Derivatives of Bivalent Germanium, Tin, and Lead. Part 21.[1] Tin(II) Formate: A Reinvestigation

By **Philip G. Harrison** * and **Edward W. Thornton**, Department of Chemistry, University of Nottingham, University Park, Nottingham NG7 2RD

The structure of the title compound has been determined from diffractometer data using Patterson and Fourier methods. Crystals are triclinic, $P\bar{1}$, with unit-cell dimensions a = 5.303(2), b = 8.834(3), c = 5.271(2) Å, α = 104.38(2), β = 105.75(2), and γ = 82.82(2)°. Refinement has been carried by full-matrix least squares to a final R value of 0.050 3 for 1 309 independent non-zero reflections. Crystals consist of infinite two-dimensional sheets in which all the formate groups bridge adjacent tin atoms. Tin forms two short [2.14(1) and 2.20(1) Å] and two long [2.36(1) Å] bonds in a pseudo-trigonal-bipyramidal arrangement with the tin lone pair occupying the remaining equatorial site. The structure is disordered such that the formate groups occupy equally two sets of positions between the tin atoms. Vibrational and tin-119m Mössbauer spectra have been recorded, and together with the unit-cell data are compared with data reported previously.

ALTHOUGH tin(II) carboxylates have been known for well over a century,[2] and their synthesis and spectroscopic properties well documented,[3] the structures of only three sophisticated and atypical examples have been determined: (*i*) dihydrogen ethylenediaminetetra-acetatostannate(II), in which the tin atom is centred in the rectangular face of a distorted triangular prism and co-ordinated by two nitrogen and four oxygen atoms;[4] (*ii*) ditin(II) ethylenediaminetetra-acetate dihydrate, in which one tin atom experiences pentagonal-bipyramidal pseudo-seven-co-ordination with the lone pair occupying an equatorial site, whilst the second tin experiences irregular pseudo-eight-co-ordinate geometry in which the lone pair is again stereochemically active;[5] and (*iii*) $[Sn^{II}Sn^{IV}(O_2CC_6H_4NO_2\text{-}o)_4O\cdot C_4H_8O]_2$, the tin(II) atoms of which enjoy pentagonal-pyramidal co-ordination, with the lone pair occupying the second apical site of a bipyramid.[6] However, the structures have been determined of the two simple carboxylatostannate(II) anions $[Sn(O_2CH)_3]^-$ (ref. 7) and $[Sn(C_2O_4)_2]^{2-}$ (ref. 8) as their alkali-metal salts. The former anion has pyramidal geometry with unidentate formate groups,[7] whilst in the latter both oxalate groups chelate the tin resulting in a distorted pseudo-trigonal-bipyramidal stereochemistry, with the lone pair in one of the equatorial sites.[8]

Donaldson and his co-workers [3,9-11] have investigated in some detail the preparation and properties of the simplest tin(II) carboxylate, tin(II) formate, and have concluded from both tin-119m Mössbauer [3] and i.r. data [11] that this derivative enjoys pyramidal co-ordination for the tin atom. In this paper we report the results of an X-ray diffraction study of this compound, which shows that the proposed structure based on spectroscopic data is erroneous, together with a reinvestigation of its tin-119m Mossbauer, and vibrational, data.

EXPERIMENTAL

The crystalline sample of anhydrous tin(II) formate was prepared from tin(II) oxide and formic acid according to Donaldson and Knifton [9] [Found: C, 11.3; H, 0.94. $Sn(O_2CH)_2$ requires C, 11.5; H, 0.97%]. The white crystals (yield 81%) started to darken at 156 °C, were black at 200 °C, but did not change shape on heating to 300 °C. A specimen of dimensions *ca.* 0.4 × 0.4 × 0.3 mm was mounted under nitrogen in a Lindemann capillary and used for both the initial photography and also the subsequent intensity-data collection.

