A Novel Redistribution Reaction of Compounds Containing a Tin–Molybdenum Bond

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Summary $(C_5H_5)Mo(CO)_2(PPh_3)SnMe_3$ is converted quantitatively into $[(C_5H_5)Mo(CO)_2(PPh_3)]_2SnMe_2$ (I) by heating at 200°, with the elimination of $SnMe_4$.

REDISTRIBUTION reactions (*e.g.* equation 1) are well-known for main-group organometallic compounds[1] but less well documented for transition-metal organometallic compounds. I report the first example of a redistribution reaction involving a metal–metal bonded compound containing hetero-metal atoms [equation 2, where L = CO, PPh_3, $PPhMe_2$, $P(OPh)_3$, $P(OCH_2)_3CMe$, $AsPh_3$, $SbPh_3$].

$$SnMe_4 + SnCl_4 \rightarrow Me_3SnCl + MeSnCl_3 \qquad (1)$$

$$2(C_5H_5)Mo(CO)_2(L)SnMe_3 \xrightarrow{200^\circ} [(C_5H_5)Mo(CO)_2(L)]_2SnMe_2 + SnMe_4 \qquad (2)$$

The new compounds *trans*-$(C_5H_5)Mo(CO)_2(L)SnMe_3$ [where L = PPh_3, $PPhMe_2$, $P(OPh)_3$, $P(OCH_2)_3CMe$, $AsPh_3$, $SbPh_3$] were prepared either by reaction of the ligand with the unsubstituted precursor $(C_5H_5)Mo(CO)_3$-$SnMe_3$ in refluxing diglyme or by treating the sodium salt of $[(C_5H_5)Mo(CO)_2(L)]^-$ with Me_3SnCl in tetrahydrofuran at room temperature. The former method was successful only for L = PPh_3 while all other derivatives were prepared by the latter method. The stereochemistry was inferred from their i.r. and ^1H n.m.r. spectra (see Table) by comparison with similar compounds.[2]

TABLE I

I.r. spectra (2000—1700 cm^{-1}) *and* ^1H *n.m.r. spectra* (C_5H_5)

Compound $(C_5H_5)Mo(CO)_2(L)SnMe_3$ L	$\nu(CO)^a$			$\tau^b C_5H_5[J^c(P\text{–}C_3H_5)]$
PPh_3	1901(0·55)	1833(1·0)		5·21(1·0)
$PPhMe_2$	1894(0·54)	1825(1·0)		5·2(1·0)
$P(OPh)_3$	1918(0·52)	1853(1·0)		5·42(0·8)
$P(OCH_2)_3CMe$	1929(0·56)	1862(1·0)		4·95(0·8)
$AsPh_3$	1902(0·53)	1835(1·0)		5·13
$SbPh_3$	1902(0·46)	1837(1·0)		5·02
$[(C_5H_5)Mo(CO)_2(L)]_2SnMe_2$ **L**				
PPh_3	1896(0·44)	1844(0·25)	1820(1·0)[d]	4·95(0·8)[e]
$P(OCH_2)_3CMe$	1918	1898	1834[f]	

[a] CCl_4 solution unless stated otherwise; cm^{-1} (relative peak height). [b] Relative to $SiMe_4$. $CDCl_3$ solution unless stated otherwise. [c] Hz. [d] CH_2Cl_2 solution. [e] 2 : 1 $CCl_2CCl_2 : CH_2Cl_2$ mixture. [f] Nujol mull.

Compound (I) was prepared either by heating $(C_5H_5)Mo$-$(CO)_2(PPh_3)$ $SnMe_3$ at 200° for 5 h or by heating PPh_3 and $(C_5H_5)Mo(CO)_3SnMe_3$ together at 200° in the absence of solvent. Tetramethyltin was isolated and identified by its mass spectrum. Compound (I) was synthesised independently from $[(C_5H_5)Mo(CO)_3]_2SnMe_2$ and Ph_3P.

Attempts to convert the other trimethyltin derivatives into dimethyltin derivatives at about 200° proved successful only for L = $P(OCH_2)_3CMe$. However, in all cases, even when only decomposition products resulted, tetramethyltin was always liberated, suggesting that the dimethyltin derivative is formed by elimination of tetramethyltin but that the resulting dimethyltin derivative decomposes spontaneously at the temperature of redistribution.

The mechanism of redistribution can best be rationalised in terms of a bimolecular process proceeding through a four-centre activated complex. The absence of products such as hexamethylditin argues against a random bond-making, bond-breaking process. In the mass spectrum of each of the trimethyltin derivatives by far the most abundant tin-containing moiety occurs at $M - 15$, where M is the parent ion, due to loss of one methyl group.

All the ligand derivatives $(C_5H_5)Mo(CO)_2(L)SnMe_3$ are more stable to air than $(C_5H_5)Mo(CO)_3SnMe_3$ except when L = $P(OPh)_3$ which is less stable. $[(C_5H_5)Mo(CO)_2$-$(PPh_3)]_2SnMe_2$ is unaffected by air for at least 1 year whereas $[(C_5H_5)Mo(CO)_3]_2SnMe_2$ decomposed rapidly in air.

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