syntheses using *NRCVAX*. All H atoms were calculated initially and refined in the final cycles. Anomalous-dispersion terms (Cromer & Liberman, 1970) were included for I, Sn and Cl. The final Fourier map showed seven peaks of 0.73–0.45 e $Å^{-3}$ at 0.41–1.69 Å from I and four peaks of 0.60–0.44 e $Å^{-3}$ at 1.32–1.49 Å from Sn. The background was ≤ 0.44 e $Å^{-3}$.

Stereodiagrams for compounds (1), (2) and (3) were produced using *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data, intermolecular contacts and H-atom geometry for (II), and stereoviews of the three unit cells, *ORTEP* views of the solvate molecules and a view of the numbering scheme for molecule (1) of (II) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71594 (179 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1044]

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{Hexakis[2,4,6-tris(trifluoromethyl)phenyl]-cyclotristannoxane}$_2$ †

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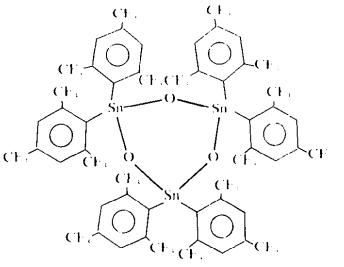
Abstract

The structure of the title compound, $[Sn_3O_3(C_9H_2F_9)_6]$, is reported. There are two molecules of hexakis[2,4,6-tris(trifluoromethyl)phenyl]cyclotristannoxane in the asymmetric unit; 20 of the 36 trifluoromethyl groups are disordered.

Comment

The title compound (I) is a derivative of the monomer bis[2,4,6-tris(trifluoromethyl)phenyl]stannylene (Grützmacher, Pritzkow & Edelmann, 1991) and is stabilized by



(I)

† Alternative name: *cyclo*-tri-μ-oxo-tris{bis[2,4,6-tris(trifluoromethyl)phenyl]tin}.

bridging O atoms. The mean values of distances and angles in the Sn environment are: Sn—O 1.931 (5), Sn—C 2.203 (8) Å, Sn—O—Sn 135.5 (3), O—Sn—O 104.3 (2) and C—Sn—C 110.1 (3)°. Thus, the compound shows pseudo-tetrahedral coordination for the Sn atoms, as expected, with bond distances and angles in good agreement with published results for similar compounds with an Sn—O six-membered ring (Masamune & Sita, 1983; Belsky, Zemlyansky, Borisova, Kolosova & Beletskaya, 1983; Puff, Schuh, Sievers, Wald & Zimmer, 1984).

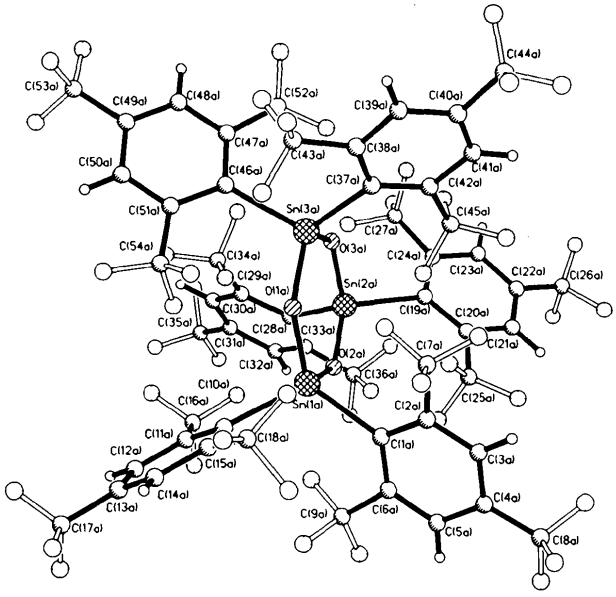


Fig. 1. View of one of the molecules of compound (I). For clarity, the minor components of the 8 disordered trifluoromethyl groups (atom labels ending in *b*) are omitted. The second molecule is also omitted for clarity.

Experimental

Compound (I) was synthesized according to the procedure described by Lay, Pritzkow & Grützmacher (1992).

Crystal data

$[Sn_3O_3(C_9H_2F_9)_6]$
M_r = 2090.2
Monoclinic
$P2_1/c$
a = 24.842 (5) Å
b = 22.471 (4) Å
c = 24.656 (5) Å
β = 111.16 (3)°
V = 12836 (4) Å^3
Z = 8
D_x = 2.164 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 56 reflections
θ = 20–25°
μ = 1.361 mm^{-1}
T = 153 (2) K
Blocks
0.6 × 0.4 × 0.4 mm
Bright yellow

Data collection

Stoe Siemens AED four-circle diffractometer
13 452 observed reflections [$I > 2\sigma(I)$]
Profile data from $2\theta/\omega$ scans
Absorption correction: empirical
T_{min} = 0.562, T_{max} = 0.725
16 442 measured reflections
16 269 independent reflections
R_{int} = 0.065
θ_{max} = 25.08°
h = 0 → 26
k = −24 → 0
l = −29 → 18
3 standard reflections
frequency: 90 min
intensity variation: 5%

