(6)-C(7)	113.92(11)
O(2)-C(6)-C(7)	108.47(11)
C(5)-C(6)-C(7)	107.36(11)
C(8)-C(7)-C(6)	112.65(11)
C(8)-C(7)-H(7A)	109.1
C(6)-C(7)-H(7A)	109.1
C(8)-C(7)-H(7B)	109.1
C(6)-C(7)-H(7B)	109.1
H(7A)-C(7)-H(7B)	107.8
C(9)-C(8)-C(13)	123.82(14)
C(9)-C(8)-C(7)	121.42(13)
C(13)-C(8)-C(7)	114.76(13)
C(8)-C(9)-C(1)	119.53(13)
C(8)-C(9)-H(9)	120.2
C(1)-C(9)-H(9)	120.2
O(2)-C(10)-C(1)	105.14(11)
O(2)-C(10)-H(10A)	110.7
C(1)-C(10)-H(10A)	110.7
O(2)-C(10)-H(10B)	110.7
C(1)-C(10)-H(10B)	110.7
H(10A)-C(10)-H(10B)	108.8
C(4)-C(11)-H(11A)	109.5
C(4)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(4)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
O(3)-C(12)-H(12A)	109.5
O(3)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5

O(3)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(8)-C(13)-H(13A)	109.5
C(8)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(8)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
Si-C(14)-H(14A)	109.5
Si-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
Si-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
Si-C(15)-H(15A)	109.5
Si-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
Si-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
Si-C(16)-H(16A)	109.5
Si-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
Si-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(2)-O(1)-H(1O1)	109.5(13)
C(10)-O(2)-C(6)	108.57(10)
C(6)-O(3)-C(12)	115.59(11)
C(14)-Si-C(15)	108.25(8)
C(14)-Si-C(16)	108.17(7)
C(15)-Si-C(16)	109.45(7)
C(14)-Si-C(3)	111.28(7)
C(15)-Si-C(3)	106.93(7)
C(16)-Si-C(3)	112.66(7)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Hart 1731. 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}
C(1)	22(1)	16(1)	20(1)	-1(1)	-1(1)	-2(1)
C(2)	21(1)	18(1)	24(1)	-1(1)	0(1)	-3(1)
C(3)	21(1)	21(1)	22(1)	-2(1)	-1(1)	-1(1)
C(4)	20(1)	17(1)	18(1)	-2(1)	-2(1)	2(1)
C(5)	20(1)	17(1)	18(1)	0(1)	-2(1)	-1(1)
C(6)	21(1)	16(1)	21(1)	0(1)	1(1)	-1(1)
C(7)	27(1)	23(1)	20(1)	1(1)	3(1)	2(1)
C(8)	32(1)	21(1)	22(1)	-4(1)	-3(1)	5(1)
C(9)	29(1)	20(1)	23(1)	-5(1)	-5(1)	-1(1)
C(10)	23(1)	18(1)	25(1)	2(1)	2(1)	-1(1)
C(11)	25(1)	29(1)	26(1)	-8(1)	4(1)	-7(1)
C(12)	21(1)	33(1)	38(1)	-4(1)	5(1)	-1(1)
C(13)	44(1)	39(1)	24(1)	-10(1)	-1(1)	2(1)
C(14)	37(1)	30(1)	30(1)	4(1)	1(1)	-3(1)
C(15)	23(1)	33(1)	39(1)	-4(1)	0(1)	0(1)
C(16)	27(1)	27(1)	26(1)	-4(1)	5(1)	2(1)
O(1)	30(1)	23(1)	28(1)	-2(1)	1(1)	-11(1)
O(2)	20(1)	19(1)	23(1)	3(1)	0(1)	2(1)
O(3)	19(1)	22(1)	31(1)	-4(1)	2(1)	-2(1)
Si	19(1)	21(1)	22(1)	0(1)	2(1)	0(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Hart 1731.

	x	y	z	U(eq)
H(2)	9205	3619	-13	25
H(5)	11313	4328	-637	22
H(7A)	12941	4049	-1685	28
H(7B)	13623	3323	-1490	28
H(9)	10214	2795	-1262	29
H(10A)	11780	2905	943	26
H(10B)	11828	2353	163	26
H(11A)	11612	5081	1600	40
H(11B)	11874	5355	654	40
H(11C)	12795	4827	1109	40
H(12A)	15134	3601	55	46
H(12B)	15549	4404	-67	46
H(12C)	15152	3946	-876	46
H(13A)	11008	2745	-2681	54
H(13B)	12431	2630	-2646	54
H(13C)	11893	3383	-2911	54
H(14A)	9235	3084	2538	48
H(14B)	8903	3663	3242	48
H(14C)	10273	3548	2967	48
H(15A)	7564	4556	1079	48
H(15B)	7129	4288	1996	48
H(15C)	7513	3728	1282	48
H(16A)	10303	5095	2736	40
H(16B)	8893	5257	2753	40
H(16C)	9675	5466	1935	40
H(10I)	9070(17)	2576(10)	508(12)	45(6)*

*Refined isotropically

Table 6. Torsion angles [°] for Hart 1731.

