

Table 1. Analytical data for the complexes  $[\text{Re}(\text{CO})_3\text{L}][\text{Re}_2(\text{CO})_6(\mu\text{-X})_3]$  ( $\text{X} = \text{Cl}, \text{Br}$  or  $\text{I}$ ) and  $[\text{Re}(\text{CO})_3\text{L}][\text{SbF}_6]$ .

Complex	Colour	$\nu(\text{CO})^{\text{a}}/\text{cm}^{-1}$		$m/z^{+\text{b}}$	$m/z^{-\text{c}}$	Analyses <sup>d</sup>		
		cation	anion <sup>e</sup>			C	H	N
$[\text{Re}(\text{CO})_3\text{L}][\text{Re}_2(\text{CO})_6(\mu\text{-Cl})_3]$	Orange	2036 1949 1920	2024 (2030) ~1920 (1917)	782	645	31.94 (31.94)	1.38 (1.62)	0.91 (0.98)
$[\text{Re}(\text{CO})_3\text{L}][\text{Re}_2(\text{CO})_6(\mu\text{-Br})_3]$	Orange	2036 1949 1920	2023 (2028) 1910sh (1915)	782	779	29.27 (29.22)	1.38 (1.48)	0.84 (0.90)
$[\text{Re}(\text{CO})_3\text{L}][\text{Re}_2(\text{CO})_6(\mu\text{-I})_3]$	Orange	2036 1949 1920	2018 (2012) 1905sh (1914)	782	920	26.96 (26.80)	1.19 (1.36)	0.77 (0.82)
$[\text{Re}(\text{CO})_3\text{L}][\text{SbF}_6]$	Yellow	2036 1949 1920		782	234	36.88 (37.77)	2.05 (2.28)	1.36 (1.38)

<sup>a</sup> Infrared data; spectra recorded in  $\text{CH}_2\text{Cl}_2$  solution.

<sup>b</sup> FAB mass spectral data (cation).

<sup>c</sup> Negative ions mass spectral data.

<sup>d</sup> Calculated values in parentheses.

<sup>e</sup> Literature value given in parentheses (see reference 14); sh = shoulder.

Table 2. Phosphorus-31 NMR data<sup>a</sup> for L and the complexes [Re(CO)<sub>3</sub>L][Re<sub>2</sub>(CO)<sub>6</sub>(μ-X)<sub>3</sub>] (X = Cl, Br or I) and [Re(CO)<sub>3</sub>L][SbF<sub>6</sub>].

Compound	δ <sup>b</sup>	δ <sub>iso</sub>	δ <sub>11</sub>	δ <sub>22</sub>	δ <sub>33</sub>	Δσ	η
Ligand	38.4 (37.7) <sup>c</sup>	41	102	98	-77	177	0.03
		38	98	98	-82	180	0.00
[Re(CO) <sub>3</sub> L][Re <sub>2</sub> (CO) <sub>6</sub> (μ-Cl) <sub>3</sub> ]	67.0	65	143	86	-35	150	0.57
		62	134	102	-49	167	0.29
[Re(CO) <sub>3</sub> L][Re <sub>2</sub> (CO) <sub>6</sub> (μ-Br) <sub>3</sub> ]	67.1	65	144	86	-35	150	0.59
		62	131	106	-51	169	0.22
[Re(CO) <sub>3</sub> L][Re <sub>2</sub> (CO) <sub>6</sub> (μ-I) <sub>3</sub> ]	66.8	65	141	84	-29	142	0.61
		62	133	104	-50	168	0.26
[Re(CO) <sub>3</sub> L][SbF <sub>6</sub> ]	66.8	71	145	84	-18	133	0.69
		66	145	79	-27	139	0.71

<sup>a</sup> Ambient temperature (298 K) solid-state NMR data except for <sup>b</sup>; CSA tensors assigned according to the Haebleren convention (reference 12).

<sup>b</sup> Ambient temperature (298 K) solution NMR data; spectra recorded in CDCl<sub>3</sub> or CD<sub>2</sub>Cl<sub>2</sub> (see text).

<sup>c</sup> Literature value give in parentheses [see reference 10 (solvent not reported)].

Table 3. Hydrogen-1 NMR data<sup>a</sup> for Land the cation,  $[\text{Re}(\text{CO})_3\text{L}]^+$ .

Compound <sup>b</sup>	$\delta(\text{H}_A/\text{H}_{A'})$	$\delta(\text{H}_B/\text{H}_{B'})$	$\delta(\text{H}_C)$	$\delta(\text{H}_D/\text{H}_{D'})$	$\delta(\text{H}_E/\text{H}_{E'})$	$\delta(\text{H}_F)$	$\delta(\text{H}_K/\text{H}_{K'})$	$\delta(\text{H}_J)$
Ligand	7.60 (7.5 <sup>c</sup> , 0.8 <sup>c</sup> 13.3 <sup>d</sup> )	7.30 (7.5 <sup>c</sup> , 2.8 <sup>d</sup> )	7.50 (7.5 <sup>c</sup> , 0.8 <sup>c</sup> , 1.2 <sup>d</sup> )				8.71 (7.8 <sup>c</sup> , 14.1 <sup>d</sup> )	8.07 (7.8 <sup>c</sup> , 3.8 <sup>d</sup> )
$[\text{Re}(\text{CO})_3\text{L}]^+ \text{A}^-$	8.07 (7.6 <sup>c</sup> , 0.8 <sup>c</sup> , 15.1 <sup>d</sup> )	7.79 (7.6 <sup>c</sup> , 3.6 <sup>d</sup> )	7.91 (7.6 <sup>c</sup> , 0.8 <sup>c</sup> , 1.9 <sup>d</sup> )	7.50 (7.4 <sup>c</sup> , 0.8 <sup>c</sup> , 14.1 <sup>d</sup> )	<sup>e</sup>	7.76 (7.4 <sup>c</sup> , 0.8 <sup>c</sup> , 1.8 <sup>d</sup> )	<sup>e</sup>	8.16 (7.7 <sup>c</sup> , 3.7 <sup>d</sup> )

<sup>a</sup> Data recorded at 298 K; <sup>1</sup>H chemical shifts quoted relative to tetramethylsilane; spectra of the complexes were recorded in  $\text{CH}_2\text{Cl}_2$ ; spectrum of the ligand recorded in  $\text{CDCl}_3$ ; see Fig. 1 for labelling.

<sup>b</sup>  $\text{A}^- = [\text{Re}_2(\text{CO})_6(\mu\text{-X})_3]^-$  (X = Cl, Br or I) or  $[\text{SbF}_6]^-$ .

<sup>c</sup> <sup>n</sup> $J_{\text{HH}}/\text{Hz}$ .

<sup>d</sup> <sup>n</sup> $J_{\text{PH}}/\text{Hz}$ .

<sup>e</sup>  $\text{H}_E/\text{H}_{E'}$  and  $\text{H}_K/\text{H}_{K'}$  overlap, giving a complex multiplet centred at *ca.* 7.6 ppm.