TABLE 1

Crystal data for tin(II) formate, $C_2H_2O_4Sn$, M = 208.72

	This work	Ref. 9
Space group	Triclinic, $P\bar{1}$	Triclinic [a]
a/Å	5.303(2)	10.56(20) (≡ 2 × 5.28)
b/Å	8.834(3)	8.81(20)
c/Å	5.271(2)	10.56(20) (≡ 2 × 5.28)
α/°	104.38(2) (≡ 90 + 14.38)	82.83(8) (≡ 90 − $\frac{1}{2}$ × 14.38)
β/°	105.75(2)	74.83(8) (≡ 180 − 105.17)
γ/°	82.82(2)	82.83(8)
U/Å^3	229.8	937.6
D_c/g cm^{-3}	3.02	2.96
D_m/g cm^{-3}	3.06 [b]	2.98 [c]
Z	2	8
F(000)	192	768
Radiation	Mo-K_α	Cu-K_α
λ/Å	0.710 7	
μ(Mo-K_α)/cm^{-1}	54.62	

[a] The b and c axes of the original cell have been interchanged. [b] By flotation. [c] By displacement.

Crystal Data.—Crystal data for tin(II) formate obtained in the present study are listed in Table 1 together with those obtained previously.[9]

The space group and initial cell parameters were deter-

---

[1] Part 20, A. B. Cornwell, C. A. Cornwell, and P. G. Harrison, *J.C.S. Dalton*, 1976, 1612.

[2] J. Liebig, *Ann. pharm. franç.*, 1836, **17**, 75.

[3] J. D. Donaldson and A. Jelen, *J. Chem. Soc.* (*A*), 1968, 1448, 2244 and refs. therein.

[4] K. G. Shields, R. C. Seccombe, and C. H. L. Kennard, *J.C.S. Dalton*, 1973, 741.

[5] F. P. van Remoortere, J. J. Flynn, F. P. Boer, and P. P. North, *Inorg. Chem.*, 1971, **10**, 1511.

[6] P. F. R. Ewings, P. G. Harrison, A. Morris, and T. J. King, *J.C.S. Dalton*, 1976, 1602.

[7] A. Jelen and O. Lindquist, *Acta Chem. Scand.*, 1969, **23**, 3071.

[8] J. D. Donaldson, M. T. Donoghue, and C. H. Smith, *Acta Cryst.*, 1976, **B32**, 2098.

[9] J. D. Donaldson and J. F. Knifton, *J. Chem. Soc.*, 1964, 4801.

[10] J. D. Donaldson, E. J. Filmore, and M. J. Tricker, *J. Chem. Soc.* (*A*), 1971, 1109.

[11] J. D. Donaldson, J. F. Knifton, and S. D. Ross, *Spectrochim. Acta*, 1964, **20**, 847.

mined from oscillation and zero- and first-layer Weissenberg photographs obtained on an equi-inclination Weissenberg camera. Relative intensities of 1 309 reflections were collected up to θ 30.0° with Mo-K_α radiation by use of a Hilger and Watts Y290 single-crystal four-circle diffractometer, accurate cell dimensions being obtained by least-squares refinement of *ca.* 20 reflections. Each reflection was counted for 30 s and the two associated background counts for 10 s. The reflections were brought to the same relative intensities by use of reference reflections taken for every one hundred reflections recorded; the diffractometer orientation was referenced every two hundred reflections. The intensities were corrected for Lorentz and polarisation effects, but not for absorption.

Structure Determination and Refinement.—The positional parameters of the tin atom were obtained from a three-dimensional Patterson synthesis, and these were used to phase the initial structure-factor calculation. Since a subsequent Fourier synthesis did not yield any chemically sensible information, the position of the tin atom was refined by isotropic least squares, and a second Fourier synthesis afforded the positions of eight light atoms around the tin. These were assigned as half-weight oxygen atoms, and the refinement continued with the tin atom being allowed to vary anisotropically. After three cycles of refinement, a third Fourier synthesis yielded the positions of the two half-weight carbon atoms. After two more cycles of full-matrix refinement with only the tin atom varying anisotropically, followed by two cycles of full-matrix refinement with all the atoms varying anisotropically, the weighting scheme $w^{\frac{1}{2}} = 1$ for $F_o \leqslant 27.0$ and $w^{\frac{1}{2}} = 27/F_o$ for $F_o > 27.0$ was applied. Two additional cycles of full-matrix anisotropic refinement produced convergence at an ' R ' value of 0.0503. The scattering factors used were those for neutral atoms.[12] Calculations were performed using the CRYSTALS suite of programs.[13] The results are collected in Tables 1—3.

