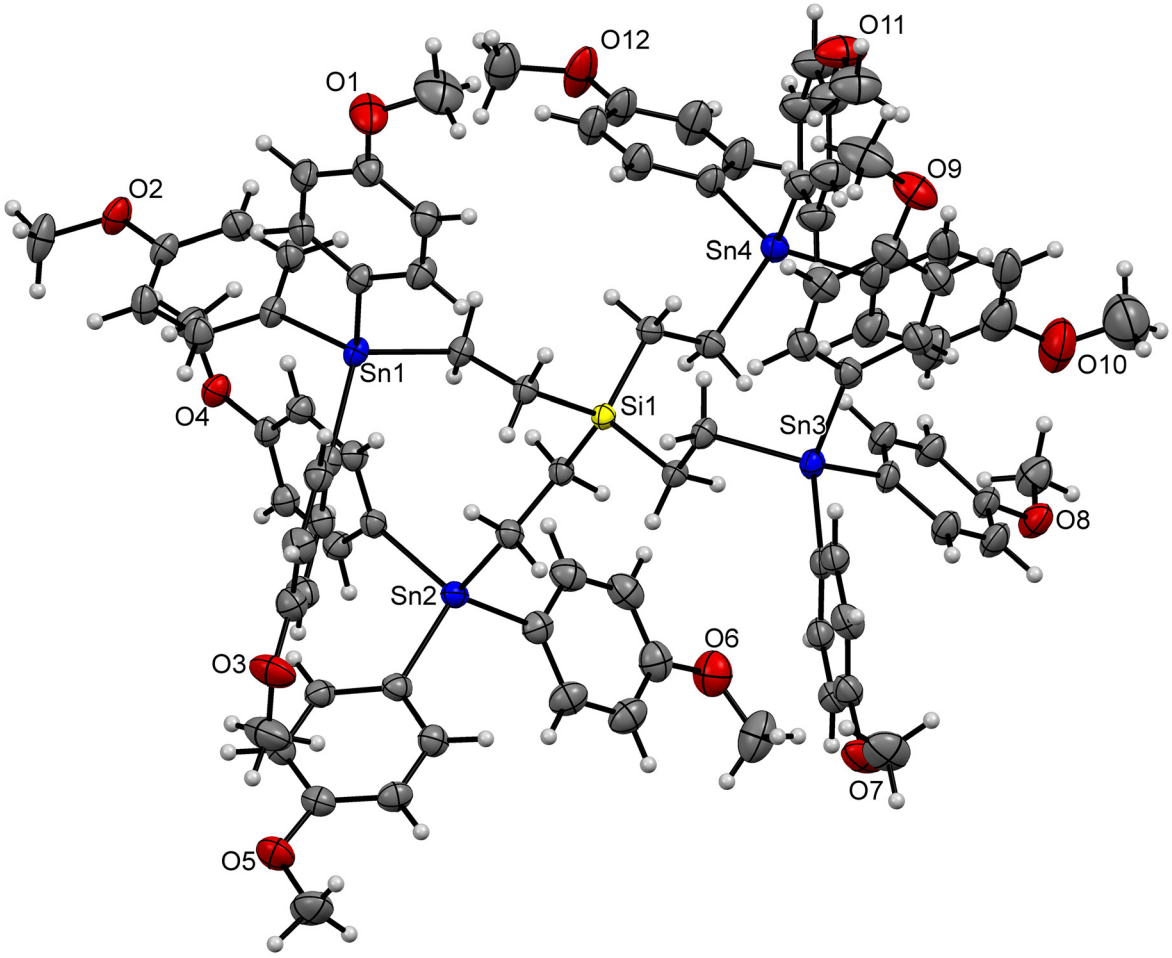
Yilmaz Aksu*, Elisabeth Irran and Sevil Aksu

The crystal structure of the co-crystal tetrakis [2-(tris(4-methoxyphenyl)stannyl)ethyl]silane – tetrahydrofuran – toluene – tetrahydrofurane (1/1/1), $C_{103}H_{116}O_{13}SiSn_4$



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Abstract

$C_{103}H_{116}O_{13}SiSn_4$, triclinic, $P\bar{1}$ (no. 2), a = 14.8676(4) Å, b = 17.6664(4) Å, c = 20.8357(6) Å, α = 106.430(2)°, β = 107.192(2)°, γ = 100.898(2)°, V = 4787.5(2) Å^3, Z = 2, $R_{gt}(F)$ = 0.0325, w$R_{ref}(F^2)$ = 0.0845, T = 150 K.

