Crystal structure of tris(tri-*tert*-butoxysilanethiolate)arsenic(III), $\text{As}\{\text{SSi}(\text{OC}_4\text{H}_9)_3\}_3$

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Source of material: The title compound was prepared from (*tert*-C₄H₉O)₃SiSH (preparation see ref. 1) and AsCl₃.

The positions of all terminal C atoms were refined isotropically, due to their high thermal motion. The positions of the H atoms were calculated by riding model and considered with $U_{\text{iso}} = 1.2 \times U_{\text{iso}}$ of the corresponding C atom.

C₃₆H₈₁AsO₉S₃Si₃, orthorhombic, $P2_12_12_1$ (No. 19), $a = 15.364(6)$ Å, $b = 24.46(1)$ Å, $c = 14.591(6)$ Å, $V = 5483.3$ Å³, $Z = 4$, $R(F) = 0.112$, $R_w(F) = 0.053$.

Table 1. Parameters used for the X-ray data collection

Crystal:	pale brown prism, size 0.75 x 1.35 x 0.4 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	8.40 cm ⁻¹
Diffractometer:	Siemens P4
Scan mode:	ω
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{unique}}$:	10745
Criterion for F_o :	$F_o > 3 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	334
Program:	SHELXTL-plus

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
C(11)	4a	0.867(1)	-0.0951(8)	0.608(2)	0.26(1)
H(11A)	4a	0.849(1)	-0.1069(8)	0.548(2)	0.32
H(11B)	4a	0.838(1)	-0.1163(8)	0.654(2)	0.32
H(11C)	4a	0.929(1)	-0.1001(8)	0.614(2)	0.32
C(12)	4a	0.769(1)	-0.025(1)	0.611(2)	0.27(2)
H(12A)	4a	0.753(1)	-0.026(1)	0.548(2)	0.38
H(12B)	4a	0.757(1)	0.011(1)	0.635(2)	0.38
H(12C)	4a	0.735(1)	-0.051(1)	0.645(2)	0.38
C(13)	4a	0.876(2)	-0.036(1)	0.706(2)	0.31(2)
H(13A)	4a	0.870(2)	-0.001(1)	0.732(2)	0.42
H(13B)	4a	0.937(2)	-0.046(1)	0.703(2)	0.42
H(13C)	4a	0.845(2)	-0.062(1)	0.743(2)	0.42
C(21)	4a	1.078(1)	-0.0190(9)	0.301(1)	0.20(1)
H(21A)	4a	1.071(1)	-0.0135(9)	0.236(1)	0.26
H(21B)	4a	1.124(1)	-0.0453(9)	0.311(1)	0.26
H(21C)	4a	1.094(1)	0.0150(9)	0.329(1)	0.26
C(22)	4a	1.002(1)	-0.1030(6)	0.321(1)	0.190(8)
H(22A)	4a	0.986(1)	-0.1087(6)	0.258(1)	0.26
H(22B)	4a	0.961(1)	-0.1219(6)	0.360(1)	0.26
H(22C)	4a	1.059(1)	-0.1167(6)	0.331(1)	0.26
C(23)	4a	0.920(2)	-0.019(1)	0.317(2)	0.25(2)
H(23A)	4a	0.921(2)	0.019(1)	0.331(2)	0.29
H(23B)	4a	0.875(2)	-0.037(1)	0.351(2)	0.29
H(23C)	4a	0.909(2)	-0.024(1)	0.252(2)	0.29
C(31)	4a	1.164(1)	-0.1043(8)	0.574(2)	0.24(1)
H(31A)	4a	1.129(1)	-0.1286(8)	0.610(2)	0.32
H(31B)	4a	1.222(1)	-0.1193(8)	0.568(2)	0.32
H(31C)	4a	1.138(1)	-0.1000(8)	0.515(2)	0.32
C(32)	4a	1.174(2)	-0.0616(9)	0.710(2)	0.28(1)
H(32A)	4a	1.177(2)	-0.0314(9)	0.752(2)	0.4
H(32B)	4a	1.222(2)	-0.0854(9)	0.720(2)	0.4
H(32C)	4a	1.120(2)	-0.0814(9)	0.720(2)	0.4
C(33)	4a	1.215(2)	-0.032(1)	0.560(2)	0.36(2)
H(33A)	4a	1.230(2)	0.006(1)	0.573(2)	0.61
H(33B)	4a	1.184(2)	-0.034(1)	0.503(2)	0.61
H(33C)	4a	1.267(2)	-0.053(1)	0.556(2)	0.61
C(41)	4a	1.102(1)	0.0288(8)	0.922(2)	0.21(1)
H(41A)	4a	1.100(1)	0.0110(8)	0.863(2)	0.25
H(41B)	4a	1.125(1)	0.0040(8)	0.967(2)	0.25
H(41C)	4a	1.138(1)	0.0605(8)	0.918(2)	0.25
C(42)	4a	1.018(1)	0.0725(6)	1.042(1)	0.181(8)
H(42A)	4a	1.044(1)	0.0471(6)	1.084(1)	0.22
H(42B)	4a	0.963(1)	0.0837(6)	1.066(1)	0.22
H(42C)	4a	1.055(1)	0.1039(6)	1.035(1)	0.22
C(43)	4a	0.952(1)	0.0007(6)	0.959(1)	0.192(9)
H(43A)	4a	0.978(1)	-0.0250(6)	1.000(1)	0.24
H(43B)	4a	0.945(1)	-0.0162(6)	0.900(1)	0.24
H(43C)	4a	0.896(1)	0.0118(6)	0.982(1)	0.24