TABLE 2

Final fractional atomic co-ordinates, with estimated standard deviations in parentheses

Atom	Occupancy	*x*/*a*	*y*/*b*	*z*/*c*
Sn(1)	1.0	0.236 35(7)	0.130 36(4)	0.301 67(7)
O(1)	0.5	0.148(2)	0.183(1)	0.732(2)
O(2)	0.5	0.308(2)	0.180(1)	0.908(2)
O(3)	0.5	0.956(2)	0.319(1)	0.228(2)
O(4)	0.5	0.477(2)	0.323(1)	0.517(2)
O(5)	0.5	0.821(2)	0.185(1)	0.401(2)
O(6)	0.5	0.644(2)	0.183(1)	0.248(2)
O(7)	0.5	0.080(2)	0.330(1)	0.113(2)
O(8)	0.5	0.355(2)	0.329(1)	0.645(2)
C(1)	0.5	0.074(3)	0.308(2)	0.862(3)
C(2)	0.5	0.358(3)	0.308(2)	0.874(3)
C(3)	0.5	0.722(3)	0.307(2)	0.224(3)
C(4)	0.5	0.717(3)	0.307(2)	0.511(3)

Atomic numbering is shown in Figure 2. Final observed and calculated structure factors, and anisotropic thermal parameters, are listed in Supplementary Publication No. SUP 22308 (15 pp.).*

Infrared spectra in the range 200—4 000 cm^{-1} were recorded as Nujol and halogenocarbon mulls using a Perkin-Elmer 577 spectrometer. Reflectance spectra were obtained from a pressed disc in the range 40—400 cm^{-1} using a Beckmann R11C F.S. 720 Fourier-transform spectrophotometer, Raman spectra using a Carey 81 spectrometer equipped with an argon-ion laser (514.9-nm exciting line). Tin-119m Mössbauer data were collected at 77 K and ambient temperature (using equipment described previously) against a Ba^{119m}SnO$_3$ source (Radiochemical Centre, Amersham). Data reduction and curve-fitting to Lorentzian line shapes was accomplished by usual least-squares methods.

TABLE 3

Bond distances (Å) and angles (°) in tin(II) formate (with estimated standard deviations in parentheses)

(*a*) Bond distances			
Sn(1)–O(1)	2.36(1)	Sn(1)–O(5)	2.36(1)
Sn(1)–O(2)	2.36(1)	Sn(1)–O(6)	2.37(1)
Sn(1)–O(3)	2.13(1)	Sn(1)–O(7)	2.20(1)
Sn(1)–O(4)	2.14$_5$(1)	Sn(1)–O(8)	2.20(1)
C(1)–O(1)	1.23(2)	C(3)–O(3)	1.25(2)
C(1)–O(7)	1.28(2)	C(3)–O(6)	1.26(2)
C(2)–O(2)	1.26(2)	C(4)–O(4)	1.27$_5$(2)
C(2)–O(8)	1.26(2)	C(4)–O(5)	1.23(2)
(*b*) Bond angles			
O(1)–Sn(1)–O(2)	158.6(4)	O(5)–Sn(1)–O(6)	157.7(4)
O(3)–Sn(1)–O(4)	80.7(4)	O(7)–Sn(1)–O(8)	78.7(4)
O(1)–Sn(1)–O(3)	85.7(4)	O(5)–Sn(1)–O(7)	87.6(4)
O(1)–Sn(1)–O(4)	78.4$_5$(4)	O(5)–Sn(1)–O(8)	84.4(4)
O(2)–Sn(1)–O(3)	77.6(4)	O(6)–Sn(1)–O(7)	84.5(4)
O(2)–Sn(1)–O(4)	85.7(4)	O(6)–Sn(1)–O(8)	78.0$_5$(4)
Sn(1)–O(1)–C(1)	128.2(9)	Sn(1)–O(5)–C(4)	131.3(9)
Sn(1)–O(2)–C(2)	127.5(9)	Sn(1)–O(6)–C(3)	131.0(9)
Sn(1)–O(3)–C(3)	118.7(9)	Sn(1)–O(7)–C(1)	116.2(9)
Sn(1)–O(4)–C(4)	117.6$_5$(9)	Sn(1)–O(8)–C(2)	115.7(9)
O(1)–C(1)–O(7)	122.3$_5$(13)	O(3)–C(3)–O(6)	122.5(12)
O(2)–C(2)–O(8)	122.7(12)	O(4)–C(4)–O(5)	123.7(12)

DISCUSSION

Tin-119m Mössbauer and vibrational spectroscopic data previously reported for simple tin(II) carboxylates have been interpreted in terms of trigonal-pyramidal co-ordination for the tin(II) atoms with bridging and/or chelating carboxylate groups.[3,10,11] As the results of the present X-ray diffraction study show, this inference is not valid, at least in the case of tin(II) formate.