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The molecular structure is shown in the figure (The DMF molecule as well as the toluene molecule are both not shown for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.44 × 0.37 × 0.32 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.10 mm^{-1}
Diffractometer, scan mode:	Xcalibur, *ω*
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	37,597, 16,825, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 14,534
$N(param)_{refined}$:	1103
Programs:	WinGX/ORTEP [1], SHELX [2], Mercury [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.463086 (17)	0.665111 (14)	0.439683 (12)	0.02608 (6)
Sn2	0.875448 (17)	0.919248 (14)	0.574697 (13)	0.02726 (6)
Sn3	0.787364 (18)	0.629606 (14)	0.839769 (12)	0.02771 (7)
Sn4	0.614274 (18)	0.943457 (15)	0.889013 (13)	0.02999 (7)
Si1	0.68738 (7)	0.78356 (5)	0.68810 (5)	0.02201 (18)
O1	0.0969 (2)	0.38438 (19)	0.39913 (16)	0.0578 (8)
O2	0.2628 (2)	0.87890 (16)	0.27855 (14)	0.0417 (6)
O3	0.7583 (2)	0.52011 (18)	0.31405 (17)	0.0559 (8)
O4	0.57430 (18)	1.06794 (14)	0.40286 (13)	0.0332 (6)
O5	1.0416 (2)	0.72702 (17)	0.35449 (15)	0.0479 (7)
O6	1.1731 (3)	1.19859 (18)	0.86854 (17)	0.0578 (8)
O7	1.0929 (2)	0.42722 (18)	0.82442 (17)	0.0522 (8)
O8	1.0558 (2)	0.94760 (16)	1.11227 (14)	0.0440 (7)
O9	0.4448 (2)	0.42605 (18)	0.90062 (17)	0.0558 (8)
O10	0.9324 (3)	1.1834 (2)	1.19529 (19)	0.0817 (11)
O11	0.3144 (2)	0.63865 (19)	0.8888 (2)	0.0620 (9)
O12	0.3944 (3)	1.17991 (19)	0.77599 (16)	0.0638 (10)
C1	0.5953 (2)	0.70304 (19)	0.60042 (17)	0.0255 (7)
H1A	0.6308	0.6725	0.5741	0.031*
H1B	0.5507	0.6629	0.6107	0.031*
C2	0.5332 (3)	0.7413 (2)	0.55211 (17)	0.0271 (7)
H2A	0.5765	0.7941	0.5567	0.032*
H2B	0.4816	0.7543	0.5705	0.032*
C3	0.3451 (3)	0.5630 (2)	0.42338 (18)	0.0278 (7)
C4	0.2539 (3)	0.5422 (2)	0.36838 (18)	0.0314 (7)
H4	0.2474	0.5705	0.3354	0.038*
C5	0.1812 (3)	0.4411 (2)	0.4085 (2)	0.0362 (8)
C6	0.1725 (3)	0.4819 (2)	0.36014 (19)	0.0365 (8)
H6	0.1114	0.4686	0.3218	0.044*
C7	0.2711 (3)	0.4584 (2)	0.4627 (2)	0.0377 (8)
H7	0.2775	0.4293	0.4950	0.045*
C8	0.3518 (3)	0.5187 (2)	0.4695 (2)	0.0354 (8)
H8	0.4134	0.5301	0.5066	0.042*
C9	0.0951 (4)	0.3620 (3)	0.4587 (3)	0.0704 (15)
H9A	0.0278	0.3308	0.4493	0.106*
H9B	0.1174	0.4120	0.5017	0.106*
H9C	0.1390	0.3278	0.4664	0.106*
C10	0.4009 (3)	0.7374 (2)	0.38254 (18)	0.0281 (7)
C11	0.3622 (3)	0.7971 (2)	0.41531 (19)	0.0329 (7)

