Acta Cryst. (1983). C**39**, 981–982

Dibromobis[tetracarbonyl(triphenylphosphine)rhenio]tin(IV), $[SnBr_2\{Re(C_{18}H_{15}P)(CO)_4\}_2]$

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(*Received* 20 *January* 1983; *accepted* 11 *April* 1983)

Abstract. $M_r = 1399{\cdot}6$, triclinic, $P\bar{1}$, $a = 9{\cdot}896$ (5), $b = 13{\cdot}404$ (5), $c = 18{\cdot}031$ (6) Å, $\alpha = 77{\cdot}7$ (1), $\beta = 85{\cdot}2$ (1), $\gamma = 75{\cdot}8$ (1)°, $U = 2264$ Å^3, $Z = 2$, $D_x = 2{\cdot}053$ Mg m^{-3}, $F(000) = 1316$, λ(Mo $K\alpha$) = $0{\cdot}71069$ Å, $\mu = 7{\cdot}5$ mm^{-1}, $T = 294$ (1) K. Final $R = 0{\cdot}084$ for 5743 unique diffractometer data ($I > 3\sigma$) and 201 refined parameters. The two $Re[(C_6H_5)_3P](CO)_4$ groups and the two Br atoms form a distorted tetrahedron round the Sn atom with Sn—Re distances 2·741 (2), 2·730 (2) Å and Sn—Br distances 2·565 (4), 2·584 (4) Å.

Introduction. In the course of our investigations on the influence of packing forces in molecular crystals on the geometry of the molecules we have prepared the compound $[SnBr_2\{Re(C_{18}H_{15}P)(CO)_4\}_2]$ and determined the crystal structure.

Experimental. The colourless crystals were prepared by an insertion reaction of $SnBr_2$ with $[Re_2\{(C_6H_5)_3P\}_2(CO)_8]$ in xylene at 448 K during a reaction time of 28 days in a bomb tube.

$\omega/2\theta$ scan, scan speed 0·8°–3·33° min^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, crystal size 0·2 × 0·3 × 0·2 mm, lattice parameters from least-squares fit with 16 reflections in the range $6{\cdot}4 \leq \theta \leq 17{\cdot}3°$; five standard reflections, recorded every 2 h – only random deviations; 9467 reflections measured, $2° \leq \theta \leq 26°$, $\pm h$, $\pm k$, $+l$; after averaging: 5743 observed reflections with $I > 3\sigma(I)$, Lorentz–polarization correction, no absorption correction; structure solution *via* Patterson function, ΔF syntheses and full-matrix least squares refinements with anisotropic temperature factors for Re, Sn, Br and P and isotropic temperature factors for C, O and H; complex neutral-atom scattering factors from Cromer & Mann (1968), Cromer & Liberman (1970) and for H atoms from Cromer (personal communication to G. M. Sheldrick), phenyl rings as rigid bodies (C—C 1·395 Å, C—H 1·08 Å, all angles 120°), least-squares refinements based on F^2 and 201 refined parameters, unit weights, maximum/average shift over error 0·5/0·01, no significant features in a final ΔF synthesis; programs: *SHELX* (Sheldrick, 1976), *ORTEP* (Johnson, 1976), *POP*1 (van de Waal, 1976).

Discussion. The structure of the title compound is shown in Fig. 1 (*ORTEP*) and in a stereoview in Fig. 2 (*POP*1). Positional parameters and the equivalent values of the anisotropic temperature factors β_{ij} or the isotropic temperature factors B are given in Table 1. Bond lengths and angles are given in Table 2.*