Crystals of tin(II) formate are composed of infinite two-dimensional sheets in which tin(II) atoms are bridged by formate groups (Figure 1). Each tin(II) atom is bonded to four oxygen atoms from four different formate groups, the resulting ' local ' stereochemistry at tin being that of a distorted pseudo-trigonal bipyramid, with the lone pair of electrons of the tin(II) atom located in one of the equatorial sites. The geometry is thus similar to that previously observed in bis(1-phenylbutane-1,3-dionato)-tin(II),[14] Sn(SO$_4$)·2SC(NH$_2$)$_2$,[15] SnCl$_2$·dioxan,[16] and the [Sn(O$_4$C$_2$)$_2$]$^{2-}$ anion.[8] The structure of tin(II) formate is, however, disordered such that the formate groups occupy equally two sets of positions between adjacent tin atoms, thus forming two superimposed independent two-dimensional networks (Figure 2). Within each network, the repeating unit involves four mutually bridged tin

---

* For details see Notices to Authors No. 7, *J.C.S. Dalton*, 1977, Index issue.

[12] ' International Tables for X-Ray Crystallography,' Kynoch Press, Birmingham, 1962, vol. 3.

[13] C. M. Carruthers, personal communication.

[14] P. F. R. Ewings, P. G. Harrison, and T. J. King, *J.C.S. Dalton*, 1975, 1455.

[15] J. D. Donaldson, D. G. Nicholson, D. C. Puxley, and R. A. Howie, *J.C.S. Dalton*, 1973, 1810.

[16] E. Hough and D. G. Nicholson, *J.C.S. Dalton*, 1976, 1631.

atoms, which is consistent with a unit cell having *a* and *c* dimensions exactly twice those observed here as obtained by Donaldson and Knifton[9] in their study, suggesting that these workers were investigating a crystal which was not disordered. The effect of the disorder observed in the present study is such as to make the overall co-ordination about each tin atom identical, since about each tin atom are half-weight contributions from two sets of four formate groups. The repeating unit along both the *a* and *c* directions therefore now involves only one tin atom, although equivalent oxygen positions about adjacent tin atoms belong to different networks. Thus, referring to Figure 2, whereas O(5), O(6), O(7), and O(8) about tin atom Sn^{I} belong to network A (full lines) and O(1), O(2), O(3), and O(4) about Sn^{I} belong to network B (broken lines), the situation is reversed for the adjacent tin atoms Sn^{II}, Sn^{III}, Sn^{IV}, and Sn^{V}. The two-dimensional sheets are stacked parallel to the *b* axis, with alternate layers inverted (Figure 3). The layers are staggered such that the tin(II) lone pair is directed towards a ' hole ' in the network of the next layer (Figure 1).

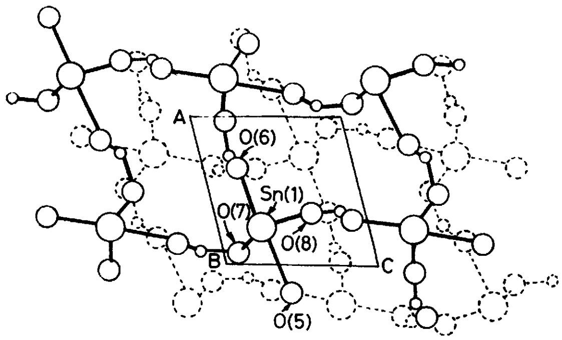

FIGURE 1 Projection on to the *ac* plane showing the formate bridging within the two-dimensional sheets. Only one of the two disordered networks is shown. The broken lines refer to symmetry-related atoms in the lower layer