Table 2. (Continued)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
C(51)	4a	1.075(1)	0.2520(6)	0.821(1)	0.176(9)
H(51A)	4a	1.105(1)	0.2858(6)	0.812(1)	0.26
H(51B)	4a	1.017(1)	0.2594(6)	0.843(1)	0.26
H(51C)	4a	1.072(1)	0.2323(6)	0.764(1)	0.26
C(52)	4a	1.110(1)	0.2433(7)	0.978(1)	0.161(8)
H(52A)	4a	1.145(1)	0.2756(7)	0.980(1)	0.19
H(52B)	4a	1.127(1)	0.2188(7)	1.026(1)	0.19
H(52C)	4a	1.050(1)	0.2533(7)	0.985(1)	0.19
C(53)	4a	1.206(1)	0.1971(8)	0.867(2)	0.24(1)
H(53A)	4a	1.247(1)	0.2269(8)	0.864(2)	0.28
H(53B)	4a	1.203(1)	0.1793(8)	0.809(2)	0.28
H(53C)	4a	1.225(1)	0.1714(8)	0.913(2)	0.28
C(61)	4a	0.841(1)	0.1903(8)	0.931(1)	0.19(1)
H(61A)	4a	0.879(1)	0.2212(8)	0.940(1)	0.25
H(61B)	4a	0.865(1)	0.1591(8)	0.962(1)	0.25
H(61C)	4a	0.784(1)	0.1983(8)	0.955(1)	0.25
C(62)	4a	0.777(1)	0.1286(9)	0.823(1)	0.25(1)
H(62A)	4a	0.769(1)	0.1182(9)	0.760(1)	0.34
H(62B)	4a	0.721(1)	0.1379(9)	0.849(1)	0.34
H(62C)	4a	0.802(1)	0.0987(9)	0.857(1)	0.34
C(63)	4a	0.804(1)	0.2301(7)	0.783(1)	0.21(1)
H(63A)	4a	0.798(1)	0.2250(7)	0.719(1)	0.27
H(63B)	4a	0.846(1)	0.2582(7)	0.795(1)	0.27
H(63C)	4a	0.749(1)	0.2405(7)	0.809(1)	0.27
C(71)	4a	0.774(1)	0.3172(9)	0.545(2)	0.27(2)
H(71A)	4a	0.786(1)	0.2902(9)	0.591(2)	0.31
H(71B)	4a	0.726(1)	0.3053(9)	0.508(2)	0.31
H(71C)	4a	0.759(1)	0.3513(9)	0.574(2)	0.31
C(72)	4a	0.917(1)	0.3412(7)	0.541(1)	0.20(1)
H(72A)	4a	0.931(1)	0.3155(7)	0.588(1)	0.25