C(9)-C(1)-C(2)-O(1)	89.65(16)
C(5)-C(1)-C(2)-O(1)	-148.11(11)
C(10)-C(1)-C(2)-O(1)	-41.28(15)
C(9)-C(1)-C(2)-C(3)	-150.67(13)
C(5)-C(1)-C(2)-C(3)	-28.43(13)
C(10)-C(1)-C(2)-C(3)	78.39(13)
O(1)-C(2)-C(3)-C(4)	138.49(12)
C(1)-C(2)-C(3)-C(4)	16.16(15)
O(1)-C(2)-C(3)-Si	-44.62(15)
C(1)-C(2)-C(3)-Si	-166.95(9)
C(2)-C(3)-C(4)-C(11)	176.30(14)
Si-C(3)-C(4)-C(11)	0.0(2)
C(2)-C(3)-C(4)-C(5)	3.41(16)
Si-C(3)-C(4)-C(5)	-172.90(11)
C(3)-C(4)-C(5)-C(6)	-132.68(14)
C(11)-C(4)-C(5)-C(6)	53.66(19)
C(3)-C(4)-C(5)-C(1)	-21.48(15)
C(11)-C(4)-C(5)-C(1)	164.86(12)
C(9)-C(1)-C(5)-C(6)	-70.50(13)
C(2)-C(1)-C(5)-C(6)	159.44(11)
C(10)-C(1)-C(5)-C(6)	44.85(12)
C(9)-C(1)-C(5)-C(4)	160.25(11)
C(2)-C(1)-C(5)-C(4)	30.18(13)
C(10)-C(1)-C(5)-C(4)	-84.40(12)
C(4)-C(5)-C(6)-O(3)	-46.05(18)
C(1)-C(5)-C(6)-O(3)	-158.96(11)
C(4)-C(5)-C(6)-O(2)	72.51(15)
C(1)-C(5)-C(6)-O(2)	-40.40(12)
C(4)-C(5)-C(6)-C(7)	-172.19(12)
C(1)-C(5)-C(6)-C(7)	74.90(13)
O(3)-C(6)-C(7)-C(8)	-168.58(11)
O(2)-C(6)-C(7)-C(8)	69.71(14)
C(5)-C(6)-C(7)-C(8)	-43.27(16)
C(6)-C(7)-C(8)-C(9)	3.9(2)

C(6)-C(7)-C(8)-C(13)	-176.38(13)
C(13)-C(8)-C(9)-C(1)	-179.37(14)
C(7)-C(8)-C(9)-C(1)	0.3(2)
C(2)-C(1)-C(9)-C(8)	154.55(13)
C(5)-C(1)-C(9)-C(8)	34.80(18)
C(10)-C(1)-C(9)-C(8)	-74.14(17)
C(9)-C(1)-C(10)-O(2)	79.63(14)
C(2)-C(1)-C(10)-O(2)	-143.87(11)
C(5)-C(1)-C(10)-O(2)	-34.84(13)
C(1)-C(10)-O(2)-C(6)	10.00(14)
O(3)-C(6)-O(2)-C(10)	140.24(11)
C(5)-C(6)-O(2)-C(10)	19.39(13)
C(7)-C(6)-O(2)-C(10)	-95.14(12)
O(2)-C(6)-O(3)-C(12)	57.29(15)
C(5)-C(6)-O(3)-C(12)	173.41(12)
C(7)-C(6)-O(3)-C(12)	-64.07(15)
C(4)-C(3)-Si-C(14)	-107.20(15)
C(2)-C(3)-Si-C(14)	76.74(12)
C(4)-C(3)-Si-C(15)	134.78(15)
C(2)-C(3)-Si-C(15)	-41.28(13)
C(4)-C(3)-Si-C(16)	14.49(17)
C(2)-C(3)-Si-C(16)	-161.58(11)

Table 7. Hydrogen bonds for Hart 1731 [\AA and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
O(1)-H(1O1)...O(2)#1	0.83(2)	1.97(2)	2.7926(14)	170.7(19)

Symmetry transformations used to generate equivalent atoms:

#1 $x-1/2, -y+1/2, -z$