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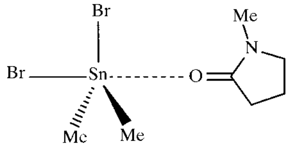
The title compound, dibromodimethyl(*N*-methylpyrrolidin-2-one-*O*)tin(IV), $[SnBr_2(CH_3)_2(C_5H_9NO)]$, exhibits pentacoordination of the Sn atom, with long and short Sn—Br bonds [2.6737 (4) and 2.5256 (4) Å, respectively]. The distorted trigonal–bipyramidal coordination polyhedron has two methyl groups and one Br atom in the equatorial plane, the second Br atom and the *N*-methylpyrrolidinone (NMP) ligand occupying the apical positions.

Comment

Recently, molecular complexes derived from 1:2 complexation of dimethyltin dihalides by *N*-methylpyrrolidinone (NMP) have been presented (König *et al.*, 2000); these have a distorted octahedral geometry. Complexes of dimethyltin dihalides with a single ligand such as DMF, DMSO or HMPT are less well known because several such electron-donor solvents favour a 1:2 composition. The 1:1 reaction of dimethyltin dibromide with NMP leads to the formation of the title complex, (I).



(I)

In the crystal of the title compound, the atoms bound to the Sn atom form a distorted trigonal–bipyramidal geometry. The angle between the two axial ligands NMP and Br2 is 177.50 (5)° and thus close to the ideal angle of 180°. The deviation from ideal geometry is manifested most clearly by the widened C1—Sn—C2 angle of 144.11 (11)° and by the narrowed C1—Sn—Br2 and C2—Sn—Br2 angles of 105.58 (8) and 108.98 (8)°, respectively, compared with a value of 120° for an ideal angle in the equatorial plane. This plane contains two methyl groups and one bromine and is displaced somewhat towards the axial NMP ligand. This becomes obvious from the values of the angles between the equatorial and axial ligands: (i) O1—Sn1—C1 89.35 (9)°, O1—Sn1—C2 84.94 (10)° and O1—Sn1—Br2 84.02 (5)°; (ii) Br1—Sn1—C1 92.82 (7)°, Br1—Sn1—C2 94.00 (8)° and Br1—Sn1—Br2 94.201 (12)°. This geometry has been observed for five other coordinated dimethyltin dihalide complexes (Matsubayashi *et al.*, 1968; Liengme *et al.*, 1972). The Sn—C bond lengths of the title compound [2.107 (3) and 2.119 (2) Å] are in the range of previously reported values of dimethyltin dihalide complexes with NMP ligands (König *et al.*, 2000) and of other five-coordinated methyltin species (Clark *et al.*, 1964; Schlemper & Britton, 1966; Forder & Sheldrick, 1970). The Sn—O bond length in this compound [2.3205 (17) Å] also correlates with known values (König *et al.*, 2000). The two Sn—Br bond lengths in the title compound differ, the values being as follows: (i) $Sn—Br_{ax}$ 2.6737 (4) Å; (ii) $Sn—Br_{eq}$ 2.5256 (4) Å. The Sn—Br bond length in the equatorial plane is comparable with reported values of uncomplexed dimethyltin dibromide (Skinner & Sutton, 1944) and (4-bromo-1,2,3,4-tetraphenyl-*cis,cis*-1,3-butadienyl)dimethyltin bromide (Boer *et al.*, 1970). The bond length between tin and the axial bromine ligand is in the range found for *cis*-dibromo-*trans*-dimethyl-*cis*-bis(*N*-methylpyrrolidinone)tin(IV) (König *et al.*, 2000). The values of the bond lengths and bond angles in the NMP ligand are comparable with those observed in other NMP coordinated organometallic compounds (Churchill & Rotella, 1979) and in free NMP (Müller *et al.*, 1996). The torsion angles vary from 1.3° for C12—C11—N1—C14 to 23.4° for C12—C13—C14—N1, these values in uncomplexed NMP being 4.5 and 19.8°, respectively.