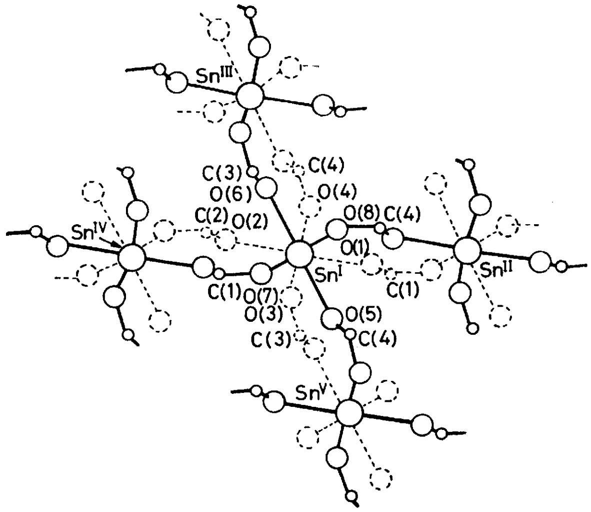

FIGURE 2 View of the structure showing the two disordered networks (solid and broken lines, respectively) along the bisector of the $\mathrm{O_{ax.}{-}Sn{-}O_{ax.}}$ and $\mathrm{O_{eq.}{-}Sn{-}O_{eq.}}$ angles, as well as the atomic numbering

Bond-distance and -angle data for the two sets of oxygen co-ordination about each tin atom are summarised and compared in Figure 4. Both have pseudo-trigonal-bipyramidal co-ordination with one vacant equatorial position, presumably occupied by the lone pair of electrons of the tin(II) atom. The $\mathrm{Sn{-}O_{ax.}}$ bond distances of each set are identical [2.36(1) Å], and are comparable with the equivalent bond distances in the $[\mathrm{Sn(O_4C_2)_2}]^{2-}$ anion[8] and $\mathrm{Sn(SO_4){\cdot}2SC(NH_2)_2}$[15] (Table 4). The shortest such bond distance (except that in SnO)[17] is observed in $\mathrm{Sn(PhCO{\cdot}CH{\cdot}COMe)_2}$ [2.290(6) Å],[13] whilst the longest occurs in $\mathrm{SnCl_2{\cdot}}$dioxan [2.527(5) Å].[16] The $\mathrm{Sn{-}O_{eq.}}$ bond distances are, not unexpectedly, significantly shorter, but are different in the two sets [2.14(1) and 2.20(1) Å]. Both, however, are not atypical (Table 4). The $\mathrm{O_{eq.}{-}Sn{-}O_{eq.}}$ and $\mathrm{O_{ax.}{-}Sn{-}O_{ax.}}$ bond angles in each set are similar, and exhibit the usual reduction from the values expected for ideal trigonal-bipyramidal geometry due to lone-pair–bond-pair repulsion. The $\mathrm{O_{ax.}{-}Sn{-}O_{ax.}}$ angles are not reduced as much as in $\mathrm{Sn(PhCO{\cdot}CH{\cdot}COMe)_2}$[13] or in the $[\mathrm{Sn(O_4C_2)_2}]^{2-}$ anion,[8] where the ligands chelate the tin, but the restrictions of the two-dimensional network do not allow the bond angle to open to the value observed in the one-dimensional co-ordinate polymer $\mathrm{SnCl_2{\cdot}}$dioxan [168.5(2)°],[16] the highest such bond observed. Viewing down the axis bisecting

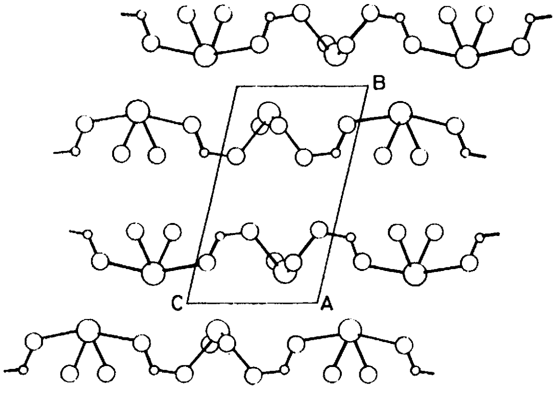

FIGURE 3 Projection on to the *bc* plane illustrating the layering of the two-dimensional sheets within the crystal