Refinement

Refinement on F^2
$R(F)$ = 0.0456
$wR(F^2)$ = 0.1141
S = 1.074
16 269 reflections
2253 parameters
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 73.5P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.927 e Å^{-3}
$\Delta\rho_{min}$ = −0.852 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Sn1*a*	0.42936 (2)	0.33236 (2)	0.82783 (2)	0.02294 (14)
Sn2*a*	0.44456 (2)	0.22865 (2)	0.94322 (2)	0.02281 (15)
Sn3*a*	0.46325 (2)	0.17891 (2)	0.81468 (2)	0.02220 (14)
O1*a*	0.4418 (2)	0.2606 (2)	0.7920 (2)	0.0307 (13)
O2*a*	0.4340 (3)	0.3036 (3)	0.9034 (2)	0.0385 (15)
O3*a*	0.4614 (2)	0.1713 (2)	0.8920 (2)	0.0278 (12)
C1*a*	0.4955 (3)	0.4033 (3)	0.8576 (3)	0.023 (2)
C2*a*	0.5520 (3)	0.3997 (3)	0.8598 (3)	0.023 (2)
C7*a*	0.5737 (4)	0.3542 (4)	0.8269 (4)	0.038 (2)
F1*a*	0.6257 (2)	0.3352 (3)	0.8574 (3)	0.075 (2)
F2*a*	0.5796 (3)	0.3814 (3)	0.7801 (3)	0.076 (2)
F3*a*	0.5400 (2)	0.3097 (2)	0.8066 (3)	0.057 (2)
C3*a*	0.5934 (3)	0.4403 (4)	0.8898 (4)	0.032 (2)
C4*a*	0.5805 (4)	0.4871 (4)	0.9187 (4)	0.031 (2)
C8*a*	0.6266 (4)	0.5288 (4)	0.9522 (4)	0.046 (2)
F4*a*	0.6724 (3)	0.5007 (3)	0.9886 (3)	0.083 (2)
F5*a*	0.6096 (3)	0.5680 (3)	0.9835 (3)	0.065 (2)
F6*a*	0.6456 (2)	0.5603 (2)	0.9174 (2)	0.0559 (15)
C5*a*	0.5242 (4)	0.4937 (4)	0.9162 (3)	0.032 (2)
C6*a*	0.4833 (3)	0.4526 (3)	0.8859 (3)	0.027 (2)
C9*a*	0.4232 (4)	0.4670 (4)	0.8822 (4)	0.032 (2)
F7*a*	0.3915 (2)	0.4194 (2)	0.8840 (2)	0.0409 (12)
F8*a*	0.3931 (2)	0.4929 (3)	0.8310 (3)	0.061 (2)
F9*a*	0.4205 (2)	0.5030 (3)	0.9233 (3)	0.060 (2)
C10*a*	0.3482 (3)	0.3666 (3)	0.7630 (3)	0.028 (2)
C11*a*	0.2940 (3)	0.3647 (4)	0.7669 (4)	0.034 (2)
C16*a*	0.2805 (4)	0.3363 (4)	0.8160 (4)	0.041 (2)
F10*a*	0.3247 (3)	0.3114 (5)	0.8555 (4)	0.054 (2)
F11*a*	0.2406 (4)	0.2942 (5)	0.7962 (4)	0.060 (2)
F12*a*	0.2597 (6)	0.3755 (4)	0.8426 (4)	0.059 (2)
F10*b*	0.2267 (7)	0.3222 (12)	0.8031 (9)	0.054 (2)
F11*b*	0.2956 (12)	0.3699 (10)	0.8631 (8)	0.060 (2)
F12*b*	0.3111 (11)	0.2863 (9)	0.8330 (11)	0.059 (2)
C12*a*	0.2458 (4)	0.3889 (4)	0.7245 (4)	0.036 (2)
C13*a*	0.2500 (4)	0.4164 (4)	0.6757 (4)	0.039 (2)
C17*a*	0.1977 (4)	0.4436 (6)	0.6311 (5)	0.056 (3)
F13*a*	0.2098 (3)	0.4726 (4)	0.5911 (3)	0.095 (3)
F14*a*	0.1716 (2)	0.4815 (3)	0.6558 (3)	0.068 (2)
F15*a*	0.1582 (3)	0.4021 (3)	0.6045 (3)	0.075 (2)
C14*a*	0.3023 (4)	0.4176 (4)	0.6701 (4)	0.039 (2)
C15*a*	0.3505 (4)	0.3942 (4)	0.7124 (4)	0.036 (2)
C18*a*	0.4051 (4)	0.4013 (4)	0.7016 (4)	0.046 (2)
F16*a*	0.4019 (4)	0.4084 (6)	0.6495 (3)	0.064 (3)
F17*a*	0.4466 (4)	0.3636 (5)	0.7291 (6)	0.045 (2)
F18*a*	0.4319 (3)	0.4563 (4)	0.7280 (5)	0.056 (2)