Table 2: (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H11	0.3675	0.8068	0.4637	0.040*
C12	0.3163 (3)	0.8424 (2)	0.3791 (2)	0.0356 (8)
H12	0.2903	0.8822	0.4025	0.043*
C13	0.3083 (3)	0.8296 (2)	0.30869 (19)	0.0329 (7)
C14	0.3464 (3)	0.7724 (2)	0.2750 (2)	0.0397 (8)
H14	0.3415	0.7637	0.2268	0.048*
C15	0.3927 (3)	0.7272 (2)	0.31214 (19)	0.0380 (8)
H15	0.4193	0.6881	0.2885	0.046*
C16	0.2522 (4)	0.8686 (3)	0.2064 (2)	0.0531 (12)
H16A	0.2157	0.9049	0.1906	0.080*
H16B	0.2160	0.8110	0.1752	0.080*
H16C	0.3175	0.8828	0.2035	0.080*
C17	0.5665 (3)	0.6196 (2)	0.39863 (18)	0.0288 (7)
C18	0.5512 (3)	0.5339 (2)	0.36903 (19)	0.0335 (7)
H18	0.4955	0.4965	0.3683	0.040*
C19	0.6161 (3)	0.5034 (2)	0.3410 (2)	0.0387 (8)
H19	0.6036	0.4454	0.3201	0.046*
C20	0.6988 (3)	0.5566 (2)	0.3431 (2)	0.0379 (8)
C21	0.7168 (3)	0.6417 (2)	0.3732 (2)	0.0370 (8)
H21	0.7738	0.6789	0.3753	0.044*
C22	0.6499 (3)	0.6715 (2)	0.40012 (19)	0.0324 (7)
H22	0.6620	0.7296	0.4202	0.039*
C23	0.8477 (4)	0.5737 (3)	0.3189 (3)	0.0654 (14)
H23A	0.8824	0.5406	0.2949	0.098*
H23B	0.8900	0.6040	0.3697	0.098*
H23C	0.8318	0.6129	0.2955	0.098*
C24	0.7599 (2)	0.8654 (2)	0.66564 (17)	0.0255 (7)
H24A	0.7171	0.8980	0.6489	0.031*
H24B	0.8160	0.9035	0.7099	0.031*
C25	0.7998 (3)	0.8291 (2)	0.60722 (18)	0.0285 (7)
H25A	0.7439	0.7882	0.5643	0.034*
H25B	0.8456	0.7994	0.6254	0.034*
C26	0.7703 (3)	0.9666 (2)	0.51552 (18)	0.0273 (7)
C27	0.8018 (3)	1.0351 (2)	0.49733 (18)	0.0301 (7)
H27	0.8704	1.0602	0.5112	0.036*
C28	0.7349 (3)	1.0668 (2)	0.45970 (18)	0.0305 (7)
H28	0.7579	1.1133	0.4482	0.037*
C29	0.6342 (3)	1.0308 (2)	0.43872 (18)	0.0276 (7)
C30	0.6005 (3)	0.9617 (2)	0.45428 (19)	0.0308 (7)
H30	0.5319	0.9357	0.4391	0.037*
C31	0.6695 (3)	0.9313 (2)	0.49261 (19)	0.0308 (7)
H31	0.6464	0.8844	0.5035	0.037*
C32	0.4702 (3)	1.0277 (3)	0.3749 (2)	0.0429 (10)
H32A	0.4351	1.0589	0.3498	0.064*
H32B	0.4489	1.0253	0.4147	0.064*
H32C	0.4556	0.9715	0.3412	0.064*
C33	0.9409 (3)	0.8601 (2)	0.50396 (19)	0.0303 (7)
C34	0.9134 (3)	0.8576 (2)	0.43314 (19)	0.0311 (7)
H34	0.8697	0.8873	0.4179	0.037*
C35	0.9477 (3)	0.8133 (2)	0.3845 (2)	0.0339 (7)
H35	0.9275	0.8129	0.3367	0.041*
C36	1.0120 (3)	0.7693 (2)	0.4059 (2)	0.0344 (7)
C37	1.0414 (3)	0.7712 (2)	0.4759 (2)	0.0354 (8)
H37	1.0854	0.7415	0.4910	0.043*
C38	1.0064 (3)	0.8163 (2)	0.5239 (2)	0.0336 (7)
H38	1.0278	0.8176	0.5720	0.040*