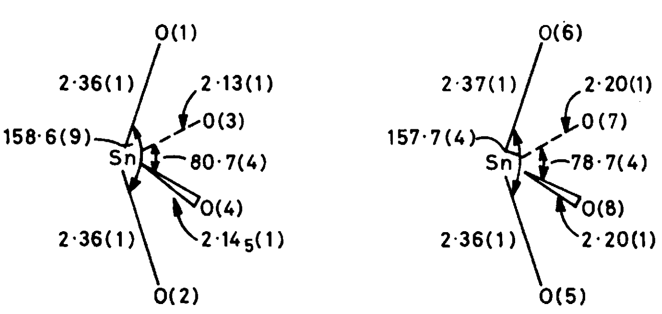

FIGURE 4 Comparison of bond-angle and -distance data for the two disordered sets of oxygen co-ordination about the tin

---

[17] W. J. Moore and L. Pauling, *J. Amer. Chem. Soc.*, 1941, **63**, 1392.

the $O_{ax.}$–Sn–$O_{ax.}$ and $O_{eq.}$–Sn–$O_{eq.}$ bond angles of each individual tin atom, the two SnO_4 skeletons are mutually rotated by *ca.* 54° with respect to each other.

All the formate groups span axial and equatorial co-ordination sites on adjacent tin atoms, and are of two types. Half the formate groups are symmetrical with a C–O bond distance of 1.26(2) Å and an O–C–O bond angle of 122.6(12)°. The remaining formate groups are unsymmetrical, with one short [1.23(2) Å] and one long [1.28(2) Å] bond, although the O–C–O bond angle remains the same [122.3$_5$(13) and 123.7(12)°]. In the latter case, the oxygen atom involved in the longer bond to carbon is bonded to tin *via* an equatorial site, and the oxygen atom involved in the longer bond to carbon bonds to tin *via* an axial site. The formate groups in sodium formate [18] are symmetrical (C–O 1.27 Å, O–C–O 124°), whilst those of ammonium formate [19] are nearly so (C–O 1.237 and 1.246 Å, O–C–O 126.3°), although the network of hydrogen bonds most probably affects the bonding somewhat. In the $[Sn(O_2CH)_3]^-$ anion [7] all the formate groups are unidentate, but one is symmetrical [C–O 1.24$_5$(2) Å, O–C–O 120.8(16)°] whilst in the other two groups the C–O bond distances differ by 0.1 Å [C–O 1.20(2) and 1.30(2) Å, O–C–O 124.3(15) and 126.5(15)°]. The $[Sn(O_4C_2)_2]^{2-}$ anion [8] has C–O bond distances in the range 1.24(2)—1.29(2) Å although the longest bonds are those where the oxygen bonds to tin. Bridging carboxylate groups occur in $[Sn^{II}Sn^{IV}(O_2CC_6H_4NO_2\text{-}o)_4O\cdot thf]_2$ [6] with the C–O–(Sn^{II}) bonds shorter [mean 1.22(1) Å] than the C–O–(Sn^{IV}) distances [1.26(2)—1.30(1) Å], and the tin–oxygen bond distances are consistent with a Sn^{IV}–O–C=O→Sn^{II}] electronic arrangement. Thus, in the present compound, two of the formate groups have a greater contribution of the ester –O–CH=O form than the other two, which are more ionic in character. The Sn–$O_{eq.}$–C bond angle varies only slightly [115.7(4)—118.7(4)°]; the Sn–$O_{ax.}$–C angles are significantly larger [127.5(9)—131.1(9)°].

TABLE 4

Comparison of structural data in tin(II) compounds possessing a distorted pseudo-trigonal-bipyramidal geometry

	Distance/Å		Angle (°)	
Compound	Sn–$O_{eq.}$	Sn–$O_{ax.}$	$O_{eq.}$–Sn–$O_{eq.}$	$O_{ax.}$–Sn–$O_{ax.}$
$Sn(O_2CH)_2$ [a] (*i*)	2.14(1)	2.36(1)	80.7(4)	158.6(4)
(*ii*)	2.20(1)	2.36(1)	78.7(4)	157.7(4)
$Sn(PhCO\cdot CH\cdot COMe)_2$ [b]	2.135(1)	2.290(6)	94.7(1)	150.4(2)
$[Sn(O_4C_2)_2]^{2-}$ [c]	2.25(1)	2.36(1)	78.7(3)	145.5(4)
$SnCl_2\cdot$dioxan [d]		2.527(5)		168.5(2)
$Sn(SO_4)\cdot 2SC(NH_2)_2$ [e]	2.24(2)	2.41(2)	89.6(5) [f]	157.3(4) [g]
SnO [h]	2.21(1)	2.21	75(4)	118(2)

[a] This work. [b] Ref. 14. [c] Ref.8. [d] Ref. 16. [e] Ref. 15. [f] $O_{eq.}$–Sn–$S_{eq.}$. [g] $O_{ax.}$–Sn–$S_{ax.}$. [h] Ref. 17.