F16*b*	0.4488 (11)	0.3680 (15)	0.7321 (14)	0.064 (3)
F17*b*	0.4177 (8)	0.4508 (7)	0.6903 (11)	0.045 (2)
F18*b*	0.3922 (9)	0.3694 (11)	0.6458 (8)	0.056 (2)
C19*a*	0.5193 (3)	0.2248 (3)	1.0259 (3)	0.027 (2)
C20*a*	0.5562 (3)	0.2723 (4)	1.0510 (3)	0.030 (2)
C25*a*	0.5578 (3)	0.3304 (4)	1.0210 (4)	0.036 (2)
F19*a*	0.5543 (4)	0.3238 (3)	0.9669 (3)	0.047 (2)
F20*a*	0.6041 (4)	0.3636 (4)	1.0458 (4)	0.063 (2)
F21*a*	0.5134 (3)	0.3682 (3)	1.0162 (4)	0.051 (2)
F19*b*	0.5498 (11)	0.3735 (7)	1.0535 (9)	0.047 (2)
F20*b*	0.5234 (11)	0.3343 (11)	0.9686 (8)	0.063 (2)
F21*b*	0.6125 (8)	0.3371 (10)	1.0227 (11)	0.051 (2)
C21*a*	0.5968 (4)	0.2695 (5)	1.1074 (4)	0.039 (2)
C22*a*	0.6024 (4)	0.2181 (4)	1.1397 (4)	0.040 (2)
C26*a*	0.6443 (4)	0.2154 (5)	1.2010 (4)	0.059 (3)
F22*a*	0.6880 (4)	0.2565 (8)	1.2098 (4)	0.074 (3)
F23*a*	0.6659 (6)	0.1661 (6)	1.2207 (4)	0.073 (3)
F24*a*	0.6174 (5)	0.2374 (6)	1.2362 (4)	0.062 (2)
F22*b*	0.6463 (13)	0.1547 (9)	1.2174 (13)	0.074 (3)
F23*b*	0.6334 (13)	0.2420 (16)	1.2410 (10)	0.073 (3)
F24*b*	0.6980 (7)	0.2222 (17)	1.2038 (9)	0.062 (2)
C23*a*	0.5695 (4)	0.1697 (4)	1.1151 (4)	0.041 (2)
C24*a*	0.5280 (3)	0.1726 (4)	1.0588 (3)	0.033 (2)
C27*a*	0.4949 (4)	0.1165 (4)	1.0385 (4)	0.042 (2)
F25*a*	0.4823 (6)	0.0858 (7)	1.0767 (5)	0.043 (3)
F26*a*	0.5264 (6)	0.0769 (5)	1.0192 (9)	0.056 (2)
F27*a*	0.4479 (7)	0.1221 (10)	0.9910 (6)	0.055 (3)
F25*b*	0.4541 (7)	0.1186 (9)	0.9877 (6)	0.043 (3)
F26*b*	0.4647 (8)	0.1082 (8)	1.0754 (6)	0.056 (2)
F27*b*	0.5271 (6)	0.0707 (5)	1.0454 (8)	0.055 (3)
C28*a*	0.3640 (3)	0.2138 (3)	0.9592 (3)	0.024 (2)
C29*a*	0.3185 (3)	0.1786 (4)	0.9246 (3)	0.030 (2)
C34*a*	0.3187 (4)	0.1498 (5)	0.8700 (4)	0.044 (2)
F28*a*	0.3406 (2)	0.1851 (2)	0.8396 (2)	0.0452 (13)
F29*a*	0.2665 (3)	0.1343 (4)	0.8346 (3)	0.084 (2)
F30*a*	0.3507 (3)	0.1006 (3)	0.8807 (3)	0.064 (2)
C30*a*	0.2701 (4)	0.1683 (4)	0.9379 (4)	0.047 (2)
C31*a*	0.2648 (5)	0.1915 (5)	0.9875 (4)	0.056 (3)
C35*a*	0.2129 (6)	0.1792 (6)	1.0014 (5)	0.089 (3)
F31*a*	0.2098 (6)	0.1186 (5)	1.0092 (7)	0.104 (3)
F32*a*	0.1637 (4)	0.1905 (8)	0.9578 (5)	0.099 (4)
F33*a*	0.2108 (6)	0.2046 (6)	1.0483 (5)	0.095 (3)
F31*b*	0.1756 (12)	0.2246 (15)	0.9846 (21)	0.104 (3)
F32*b*	0.2315 (12)	0.1782 (21)	1.0619 (8)	0.099 (4)
F33*b*	0.1843 (15)	0.1311 (15)	0.9844 (17)	0.095 (3)
C32*a*	0.3081 (4)	0.2274 (4)	1.0224 (4)	0.041 (2)
C33*a*	0.3562 (4)	0.2386 (4)	1.0089 (3)	0.030 (2)
C36*a*	0.4002 (4)	0.2782 (4)	1.0499 (4)	0.034 (2)
F34*a*	0.4294 (2)	0.3100 (2)	1.0234 (2)	0.0430 (13)
F35*a*	0.4398 (2)	0.2482 (3)	1.0930 (2)	0.0483 (13)
F36*a*	0.3762 (2)	0.3180 (2)	1.0746 (2)	0.0477 (13)
C37*a*	0.5499 (3)	0.1604 (3)	0.8125 (3)	0.026 (2)