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C39	1.1080 (3)	0.6815 (3)	0.3741 (3)	0.0592 (13)
H39A	1.1262	0.6568	0.3332	0.089*
H39B	1.0760	0.6377	0.3874	0.089*
H39C	1.1674	0.7188	0.4152	0.089*
C40	0.9820 (3)	1.0153 (2)	0.6703 (2)	0.0338 (7)
C41	0.9515 (3)	1.0730 (2)	0.7138 (2)	0.0409 (8)
H41	0.8839	1.0709	0.6980	0.049*
C42	1.0158 (3)	1.1326 (2)	0.7787 (2)	0.0442 (8)
H42	0.9926	1.1712	0.8064	0.053*
C43	1.1136 (3)	1.1361 (2)	0.8031 (2)	0.0441 (8)
C44	1.1482 (3)	1.0816 (2)	0.7626 (2)	0.0460 (9)
H44	1.2160	1.0845	0.7794	0.055*
C45	1.0821 (3)	1.0213 (2)	0.6957 (2)	0.0436 (9)
H45	1.1062	0.9841	0.6674	0.052*
C46	1.2656 (4)	1.1923 (3)	0.9043 (3)	0.0643 (14)
H46A	1.2972	1.2370	0.9515	0.096*
H46B	1.3068	1.1966	0.8757	0.096*
H46C	1.2575	1.1388	0.9106	0.096*
C47	0.7715 (2)	0.7348 (2)	0.73876 (18)	0.0261 (7)
H47A	0.8125	0.7157	0.7116	0.031*
H47B	0.8162	0.7768	0.7862	0.031*
C48	0.7130 (3)	0.6613 (2)	0.74994 (18)	0.0294 (7)
H48A	0.6915	0.6121	0.7055	0.035*
H48B	0.6528	0.6731	0.7556	0.035*
C49	0.8871 (3)	0.5588 (2)	0.82693 (18)	0.0297 (7)
C50	0.9819 (3)	0.5933 (2)	0.82992 (19)	0.0345 (7)
H50	1.0013	0.6493	0.8336	0.041*
C51	1.0479 (3)	0.5479 (2)	0.82758 (19)	0.0365 (8)
H51	1.1116	0.5725	0.8293	0.044*
C52	1.0205 (3)	0.4662 (2)	0.8227 (2)	0.0369 (8)
C53	0.9267 (3)	0.4298 (2)	0.8177 (2)	0.0379 (8)
H53	0.9071	0.3734	0.8127	0.045*
C54	0.8614 (3)	0.4761 (2)	0.82011 (19)	0.0345 (7)
H54	0.7971	0.4507	0.8170	0.041*
C55	1.0708 (4)	0.3458 (3)	0.8267 (3)	0.0616 (13)
H55A	1.1279	0.3251	0.8285	0.092*
H55B	1.0138	0.3090	0.7835	0.092*
H55C	1.0556	0.3475	0.8697	0.092*
C56	0.8714 (3)	0.7388 (2)	0.93269 (18)	0.0309 (7)
C57	0.9579 (3)	0.7387 (2)	0.9827 (2)	0.0392 (8)
H57	0.9770	0.6895	0.9756	0.047*
C58	1.0162 (3)	0.8085 (2)	1.0423 (2)	0.0432 (9)
H58	1.0740	0.8064	1.0760	0.052*
C59	0.9911 (3)	0.8815 (2)	1.05346 (19)	0.0342 (8)
C60	0.9044 (3)	0.8826 (2)	1.0058 (2)	0.0363 (8)
H60	0.8847	0.9315	1.0134	0.044*
C61	0.8463 (3)	0.8113 (2)	0.9465 (2)	0.0363 (8)
H61	0.7867	0.8127	0.9142	0.044*
C62	1.0251 (3)	1.0200 (2)	1.1296 (2)	0.0486 (11)
H62A	1.0767	1.0631	1.1728	0.073*
H62B	0.9641	1.0070	1.1388	0.073*
H62C	1.0135	1.0397	1.0893	0.073*
C63	0.6733 (3)	0.5582 (2)	0.86088 (19)	0.0311 (7)
C64	0.6688 (3)	0.5769 (2)	0.9295 (2)	0.0380 (8)
H64	0.7189	0.6216	0.9690	0.046*
C65	0.5922 (3)	0.5310 (2)	0.9408 (2)	0.0422 (8)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H65	0.5904	0.5441	0.9878	0.051*
C66	0.5182 (3)	0.4661 (2)	0.8835 (2)	0.0401 (8)
C67	0.5210 (3)	0.4466 (2)	0.8153 (2)	0.0414 (8)
H67	0.4707	0.4022	0.7758	0.050*
C68	0.5983 (3)	0.4929 (2)	0.8055 (2)	0.0378 (8)
H68	0.5998	0.4792	0.7585	0.045*
C69	0.3626 (4)	0.3628 (3)	0.8427 (3)	0.0692 (15)
H69A	0.3131	0.3430	0.8613	0.104*
H69B	0.3848	0.3168	0.8208	0.104*
H69C	0.3334	0.3854	0.8064	0.104*
C70	0.6205 (2)	0.8299 (2)	0.74558 (18)	0.0271 (7)
H70A	0.5702	0.8490	0.7168	0.033*
H70B	0.5854	0.7860	0.7580	0.033*
C71	0.6871 (3)	0.9028 (2)	0.81588 (18)	0.0299 (7)
H71A	0.7139	0.9500	0.8034	0.036*
H71B	0.7435	0.8865	0.8410	0.036*
C72	0.7184 (3)	1.0197 (2)	0.9956 (2)	0.0398 (8)
C73	0.7970 (3)	1.0849 (3)	1.0040 (2)	0.0517 (10)
H73	0.8033	1.0929	0.9622	0.062*
C74	0.8647 (4)	1.1375 (3)	1.0702 (2)	0.0607 (11)
H74	0.9163	1.1813	1.0738	0.073*
C75	0.8572 (4)	1.1264 (3)	1.1300 (3)	0.0588 (10)
C76	0.7827 (4)	1.0651 (3)	1.1267 (2)	0.0556 (10)
H76	0.7781	1.0590	1.1696	0.067*
C77	0.7106 (4)	1.0093 (3)	1.0567 (2)	0.0499 (9)
H77	0.6585	0.9660	1.0533	0.060*
C78	0.9315 (6)	1.1711 (5)	1.2560 (3)	0.101 (2)
H78A	0.9729	1.2212	1.2978	0.151*
H78B	0.9572	1.1247	1.2598	0.151*
H78C	0.8637	1.1585	1.2547	0.151*
C79	0.5180 (3)	0.8392 (2)	0.8914 (2)	0.0364 (8)
C80	0.4271 (3)	0.8421 (3)	0.8958 (2)	0.0459 (9)
H80	0.4095	0.8916	0.8988	0.055*
C81	0.3613 (3)	0.7751 (3)	0.8958 (3)	0.0513 (9)
H81	0.3003	0.7796	0.8999	0.062*
C82	0.3845 (3)	0.7016 (3)	0.8900 (2)	0.0468 (9)
C83	0.4744 (3)	0.6966 (3)	0.8858 (2)	0.0447 (9)
H83	0.4914	0.6467	0.8823	0.054*
C84	0.5398 (3)	0.7651 (2)	0.8868 (2)	0.0418 (8)
H84	0.6015	0.7611	0.8842	0.050*
C85	0.3365 (4)	0.5621 (3)	0.8832 (3)	0.0632 (13)
H85A	0.2798	0.5212	0.8809	0.095*
H85B	0.3941	0.5707	0.9255	0.095*
H85C	0.3507	0.5420	0.8395	0.095*
C86	0.5305 (3)	1.0189 (2)	0.85138 (19)	0.0344 (8)
C87	0.5418 (3)	1.0986 (3)	0.8957 (2)	0.0519 (10)
H87	0.5822	1.1177	0.9453	0.062*
C88	0.4958 (4)	1.1504 (3)	0.8694 (2)	0.0577 (11)
H88	0.5054	1.2046	0.9007	0.069*
C89	0.4356 (3)	1.1235 (3)	0.7976 (2)	0.0452 (9)
C90	0.4200 (3)	1.0447 (3)	0.7531 (2)	0.0462 (9)
H90	0.3772	1.0252	0.7040	0.055*
C91	0.4671 (3)	0.9934 (2)	0.7803 (2)	0.0431 (9)
H91	0.4555	0.9387	0.7490	0.052*
C92	0.3467 (4)	1.1602 (3)	0.7009 (2)	0.0645 (14)
H92A	0.3214	1.2054	0.6927	0.097*