TABLE 5

Infrared and Raman bands (cm^{-1}) of tin(II) formate

		Type [a]			I.r.		Raman	
Band	Mode	C_{2v}	C_s	From ref. 11	$\tilde{\nu}$	I [b]	$\tilde{\nu}$	I [b]
ν_1	ν(C–H)	A_1	A	2 874	2 876	7	2 873	66
ν_4	ν_{asym}(OCO)		B	1 563	1 561	100	1 565	66
		B_1			1 548	100		
ν_5	δ(C–H) (in plane)	B_2	B	1 385 1 370	1 386 1 371	26 39	1 388 1 369	49 100
ν_2	ν_{sym}(OCO)	A_1		1 339	1 341	49	1 349	46
			A	1 311	1 330—1 320 (sh) vbr		1 327	63
ν_6	δ(C–H) (out of plane)	B_2	B	1 064	1 071	1	1 069	9
					1 057	1	1 057	9
ν_3	δ_{sym}(OCO)	A_1	A	781	782	31	785	17
	ν(Sn–$O_{eq.}$)				350—360 (sh)		345	
	ν(Sn–$O_{eq.}$)				329	11	327	
	ν(Sn–$O_{ax.}$)				260	9	247	
	δ(OSnO)				189 111			

I.r. overtone: 2 920 (1 371 + 1 548 = 2 919 cm^{-1})

[a] Symmetry types based on ' local ' symmetry for the formate groups. [b] Relative intensity.

The two types of formate groups are manifest in the vibrational spectra, which are listed together with assignments in Table 5 and with the data of Donaldson *et al.*[11] The majority of the fundamentals of the formate groups have two components, although the assignment of bands to the symmetrical formate groups (C_{2v} ' local ' symmetry) or the unsymmetrical formate groups (C_s ' local ' symmetry) is to a large part indeterminate. The bands at <500 cm^{-1} are all metal–ligand vibrations. Those at 350—360 and 329 cm^{-1} may be assigned to Sn–$O_{eq.}$ stretching vibrations, the higher-energy band to the shorter bond, and *vice versa*. The Sn–$O_{ax.}$ stretching vibration is assigned to the band at 260 cm^{-1}. Bands at 189 and 111 cm^{-1} are assigned as O–Sn–O angle-deformation modes.

The tin-119m Mössbauer spectrum of tin(II) formate

---

[18] W. H. Zachariasen, *J. Amer. Chem. Soc.*, 1940, **62**, 1011.

[19] I. Nahringbauer, *Acta Cryst.*, 1968, **B24**, 565.

has been measured previously at 77 K by Donaldson and Jelen,[3] and the sign of the quadrupole coupling constant, $eV_{zz}Q$, has been shown to be positive.[10] As expected, because of its polymeric lattice, tin(II) formate exhibits a Mössbauer spectrum at both 77 K and ambient temperature. The data are listed in Table 6. Both the isomer shift and quadrupole splitting decrease with increasing temperature, and are somewhat higher than those reported previously. A distinct Goldaskii–Karyagin effect is observed at both temperatures. A more detailed investigation of the temperature dependence of the Mössbauer parameters will be reported subsequently.

TABLE 6

Tin-119m Mössbauer data (mm s^{-1}) for tin(II) formate [a]

T/K	I.s.	Q.s.	Γ_1	Γ_2	A_1/A_2
77	3.33	1.70	1.02	1.11	0.78
Ambient	3.27	1.61	0.77	0.78	0.74
b	3.15	1.56			

[a] Relative to $BaSnO_3$ (0 mm s^{-1}). [b] Data at 77 K from ref. 3.

We thank the S.R.C. for support, and Professor T. J. King for collecting the *X*-ray intensity data and helpful discussions.

[7/1973 *Received, 9th November,* 1977]