C38*a*	0.5573 (3)	0.1365 (4)	0.7635 (3)	0.029 (2)
C43*a*	0.5085 (4)	0.1273 (5)	0.7073 (4)	0.042 (2)
F37*a*	0.4819 (2)	0.0747 (3)	0.7053 (3)	0.063 (2)
F38*a*	0.5243 (2)	0.1289 (3)	0.6616 (2)	0.068 (2)
F39*a*	0.4681 (2)	0.1690 (3)	0.6986 (2)	0.057 (2)
C39*a*	0.6111 (4)	0.1187 (4)	0.7642 (4)	0.036 (2)
C40*a*	0.6593 (4)	0.1260 (4)	0.8128 (4)	0.045 (2)
C44*a*	0.7156 (4)	0.1056 (5)	0.8124 (4)	0.060 (3)
F40*a*	0.7604 (3)	0.1217 (6)	0.8589 (4)	0.071 (3)
F41*a*	0.7285 (4)	0.1356 (5)	0.7696 (4)	0.074 (2)
F42*a*	0.7190 (5)	0.0511 (4)	0.7997 (6)	0.087 (3)
F40*b*	0.7059 (11)	0.0547 (11)	0.7788 (12)	0.071 (3)
F41*b*	0.7467 (9)	0.0786 (15)	0.8638 (8)	0.074 (2)
F42*b*	0.7466 (10)	0.1385 (11)	0.7961 (15)	0.087 (3)
C41*a*	0.6544 (4)	0.1504 (5)	0.8616 (4)	0.046 (2)
C42*a*	0.6001 (4)	0.1667 (4)	0.8617 (4)	0.040 (2)
C45*a*	0.5997 (4)	0.1929 (6)	0.9174 (4)	0.061 (3)
F43*a*	0.6504 (3)	0.2178 (5)	0.9487 (3)	0.115 (4)
F44*a*	0.5872 (3)	0.1520 (4)	0.9499 (3)	0.086 (2)
F45*a*	0.5609 (2)	0.2367 (3)	0.9083 (3)	0.061 (2)
C46*a*	0.3958 (3)	0.1175 (3)	0.7619 (3)	0.025 (2)
C47*a*	0.4005 (3)	0.0578 (4)	0.7761 (3)	0.028 (2)
C52*a*	0.4484 (4)	0.0306 (4)	0.8257 (4)	0.034 (2)
F46*a*	0.4320 (3)	0.0172 (3)	0.8706 (3)	0.076 (2)
F47*a*	0.4941 (2)	0.0657 (2)	0.8488 (2)	0.0477 (13)
F48*a*	0.4678 (3)	−0.0193 (2)	0.8124 (3)	0.068 (2)
C48*a*	0.3592 (4)	0.0161 (4)	0.7455 (4)	0.031 (2)
C49*a*	0.3123 (4)	0.0343 (4)	0.6988 (4)	0.038 (2)
C53*a*	0.2680 (4)	−0.0092 (4)	0.6643 (4)	0.046 (2)
F49*a*	0.2724 (4)	−0.0218 (5)	0.6144 (4)	0.053 (2)
F50*a*	0.2155 (3)	0.0103 (6)	0.6551 (7)	0.075 (3)
F51*a*	0.2715 (6)	−0.0621 (4)	0.6909 (4)	0.071 (3)
F49*b*	0.2204 (8)	−0.0116 (13)	0.6724 (11)	0.053 (2)
F50*b*	0.2881 (9)	−0.0641 (8)	0.6674 (17)	0.075 (3)
F51*b*	0.2514 (14)	0.0027 (14)	0.6060 (7)	0.071 (3)
C50*a*	0.3054 (4)	0.0944 (4)	0.6840 (4)	0.034 (2)
C51*a*	0.3460 (3)	0.1353 (4)	0.7142 (3)	0.024 (2)
C54*a*	0.3331 (3)	0.1985 (4)	0.6944 (3)	0.028 (2)
F52*a*	0.3209 (2)	0.2318 (2)	0.7341 (2)	0.0347 (11)
F53*a*	0.2868 (2)	0.2027 (2)	0.6453 (2)	0.0438 (13)
F54*a*	0.3753 (2)	0.2259 (2)	0.6834 (2)	0.0353 (11)
Sn1*y*	−0.07383 (2)	0.26525 (2)	0.71810 (2)	0.02543 (15)
Sn2*y*	−0.06389 (2)	0.37529 (2)	0.82637 (2)	0.02605 (15)
Sn3*y*	−0.10816 (3)	0.41660 (3)	0.67688 (2)	0.0303 (2)
O1*y*	−0.0948 (2)	0.3336 (2)	0.6674 (2)	0.0328 (13)
O2*y*	−0.0600 (2)	0.2979 (2)	0.7944 (2)	0.0302 (13)
O3*y*	−0.0936 (3)	0.4262 (3)	0.7592 (2)	0.045 (2)
C1*y*	0.0042 (3)	0.2316 (4)	0.7055 (3)	0.030 (2)
C2*y*	−0.0006 (4)	0.2065 (5)	0.6520 (4)	0.053 (3)
C7*y*	−0.0561 (4)	0.1966 (5)	0.6048 (4)	0.074 (3)
F1*y*	−0.0983 (7)	0.2301 (8)	0.6052 (9)	0.050 (2)
F2*y*	−0.0833 (5)	0.1399 (5)	0.6211 (5)	0.069 (2)