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H92B	0.2919	1.1091	0.6809	0.097*
H92C	0.3940	1.1525	0.6771	0.097*
O13	0.1527 (4)	0.6799 (3)	0.0591 (2)	0.1061 (14)
C100	0.1066 (5)	0.5940 (4)	0.0231 (3)	0.0869 (15)
H10A	0.0603	0.5826	−0.0261	0.104*
H10B	0.0689	0.5726	0.0496	0.104*
C101	0.2401 (6)	0.6986 (5)	0.0437 (4)	0.1161 (19)
H10C	0.2863	0.7531	0.0778	0.139*
H10D	0.2244	0.6975	−0.0062	0.139*
C102	0.2817 (6)	0.6285 (5)	0.0538 (4)	0.123 (2)
H10E	0.3168	0.6391	0.1054	0.147*
H10F	0.3269	0.6194	0.0277	0.147*
C103	0.1847 (6)	0.5531 (5)	0.0197 (3)	0.1052 (19)
H10G	0.1734	0.5204	−0.0307	0.126*
H10H	0.1878	0.5165	0.0480	0.126*
C93	0.7983 (5)	0.3304 (4)	0.3307 (3)	0.0979 (17)
C94	0.7236 (6)	0.2598 (5)	0.3123 (4)	0.134 (2)
H94	0.6929	0.2229	0.2636	0.160*
C95	0.6931 (6)	0.2422 (5)	0.3635 (4)	0.134 (2)
H95	0.6441	0.1918	0.3508	0.161*
C96	0.7327 (5)	0.2965 (5)	0.4323 (3)	0.111 (2)
H96	0.7095	0.2856	0.4676	0.133*
C97	0.8056 (5)	0.3665 (4)	0.4506 (3)	0.1010 (18)
H97	0.8334	0.4044	0.4990	0.121*
C98	0.8396 (5)	0.3836 (4)	0.4013 (3)	0.1009 (17)
H98	0.8920	0.4323	0.4154	0.121*
C99	0.8357 (7)	0.3493 (5)	0.2748 (4)	0.128 (3)
H99A	0.9076	0.3732	0.2961	0.193*
H99B	0.8057	0.3888	0.2586	0.193*
H99C	0.8179	0.2982	0.2335	0.193*