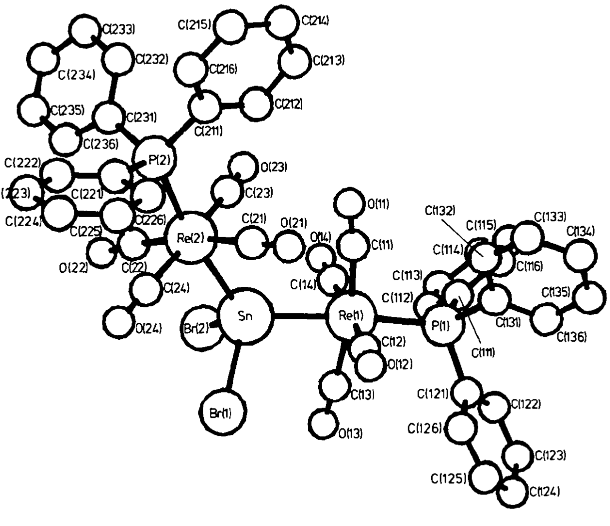

Fig. 1. General view of the molecule.

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* Lists of structure factors, anisotropic thermal parameters and a complete list of atom parameters have been deposited with the Britsh Library Lending Division as Supplementary Publication No. SUP 38523 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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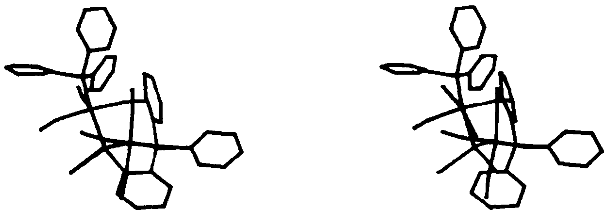

Fig. 2. Stereoscopic view of the molecule.

Table 1. *Fractional atomic coordinates and temperature factors* (Å^2)

For each rigid phenyl ring only the coordinates of three C atoms are given.

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}/B
Sn(1)	0·6638 (2)	0·3630 (1)	0·2647 (1)	2·53 (4)
Br(1)	0·4581 (3)	0·2741 (3)	0·2901 (2)	4·7 (1)
Br(2)	0·8116 (4)	0·2314 (3)	0·1867 (2)	4·52 (9)
Re(1)	0·7925 (1)	0·31905 (8)	0·40062 (6)	2·15 (2)
C(11)	0·808 (3)	0·466 (2)	0·377 (2)	3·0 (5)
O(11)	0·812 (2)	0·552 (2)	0·357 (1)	4·7 (5)
C(12)	0·609 (3)	0·365 (2)	0·448 (2)	3·6 (6)
O(12)	0·500 (3)	0·399 (2)	0·473 (1)	5·5 (5)
C(13)	0·763 (3)	0·176 (2)	0·404 (2)	3·1 (5)
O(13)	0·743 (2)	0·097 (2)	0·404 (1)	5·1 (5)
C(14)	0·971 (3)	0·286 (2)	0·342 (2)	3·4 (6)
O(14)	1·069 (2)	0·274 (2)	0·305 (1)	4·9 (5)
P(1)	0·9010 (7)	0·2644 (5)	0·5227 (4)	2·4 (2)
C(111)	1·092 (2)	0·220 (1)	0·518 (1)	2·9 (5)
C(113)	1·298 (2)	0·112 (1)	0·469 (1)	5·1 (8)
C(115)	1·321 (2)	0·226 (1)	0·552 (1)	4·3 (7)
C(121)	0·848 (1)	0·153 (1)	0·586 (1)	2·5 (5)
C(123)	0·900 (1)	−0·008 (1)	0·680 (1)	4·6 (7)
C(125)	0·660 (1)	0·086 (1)	0·658 (1)	4·9 (7)
C(131)	0·870 (2)	0·355 (1)	0·5874 (7)	2·8 (5)
C(133)	0·814 (2)	0·533 (1)	0·6094 (7)	4·7 (7)
C(135)	0·873 (2)	0·386 (1)	0·7144 (7)	4·0 (6)
Re(2)	0·5983 (1)	0·55700 (8)	0·17260 (7)	2·59 (3)
C(21)	0·536 (3)	0·601 (2)	0·268 (2)	4·1 (6)
O(21)	0·493 (3)	0·629 (2)	0·324 (1)	6·0 (6)
C(22)	0·653 (4)	0·492 (3)	0·084 (2)	4·8 (7)
O(22)	0·681 (3)	0·447 (2)	0·035 (2)	8·4 (8)
C(23)	0·805 (4)	0·546 (3)	0·181 (2)	4·8 (7)
O(23)	0·918 (3)	0·541 (2)	0·181 (2)	7·3 (7)
C(24)	0·393 (4)	0·556 (3)	0·153 (2)	5·6 (8)
O(24)	0·298 (3)	0·538 (2)	0·140 (1)	6·1 (6)
P(2)	0·5538 (8)	0·7380 (6)	0·1056 (5)	3·0 (2)
C(211)	0·596 (2)	0·833 (1)	0·155 (1)	4·4 (7)
C(213)	0·734 (2)	0·872 (1)	0·245 (1)	5·7 (8)
C(215)	0·568 (2)	1·012 (1)	0·170 (1)	8(1)
C(221)	0·368 (3)	0·796 (2)	0·088 (1)	4·0 (6)
C(223)	0·172 (3)	0·854 (2)	0·005 (1)	12(2)
C(225)	0·135 (3)	0·855 (2)	0·139 (1)	7(1)
C(231)	0·646 (3)	0·762 (2)	0·014 (1)	4·5 (7)
C(233)	0·809 (3)	0·844 (2)	−0·068 (1)	9(1)
C(235)	0·716 (3)	0·713 (2)	−0·107 (1)	7(1)