F3*y*	−0.0595 (5)	0.1792 (8)	0.5561 (5)	0.064 (3)
F1*z*	−0.0615 (5)	0.1522 (5)	0.5751 (5)	0.050 (2)
F2*z*	−0.0527 (5)	0.2405 (6)	0.5556 (5)	0.069 (2)
F3*z*	−0.1008 (7)	0.2222 (10)	0.6065 (10)	0.064 (3)
C3*y*	0.0460 (4)	0.1868 (6)	0.6405 (5)	0.071 (4)
C4*y*	0.1008 (5)	0.1924 (6)	0.6801 (5)	0.083 (4)
C8*y*	0.1512 (6)	0.1732 (7)	0.6676 (5)	0.114 (5)
F4*y*	0.1380 (8)	0.1416 (11)	0.6202 (8)	0.103 (4)
F5*y*	0.1958 (7)	0.1591 (12)	0.7058 (9)	0.130 (7)
F6*y*	0.1714 (8)	0.2282 (8)	0.6481 (11)	0.138 (4)
F4*z*	0.2007 (6)	0.1907 (10)	0.7016 (8)	0.103 (4)
F5*z*	0.1487 (7)	0.1674 (13)	0.6163 (7)	0.130 (7)
F6*z*	0.1617 (7)	0.1111 (8)	0.6906 (10)	0.138 (4)
C5*y*	0.1079 (4)	0.2176 (4)	0.7335 (4)	0.047 (2)
C6*y*	0.0607 (3)	0.2360 (3)	0.7464 (3)	0.028 (2)
C9*y*	0.0741 (4)	0.2632 (4)	0.8047 (4)	0.034 (2)
F7*y*	0.0478 (2)	0.2361 (2)	0.8361 (2)	0.0430 (12)
F8*y*	0.1303 (2)	0.2620 (3)	0.8362 (2)	0.067 (2)
F9*y*	0.0579 (2)	0.3203 (2)	0.8006 (2)	0.0495 (14)
C10*y*	−0.1434 (3)	0.1993 (4)	0.6971 (3)	0.029 (2)
C11*y*	−0.1993 (3)	0.2078 (4)	0.6555 (3)	0.032 (2)
C16*y*	−0.2207 (4)	0.2667 (4)	0.6265 (4)	0.042 (2)
F10*y*	−0.2085 (2)	0.3110 (2)	0.6648 (2)	0.0450 (13)
F11*y*	−0.1989 (2)	0.2809 (2)	0.5862 (2)	0.0442 (13)
F12*y*	−0.2784 (2)	0.2668 (3)	0.5986 (3)	0.061 (2)
C12*y*	−0.2383 (4)	0.1608 (5)	0.6394 (4)	0.043 (2)
C13*y*	−0.2241 (4)	0.1063 (5)	0.6649 (4)	0.056 (2)
C17*y*	−0.2660 (5)	0.0570 (6)	0.6478 (5)	0.078 (3)
F13*y*	−0.3146 (6)	0.0609 (7)	0.6140 (8)	0.080 (3)
F14*y*	−0.2582 (10)	0.0185 (8)	0.6925 (7)	0.076 (3)
F15*y*	−0.2423 (6)	0.0119 (6)	0.6150 (6)	0.067 (2)
F13*z*	−0.2625 (8)	0.0127 (6)	0.6788 (7)	0.080 (3)
F14*z*	−0.2929 (6)	0.0513 (6)	0.5914 (5)	0.076 (3)
F15*z*	−0.3217 (4)	0.0841 (5)	0.6544 (5)	0.067 (2)
C14*y*	−0.1714 (4)	0.0971 (4)	0.7067 (4)	0.046 (2)
C15*y*	−0.1321 (4)	0.1426 (4)	0.7220 (4)	0.042 (2)
C18*y*	−0.0766 (5)	0.1281 (4)	0.7668 (5)	0.065 (3)
F16*y*	−0.0354 (4)	0.1157 (5)	0.7368 (5)	0.066 (2)
F17*y*	−0.0721 (5)	0.0780 (4)	0.7931 (5)	0.075 (4)
F18*y*	−0.0478 (6)	0.1703 (5)	0.8004 (5)	0.053 (2)
F16*z*	−0.0904 (8)	0.1024 (10)	0.8157 (8)	0.066 (2)
F17*z*	−0.0496 (12)	0.1772 (10)	0.7968 (13)	0.075 (4)
F18*z*	−0.0417 (8)	0.0937 (9)	0.7607 (9)	0.053 (2)
C19*y*	−0.1209 (4)	0.3798 (4)	0.8759 (3)	0.036 (2)
C20*y*	−0.1608 (4)	0.3352 (4)	0.8753 (3)	0.035 (2)
C25*y*	−0.1764 (4)	0.2854 (4)	0.8325 (4)	0.040 (2)
F19*y*	−0.1788 (2)	0.3036 (2)	0.7799 (2)	0.0450 (13)
F20*y*	−0.1385 (2)	0.2406 (2)	0.8476 (2)	0.0532 (14)
F21*y*	−0.2279 (2)	0.2625 (3)	0.8249 (3)	0.066 (2)
C21*y*	−0.1895 (4)	0.3363 (5)	0.9149 (4)	0.047 (3)
C22*y*	−0.1805 (5)	0.3803 (5)	0.9544 (5)	0.060 (3)
C26*y*	−0.2125 (5)	0.3800 (5)	0.9956 (5)	0.079 (3)