Source of material

Tri(4-methoxyphenyl)tin hydride (33.08 g; 75.0 mmol) was treated with tetravinylsilane (2.04 g; 15.0 mmol) and five drops of a 0.1 M solution of hexachloroplatinic acid in 2-propanol. After vigorously stirring the reaction mixture at room temperature for 12 h, pentane was added to the viscous product to give a solid, which was filtered and washed with pentane. Recrystallization from toluene-tetrahydrofuran yielded the product as colorless crystals.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. Some disordered C atoms (C72–C77 and C93–C98) were refined using tools available from SHELXL [2].

Comment

The synthesis and characterization of well-defined heteroatomic macromolecules such as single-source organometallic precursors [4–11] or metallodendrimers has developed very rapidly in recent years due to their diverse application possibilities in combination with very special properties [4]. Metallodendrimers are specified by the incorporation of metal atoms as the central unit, as branching centers, in the branches themselves or at the periphery of the dendritic skeleton [12]. We systematically study the synthesis and properties of dendritic organotin compounds [13–17]. The title compound tetrakis[2-(tris(4-methoxyphenyl)stannyl) ethyl]silane combines the properties of a dendritic molecule with the potential functionalizable organotin moities by replacing peripheral 4-methoxyphenyl groups with halogens and other modifications. The title compoud thereby is an appreciable key intermediate for new approaches to further derivatization and syntheses of higher-order organotin dendrimers with metal atoms as branch sites.

X-ray structure analysis reveals crystallographic data of a first-generation Si–Sn dendrimer. Four dendritic molecules are present in the unit cell. The central Si atom is tetrahedrally coordinated by four dendritic branches. The title compound shows a twisted internal geometry instead of an elongated dendritic backbone, indicating a conformational restriction that is likely due to the tendency to utilize the interior of the dendrimer when large substituents are present on the surface. The C–Si–C angles in the range from 107.37(14)° to 110.81(15)° evidence further an increased conformational restriction. All Sn–C bond lengths are comparable with that of closely related tetrakis [2-(triphenylstannyl)-ethylene]silane (2.144 Å(mean)) [18] and other similar compounds in literature [18, 19]. In addition, all bond lengths and bond angles are in the normal range. The structure of the title compound contains tetrahydrofuran and toluene as independent molecules.

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