Experimental

The title compound was prepared by the reaction of *N*-methylpyrrolidinone (0.99 g, 0.96 ml, 20 mmol) with freshly sublimed dibromodimethylstannane (3.09 g, 10 mmol) derived from the reaction of dimethyltin oxide with HBr (Pfeiffer, 1902) in 10 ml of dry diethyl ether. The reaction mixture is stirred for 30 min and afterwards stored in a refrigerator at 278 K. Colourless crystals are obtained in quantitative yield after filtration and drying *in vacuo*, m.p. 315 K. A solution of the complex (80 mg) in C_6D_6 (410 mg) gives the following values for the structure-relevant NMR parameters: $^{2}J(^{119}\mathrm{Sn}–^{13}\mathrm{C}–^{1}\mathrm{H})$ = 77 Hz, $^{1}J(^{119}\mathrm{Sn}–^{13}\mathrm{C})$ = 561 Hz and $\delta(^{119}\mathrm{Sn})$ = −34.8 p.p.m. These values represent an equilibrium between complexed and uncomplexed dimethyltin dibromide molecules, which is as expected shifted when the pure complex is studied at 325 K: $^{2}J(^{119}\mathrm{Sn}–^{13}\mathrm{C}–^{1}\mathrm{H})$ = 83 Hz, $^{1}J(^{119}\mathrm{Sn}–^{13}\mathrm{C})$ = 637 Hz, $\delta(^{119}\mathrm{Sn})$ = −95.8 p.p.m.. The structure-relevant FT—IR data are as follows (neat, CsBr plates): ν (cm^{-1}) = 2982 (*vw*, $\nu_{\mathrm{C—H}}$), 2928 (*vw*, $\nu_{\mathrm{C—H}}$), 2897 (*vw*, $\nu_{\mathrm{C—H}}$), 1637 (*versus*, $\nu_{\mathrm{C=O}}$), 562 (*w*, $\nu_{\mathrm{Sn–C}}$), 514 (*w*, $\nu_{\mathrm{Sn–C}}$), 222 (*s*, $\nu_{\mathrm{Sn–Br}}$), 211 (*m*, $\nu_{\mathrm{Sn–Br}}$) (McDermott, 1986; Matsubayashi *et al.*, 1968).

Crystal data

$[SnBr_2(CH_3)_2(C_5H_9NO)]$
$M_r = 407.71$
Monoclinic, $P2_1/c$
$a = 10.652$ (1) Å
$b = 7.002$ (1) Å
$c = 16.427$ (1) Å
$\beta = 93.36$ (1)°
$V = 1223.1$ (2) Å^3
$Z = 4$
$D_x = 2.214$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9229 reflections
$\theta = 2.48$–27.11°
$\mu = 8.585$ mm^{-1}
$T = 173$ (2) K
Parallelepiped, colourless
0.2 × 0.2 × 0.2 mm

Data collection

Nonius KappaCCD diffractometer
626 frames *via* ω rotation ($\Delta\omega = 1°$) at different θvalues and two times 38 s per frame
9229 measured reflections
2682 independent reflections
2379 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\rm max} = 27.11°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.059$
$S = 1.049$
2682 reflections
113 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0317P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.884$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.931$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0025 (3)

The data collections covered the whole sphere of reciprocal space. The crystal to detector distance was 3.5 cm. Crystal decay was monitored by repeating the initial frames at the end of data collection. Analysing the duplicate reflections there were no indications for any decay. The structures were solved by direct methods (Sheldrick, 1990) and successive difference Fourier syntheses. Refinement applied full-matrix least-squares methods (Sheldrick, 1997). All H atoms were placed in calculated positions and refined with a riding model (including free rotation about C—C bonds), and with $U_{\rm iso}$ constrained to be $1.5U_{\rm eq}$ of the carrier atom.

Data collection: Nonius KappaCCD software; cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997) and *PARST*95 (Nardelli, 1995).

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