F22y	−0.1977 (7)	0.4241 (6)	1.0330 (6)	0.099 (3)
F23y	−0.2030 (6)	0.3308 (5)	1.0264 (5)	0.082 (3)
F24y	−0.2693 (4)	0.3837 (8)	0.9672 (5)	0.088 (3)
F22z	−0.2276 (23)	0.3243 (11)	1.0046 (21)	0.099 (3)
F23z	−0.2587 (14)	0.4114 (20)	0.9805 (15)	0.082 (3)
F24z	−0.1767 (12)	0.3979 (23)	1.0485 (9)	0.088 (3)
C23y	−0.1438 (5)	0.4257 (5)	0.9550 (4)	0.055 (3)
C24y	−0.1152 (4)	0.4263 (4)	0.9165 (4)	0.043 (2)
C27y	−0.0768 (5)	0.4786 (5)	0.9182 (5)	0.054 (3)
F25y	−0.0915 (3)	0.5264 (3)	0.9410 (3)	0.073 (2)
F26y	−0.0780 (3)	0.4932 (3)	0.8654 (3)	0.079 (2)
F27y	−0.0219 (3)	0.4682 (3)	0.9500 (3)	0.088 (2)
C28y	0.0265 (3)	0.3878 (4)	0.8851 (3)	0.026 (2)
C29y	0.0650 (4)	0.4293 (4)	0.8765 (4)	0.038 (2)
C34y	0.0479 (4)	0.4771 (4)	0.8312 (4)	0.050 (2)
F28y	0.0336 (5)	0.4530 (4)	0.7776 (3)	0.072 (2)
F29y	0.0897 (4)	0.5145 (4)	0.8352 (5)	0.074 (3)
F30y	0.0035 (4)	0.5082 (4)	0.8292 (4)	0.062 (2)
F28z	0.0551 (10)	0.5297 (7)	0.8604 (7)	0.072 (2)
F29z	−0.0056 (6)	0.4754 (9)	0.7958 (9)	0.074 (3)
F30z	0.0822 (8)	0.4808 (9)	0.8023 (8)	0.062 (2)
C30y	0.1234 (4)	0.4290 (4)	0.9110 (4)	0.043 (2)
C31y	0.1452 (4)	0.3876 (5)	0.9544 (4)	0.052 (2)
C35y	0.2082 (6)	0.3874 (6)	0.9903 (5)	0.094 (4)
F31y	0.2240 (6)	0.4425 (7)	1.0128 (12)	0.181 (9)
F32y	0.2398 (6)	0.3780 (13)	0.9626 (6)	0.153 (7)
F33y	0.2243 (4)	0.3529 (8)	1.0343 (6)	0.098 (4)
F31z	0.2263 (11)	0.3304 (9)	0.9923 (27)	0.181 (9)
F32z	0.2194 (11)	0.4026 (28)	1.0414 (9)	0.153 (7)
F33z	0.2394 (10)	0.4120 (20)	0.9667 (14)	0.098 (4)
C32y	0.1088 (4)	0.3476 (4)	0.9649 (4)	0.040 (2)
C33y	0.0504 (3)	0.3482 (4)	0.9311 (3)	0.033 (2)
C36y	0.0161 (4)	0.3047 (4)	0.9511 (4)	0.048 (2)
F34y	0.0440 (3)	0.2641 (4)	0.9844 (4)	0.063 (3)
F35y	−0.0304 (3)	0.2858 (5)	0.9104 (4)	0.039 (2)
F36y	−0.0076 (4)	0.3385 (4)	0.9867 (3)	0.056 (2)
F34z	−0.0362 (8)	0.2925 (15)	0.9139 (12)	0.063 (3)
F35z	0.0168 (9)	0.3065 (10)	1.0022 (7)	0.039 (2)
F36z	0.0407 (10)	0.2465 (7)	0.9492 (11)	0.056 (2)
C37y	−0.0535 (4)	0.4782 (4)	0.6503 (4)	0.037 (2)
C38y	−0.0719 (4)	0.5364 (4)	0.6361 (4)	0.041 (2)
C43y	−0.1166 (5)	0.5647 (4)	0.6557 (4)	0.053 (3)
F37y	−0.1244 (3)	0.5349 (2)	0.6990 (2)	0.066 (2)
F38y	−0.1690 (3)	0.5670 (3)	0.6131 (3)	0.067 (2)
F39y	−0.1031 (3)	0.6201 (3)	0.6733 (3)	0.077 (2)
C39y	−0.0490 (5)	0.5732 (4)	0.6042 (4)	0.046 (2)
C40y	−0.0073 (5)	0.5517 (5)	0.5870 (5)	0.065 (3)
C44y	0.0151 (6)	0.5907 (5)	0.5491 (5)	0.078 (3)
F40y	−0.0312 (5)	0.6045 (7)	0.4985 (5)	0.106 (4)
F41y	0.0518 (6)	0.5670 (5)	0.5319 (6)	0.090 (3)
F42y	0.0319 (7)	0.6422 (5)	0.5731 (5)	0.104 (4)
F40z	0.0765 (8)	0.5909 (16)	0.5766 (12)	0.106 (4)
F41z	0.0034 (14)	0.6455 (9)	0.5460 (14)	0.090 (3)
F42z	0.0105 (17)	0.5710 (12)	0.5000 (9)	0.104 (4)
C41y	0.0154 (4)	0.4962 (4)	0.6029 (4)	0.044 (2)
C42y	−0.0073 (4)	0.4593 (4)	0.6345 (4)	0.043 (2)
C45y	0.0224 (4)	0.4008 (5)	0.6522 (5)	0.062 (2)
F43y	0.0782 (3)	0.4018 (5)	0.6619 (6)	0.078 (3)
F44y	−0.0007 (5)	0.3577 (4)	0.6117 (4)	0.069 (3)
F45y	0.0167 (4)	0.3803 (4)	0.7010 (4)	0.066 (2)
F43z	0.0079 (17)	0.3685 (14)	0.6883 (16)	0.078 (3)
F44z	0.0803 (8)	0.4169 (13)	0.6858 (15)	0.069 (3)
F45z	0.0273 (16)	0.3707 (10)	0.6091 (10)	0.066 (2)
C46y	−0.1946 (4)	0.4366 (4)	0.6125 (4)	0.033 (2)
C47y	−0.2427 (4)	0.4539 (4)	0.6250 (4)	0.037 (2)
C52y	−0.2436 (4)	0.4539 (4)	0.6858 (4)	0.047 (2)
F46y	−0.2177 (8)	0.4077 (8)	0.7180 (9)	0.058 (3)
F47y	−0.2933 (6)	0.4602 (11)	0.6912 (7)	0.056 (3)
F48y	−0.2110 (10)	0.5018 (7)	0.7176 (7)	0.061 (2)
F46z	−0.2409 (10)	0.5061 (5)	0.7066 (6)	0.058 (3)
F47z	−0.2062 (7)	0.4179 (7)	0.7207 (7)	0.056 (3)
F48z	−0.2969 (5)	0.4322 (9)	0.6818 (6)	0.061 (2)
C48y	−0.2931 (4)	0.4731 (4)	0.5826 (4)	0.040 (2)
C49y	−0.2980 (4)	0.4751 (4)	0.5258 (4)	0.046 (2)
C53y	−0.3502 (4)	0.5008 (4)	0.4802 (5)	0.057 (2)
F49y	−0.3962 (4)	0.4997 (11)	0.4958 (8)	0.075 (3)
F50y	−0.3636 (9)	0.4747 (7)	0.4293 (5)	0.078 (3)
F51y	−0.3429 (5)	0.5584 (4)	0.4710 (6)	0.068 (3)
F49z	−0.3499 (18)	0.4913 (28)	0.4263 (9)	0.075 (3)
F50z	−0.3531 (20)	0.5597 (9)	0.4830 (22)	0.078 (3)
F51z	−0.3997 (9)	0.4795 (22)	0.4790 (22)	0.068 (3)
C50y	−0.2531 (4)	0.4562 (4)	0.5106 (4)	0.041 (2)
C51y	−0.2027 (4)	0.4360 (4)	0.5534 (4)	0.034 (2)
C54y	−0.1574 (4)	0.4171 (5)	0.5286 (4)	0.050 (3)
F52y	−0.1147 (2)	0.3855 (2)	0.5655 (2)	0.0450 (13)
F53y	−0.1328 (3)	0.4647 (3)	0.5145 (3)	0.072 (2)
F54y	−0.1801 (3)	0.3852 (4)	0.4817 (2)	0.085 (2)