Table 2. (Continued)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(72B)	4a	0.904(1)	0.3761(7)	0.568(1)	0.25
H(72C)	4a	0.965(1)	0.3448(7)	0.500(1)	0.25
C(73)	4a	0.832(1)	0.3609(8)	0.430(1)	0.25(1)
H(73A)	4a	0.782(1)	0.3500(8)	0.395(1)	0.32
H(73B)	4a	0.882(1)	0.3642(8)	0.391(1)	0.32
H(73C)	4a	0.821(1)	0.3955(8)	0.459(1)	0.32
C(81)	4a	0.690(1)	0.1419(6)	0.533(1)	0.166(9)
H(81A)	4a	0.637(1)	0.1243(6)	0.512(1)	0.21
H(81B)	4a	0.675(1)	0.1763(6)	0.561(1)	0.21
H(81C)	4a	0.718(1)	0.1190(6)	0.577(1)	0.21
C(82)	4a	0.7080(9)	0.1848(6)	0.381(1)	0.137(6)
H(82A)	4a	0.6591(9)	0.1649(6)	0.357(1)	0.16
H(82B)	4a	0.7486(9)	0.1919(6)	0.332(1)	0.16
H(82C)	4a	0.6884(9)	0.2188(6)	0.406(1)	0.16
C(83)	4a	0.7755(9)	0.0972(5)	0.421(1)	0.136(6)
H(83A)	4a	0.7252(9)	0.0802(5)	0.394(1)	0.16
H(83B)	4a	0.7963(9)	0.0751(5)	0.470(1)	0.16
H(83C)	4a	0.8203(9)	0.1005(5)	0.375(1)	0.16
C(91)	4a	1.030(2)	0.1554(7)	0.257(1)	0.24(1)
H(91A)	4a	0.990(2)	0.1272(7)	0.239(1)	0.33
H(91B)	4a	1.065(2)	0.1426(7)	0.307(1)	0.33
H(91C)	4a	1.066(2)	0.1646(7)	0.206(1)	0.33
C(92)	4a	0.933(1)	0.2294(7)	0.212(1)	0.20(1)
H(92A)	4a	0.885(1)	0.2065(7)	0.195(1)	0.26
H(92B)	4a	0.971(1)	0.2348(7)	0.161(1)	0.26
H(92C)	4a	0.911(1)	0.2640(7)	0.233(1)	0.26
C(93)	4a	1.051(1)	0.2418(8)	0.309(1)	0.25(1)
H(93A)	4a	1.027(1)	0.2760(8)	0.329(1)	0.32
H(93B)	4a	1.086(1)	0.2478(8)	0.256(1)	0.32
H(93C)	4a	1.085(1)	0.2258(8)	0.357(1)	0.32