The coordination polyhedron around tin(IV) is a distorted tetrahedron in which the bond angles Re(1)—Sn—Re(2) and Br(1)—Sn—Br(2) differ significantly from the ideal angles. The enlargement of Re(1)—Sn—Re(2) and the reduction of Br(1)—Sn—Br(2) is comparable with observations for the compound $[SnBr_2\{Mn(CO)_5\}_2]$ [Mn—Sn—Mn 125·88 (9)°, Br—Sn—Br 98·44 (8)° (Preut, Wolfes & Haupt, 1975)] and other similar tin(IV)–transition-metal carbonyls or derivatives (Zuckerman & Ho, 1973).

In the series of compounds $[SnBr_2\{Mn(CO)_5\}_2]$, $[SnBr_2\{Re(C_6H_5)_3P(CO)_4\}_2]$ the angle Br—Sn—Br decreases for the homologous transition metals by about 3·3 (1)°. The corresponding increase of *p* character in the Sn^{IV}—Br bonds should mainly be caused by the different ligands: the π-acceptor ability of a CO group is stronger than that of $(C_6H_5)_3P$ in the *trans* position. The Sn—Br bond distances in the title compound are the longest found to date in such Sn^{IV} compounds.

The Sn—Re bond lengths in the title compound agree with the value (Sn—Re 2·74 Å) in $(C_6H_5)_3SnRe(CO)_5$ (Struchkov, Anisimov, Osipova, Kolobova & Nesmeyanov, 1967). Intermolecular distances do not indicate interactions exceeding van der Waals forces.

References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

Preut, H., Wolfes, W. & Haupt, H.-J. (1975). *Z. Anorg. Allg. Chem.* **412**, 121–128.

Sheldrick, G. M. (1976). *SHELX*. A program for crystal structure determination. Univ. of Cambridge, England.

Struchkov, Yu. T., Anisimov, K. N., Osipova, O. P., Kolobova, N. E. & Nesmeyanov, A. N. (1967). *Dokl. Akad. Nauk SSSR*, **172**, 107–111.

Waal, B. W. van de (1976). *POP*1. *Plot-of-Packing Program*. Twente Univ. of Technology, Enschede, The Netherlands.

Zuckerman, J. J. & Ho, B. Y. K. (1973). *J. Organomet. Chem.* **49**, 1–84.