Table 2. *Selected geometric parameters* (Å, °)

Sn1*a*—O1*a*	1.917 (5)	Sn1*y*—O1*y*	1.930 (5)
Sn1*a*—O2*a*	1.936 (5)	Sn1*y*—O2*y*	1.931 (5)
Sn1*a*—C10*a*	2.210 (8)	Sn1*y*—C10*y*	2.191 (8)
Sn1*a*—C1*a*	2.215 (7)	Sn1*y*—C1*y*	2.204 (8)
Sn2*a*—O2*a*	1.920 (6)	Sn2*y*—O3*y*	1.926 (6)
Sn2*a*—O3*a*	1.954 (5)	Sn2*y*—O2*y*	1.927 (5)
Sn2*a*—C28*a*	2.201 (7)	Sn2*y*—C19*y*	2.182 (8)
Sn2*a*—C19*a*	2.208 (8)	Sn2*y*—C28*y*	2.207 (8)
Sn3*a*—O3*a*	1.930 (5)	Sn3*y*—O1*y*	1.923 (5)
Sn3*a*—O1*a*	1.936 (5)	Sn3*y*—O3*y*	1.941 (5)
Sn3*a*—C46*a*	2.199 (8)	Sn3*y*—C37*y*	2.198 (9)
Sn3*a*—C37*a*	2.212 (8)	Sn3*y*—C46*y*	2.206 (8)
O1*a*—Sn1*a*—O2*a*	101.5 (2)	O1*y*—Sn1*y*—O2*y*	103.9 (2)
O1*a*—Sn1*a*—C10*a*	102.5 (3)	O1*y*—Sn1*y*—C10*y*	112.3 (3)
O2*a*—Sn1*a*—C10*a*	122.3 (3)	O2*y*—Sn1*y*—C10*y*	109.6 (3)
O1*a*—Sn1*a*—C1*a*	122.3 (3)	O1*y*—Sn1*y*—C1*y*	103.1 (3)
O2*a*—Sn1*a*—C1*a*	98.1 (3)	O2*y*—Sn1*y*—C1*y*	114.3 (3)
C10*a*—Sn1*a*—C1*a*	111.3 (3)	C10*y*—Sn1*y*—C1*y*	113.2 (3)
O2*a*—Sn2*a*—O3*a*	105.7 (2)	O3*y*—Sn2*y*—O2*y*	104.1 (2)
O2*a*—Sn2*a*—C28*a*	105.1 (3)	O3*y*—Sn2*y*—C19*y*	109.2 (3)
O3*a*—Sn2*a*—C28*a*	115.9 (3)	O2*y*—Sn2*y*—C19*y*	114.2 (3)
O2*a*—Sn2*a*—C19*a*	114.8 (3)	O3*y*—Sn2*y*—C28*y*	118.2 (3)
O3*a*—Sn2*a*—C19*a*	105.4 (3)	O2*y*—Sn2*y*—C28*y*	101.2 (3)
C28*a*—Sn2*a*—C19*a*	110.2 (3)	C19*y*—Sn2*y*—C28*y*	109.8 (3)
O3*a*—Sn3*a*—O1*a*	105.5 (2)	O1*y*—Sn3*y*—O3*y*	105.0 (2)
O3*a*—Sn3*a*—C46*a*	104.1 (2)	O1*y*—Sn3*y*—C37*y*	114.9 (3)
O1*a*—Sn3*a*—C46*a*	110.7 (3)	O3*y*—Sn3*y*—C37*y*	109.9 (3)
O3*a*—Sn3*a*—C37*a*	112.2 (3)	O1*y*—Sn3*y*—C46*y*	106.0 (3)
O1*a*—Sn3*a*—C37*a*	109.9 (3)	O3*y*—Sn3*y*—C46*y*	119.7 (3)
C46*a*—Sn3*a*—C37*a*	114.1 (3)	C37*y*—Sn3*y*—C46*y*	101.7 (3)
Sn1*a*—O1*a*—Sn3*a*	137.6 (3)	Sn3*y*—O1*y*—Sn1*y*	135.3 (3)
Sn2*a*—O2*a*—Sn1*a*	137.3 (3)	Sn2*y*—O2*y*—Sn1*y*	136.4 (3)
Sn3*a*—O3*a*—Sn2*a*	131.8 (3)	Sn2*y*—O3*y*—Sn3*y*	134.6 (3)