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
As	4a	0.96965(9)	0.11731(5)	0.60568(9)	0.0825(9)	0.0567(7)	0.0642(8)	−0.0063(8)	0.0009(9)	0.0044(8)
S(1)	4a	1.0466(3)	0.0716(1)	0.4983(2)	0.127(3)	0.066(2)	0.073(2)	−0.010(2)	0.017(3)	0.008(2)
S(2)	4a	1.0739(2)	0.1127(2)	0.7136(2)	0.088(3)	0.097(3)	0.077(2)	0.010(3)	−0.004(2)	0.006(3)
S(3)	4a	1.0012(3)	0.2021(1)	0.5557(2)	0.101(3)	0.057(2)	0.097(3)	−0.018(2)	−0.015(3)	0.007(2)
Si(1)	4a	1.0124(4)	−0.0104(2)	0.5290(3)	0.119(4)	0.064(3)	0.090(3)	0.003(3)	0.021(3)	−0.003(2)
Si(2)	4a	1.0017(3)	0.1341(1)	0.8331(3)	0.091(3)	0.071(2)	0.062(2)	−0.001(2)	−0.000(2)	−0.001(2)
Si(3)	4a	0.9012(3)	0.2140(2)	0.4591(3)	0.085(3)	0.062(2)	0.073(3)	0.000(2)	−0.005(2)	0.003(2)
O(1)	4a	0.9155(7)	−0.0079(3)	0.5670(7)	0.131(9)	0.057(6)	0.119(9)	0.020(6)	0.019(7)	−0.002(6)
O(2)	4a	1.0261(8)	−0.0473(3)	0.4393(7)	0.19(1)	0.063(6)	0.121(8)	−0.017(8)	0.02(1)	−0.019(6)
O(3)	4a	1.0694(7)	−0.0385(3)	0.6066(7)	0.15(1)	0.070(6)	0.097(8)	0.005(6)	0.024(8)	−0.003(6)
O(4)	4a	0.9679(6)	0.0822(3)	0.8910(6)	0.111(7)	0.084(6)	0.089(6)	−0.019(6)	0.008(8)	0.018(6)
O(5)	4a	1.0631(6)	0.1690(4)	0.8968(7)	0.112(8)	0.092(7)	0.097(7)	−0.037(6)	−0.020(7)	−0.002(7)
O(6)	4a	0.9186(6)	0.1627(4)	0.7936(7)	0.080(7)	0.115(8)	0.092(7)	0.037(6)	0.014(6)	−0.002(6)
O(7)	4a	0.8783(8)	0.2743(4)	0.4519(8)	0.19(1)	0.093(8)	0.19(1)	0.029(8)	−0.08(1)	0.001(8)
O(8)	4a	0.8233(6)	0.1766(4)	0.4945(8)	0.089(8)	0.105(8)	0.19(1)	−0.037(7)	−0.056(8)	0.033(8)
O(9)	4a	0.9240(8)	0.1904(5)	0.3609(7)	0.15(1)	0.20(1)	0.083(8)	0.01(1)	−0.006(8)	0.003(9)
C(1)	4a	0.840(2)	−0.035(2)	0.621(3)	0.17(3)	0.45(5)	0.48(5)	−0.08(3)	−0.02(3)	0.42(4)
C(2)	4a	0.999(3)	−0.042(1)	0.340(1)	0.39(5)	0.19(2)	0.11(2)	−0.13(3)	−0.00(3)	−0.11(2)
C(3)	4a	1.174(2)	−0.044(2)	0.623(2)	0.43(4)	0.62(5)	0.10(2)	0.50(4)	−0.00(2)	−0.05(3)
C(4)	4a	1.008(2)	0.0450(6)	0.950(1)	0.26(3)	0.09(1)	0.11(1)	−0.09(2)	−0.10(2)	0.06(1)
C(5)	4a	1.121(1)	0.2185(6)	0.893(1)	0.21(2)	0.09(1)	0.060(9)	−0.02(1)	−0.02(1)	−0.00(1)
C(6)	4a	0.835(2)	0.178(1)	0.829(1)	0.18(2)	0.17(2)	0.12(2)	0.07(2)	−0.01(2)	0.02(2)
C(7)	4a	0.846(2)	0.3248(7)	0.495(2)	0.26(3)	0.08(1)	0.14(2)	0.06(2)	−0.05(2)	−0.03(1)
C(8)	4a	0.751(1)	0.1507(5)	0.452(1)	0.09(1)	0.062(9)	0.13(1)	0.001(8)	−0.03(1)	−0.01(1)
C(9)	4a	0.978(1)	0.2038(9)	0.286(1)	0.14(2)	0.23(2)	0.07(1)	0.04(2)	0.02(1)	0.02(1)

References

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