Table 2. *Bond lengths* (Å) *and angles* (°)

	$i = 1$	$i = 2$		$i = 1$	$i = 2$		$i = 1$	$i = 2$
Sn(1)—Br(*i*)	2·565 (4)	2·584 (4)	Br(1)—Sn(1)—Br(2)	95·1 (1)		C(*i*3)—Re(*i*)—C(*i*4)	91 (1)	172 (2)
Sn(1)—Re(*i*)	2·741 (2)	2·730 (2)	Br(1)—Sn(1)—Re(*i*)	105·6 (1)	112·9 (1)	C(*i*4)—Re(*i*)—C(*i*1)	87 (1)	90 (2)
Re(*i*)—C(*i*1)	1·96 (3)	1·93 (3)	Br(2)—Sn(1)—Re(*i*)	105·6 (1)	108·7 (1)	C(*i*1)—Re(*i*)—C(*i*3)	169 (1)	98 (1)
Re(*i*)—C(*i*2)	1·96 (3)	1·95 (4)	Re(1)—Sn(1)—Re(2)	124·8 (1)		C(*i*2)—Re(*i*)—C(*i*4)	173 (1)	87 (2)
Re(*i*)—C(*i*3)	2·00 (3)	2·03 (4)	Sn(1)—Re(*i*)—C(*i*1)	86·2 (8)	82·7 (8)	Sn(1)—Re(*i*)—P(*i*)	174·7 (2)	171·5 (2)
Re(*i*)—C(*i*4)	1·99 (3)	2·09 (4)	Sn(1)—Re(*i*)—C(*i*2)	88·1 (9)	89·7 (9)	C(*i*1)—Re(*i*)—P(*i*)	99·1 (8)	90·8 (9)
C(*i*1)—O(*i*1)	1·16 (4)	1·18 (4)	Sn(1)—Re(*i*)—C(*i*3)	82·8 (8)	81·8 (9)	C(*i*2)—Re(*i*)—P(*i*)	91·4 (9)	97·2 (10)
C(*i*2)—O(*i*2)	1·16 (4)	1·16 (5)	Sn(1)—Re(*i*)—C(*i*4)	86·6 (8)	96·8 (10)	C(*i*3)—Re(*i*)—P(*i*)	91·9 (8)	93·5 (9)
C(*i*3)—O(*i*3)	1·12 (4)	1·11 (5)	Re(*i*)—C(*i*1)—O(*i*1)	174 (3)	177 (3)	C(*i*4)—Re(*i*)—P(*i*)	94·3 (8)	88·7 (10)
C(*i*4)—O(*i*4)	1·13 (4)	1·09 (5)	Re(*i*)—C(*i*2)—O(*i*2)	175 (2)	174 (3)	Re(*i*)—P(*i*)—C(*i*11)	114·6 (7)	116·4 (7)
Re(*i*)—P(*i*)	2·421 (7)	2·421 (7)	Re(*i*)—C(*i*3)—O(*i*3)	177 (2)	176 (3)	Re(*i*)—P(*i*)—C(*i*21)	115·0 (7)	113·1 (10)
P(*i*)—C(*i*11)	1·84 (2)	1·85 (3)	Re(*i*)—C(*i*4)—O(*i*4)	175 (2)	167 (3)	Re(*i*)—P(*i*)—C(*i*31)	119·3 (5)	115·7 (8)
P(*i*)—C(*i*21)	1·84 (2)	1·84 (2)	C(*i*1)—Re(*i*)—C(*i*2)	89 (1)	171 (1)	C(*i*11)—P(*i*)—C(*i*21)	102·9 (7)	101·0 (12)
P(*i*)—C(*i*31)	1·82 (2)	1·82 (2)	C(*i*2)—Re(*i*)—C(*i*3)	92 (1)	86 (2)	C(*i*11)—P(*i*)—C(*i*31)	103·5 (10)	102·9 (12)
						C(*i*21)—P(*i*)—C(*i*31)	99·1 (9)	106·1 (11)