Data were collected by the real-time learnt-profile method (Clegg, 1981) and the structure was solved by direct methods using *SHELXS*90 (Sheldrick, 1990). Refinement of the 20 very disordered trifluoromethyl groups was carried out with a constraint-restraint model, refining the occupation factors of the two $-CF_3$ components as p and $1-p$, constraining thermal parameters of the 'opposite' F atoms, restraining displacement components along the bond directions for F and C atoms in each group, and restraining chemically equivalent 1,2 and 1,3 distances to be equal for all members of each trifluoromethyl group. The H atoms were refined with a riding model for the positional parameters and $U(H) = 1.2U_{eq}(C)$. Data collection: *DIF*4 (Stoe & Cie, 1988; Clegg, 1981). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93 (Sheldrick, 1994).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71669 (66 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1066]

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mer-Trichlorotris(pyridine)vanadium(III)

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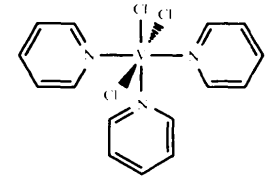
Abstract

The title compound, $[VCl_3(C_5H_5N)_3]$, adopts an octahedral coordination geometry with three chlorides and three pyridine ligands attached to vanadium(III) in the *mer* configuration and with the pyridine planes twisted significantly from the metal–ligand planes.

Comment

In the course of our recent studies of the synthesis and chemistry of mixed-valence nitride-bridged vanadium compounds, we examined the reaction of pyridine with $[V_3(N)_2Cl_7(THF)_2].THF_x$ (Sorensen, Lerchen, Ziller & Doherty, 1992). We had formulated this trivanadium dinitrido species as containing a trichlorovanadium(III) center linked to two vanadium(V) nitrido fragments *via* labile bridging interactions; therefore, we expected to observe the formation of the pyridine adduct of vanadium(III) chloride upon addition of this ligand. Crystalline $[VCl_3(C_5H_5N)_3]$ was isolated from the reaction mixture and its identity confirmed by X-ray crystallography. We describe herein the crystal and molecular structure of this complex (I).



(I)

Although $[VCl_3(C_5H_5N)_3]$ was first prepared over 25 years ago (Fowles & Greene, 1967), the structure of this compound has not been reported. $[VCl_3(C_5H_5N)_3]$ is described as a green crystalline solid isolated from the reaction of $[VCl_3(NMe_3)_2]$ with pyridine in benzene (Fowles & Greene, 1967) or as a blue–violet crystalline compound isolated from the reaction of VCl_3 with pyridine in diethyl ether (Rupp, 1970). We obtained $[VCl_3(C_5H_5N)_3]$ as pale yellow plates at room temperature from the reaction of $[V_3(N)_2Cl_7(THF)_2].THF_x$ with approximately 10 equivalents of pyridine in C_6D_6 in a sealed NMR tube (although bulk samples of the crude product appear green in color).

The molecular structure of $[VCl_3(C_5H_5N)_3]$ shows a roughly octahedral geometry with meridional coordination of the three chlorides and three pyridine ligands, similar to the structure observed at the vanadium(III) center in $[(Me_2NCH_2CH_2NMe_2)Cl_2V^V{=}N{:}]{\rightarrow}[V^{III}Cl_3(Me_2NCH_2CH_2NMe_2)]$ (Sorensen, Lerchen, Ziller & Doherty, 1992). The pyridine ligands in $[VCl_3(C_5H_5N)_3]$ are planar, coordinated end-on and twisted about the V—N bond axes by angles of 52 [N(1)–pyridine], 42 [N(2)–pyridine] and 55 [N(3)–pyridine], relative to the N(1)–N(2)–N(3)–Cl(2) ligand plane. We have noted a similar twisting of the pyridine ligands in the solid-state structure of $[V(N)Cl_2(C_5H_5N)_2]_\infty$, which additionally exhibits a strong intermolecular π-stacking interaction between pyridines on adjacent vanadium–nitride linear chains (Critchlow, Lerchen, Smith & Doherty, 1988). However, a π interaction is not observed between the pyridine ligands on neighboring molecules in the solid-state structure of $[VCl_3(C_5H_5N)_3]$, suggesting that the orientation of the pyridine ligands may be driven by minimization of intramolecular ligand repulsions.

The V—Cl distances for $[VCl_3(C_5H_5N)_3]$ are in the range observed for other nitrogen-base adducts of