A Mixed Manganese and Chromium Carbonyl Complex of 2-Benzylpyrrole

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Summary A tricarbonylmanganesetricarbonylchromium complex of 2-benzylpyrrole and the separate tricarbonylmetal complexes have been prepared and characterized.

WE report the preparation and characterization of 2-(π-benzylchromium tricarbonyl)-π-pyrrolylmanganese tricarbonyl which we believe to be the first reported compound in which two different transition metals are π-bonded to two different aromatic rings of the same ligand molecule. We have also prepared and characterized the two compounds in which only one of the aromatic rings of 2-benzylpyrrole is complexed with a metal tricarbonyl group. These are 2-benzyl-π-pyrrolylmanganese tricarbonyl and 2-(π-benzylchromium tricarbonyl)pyrrole.

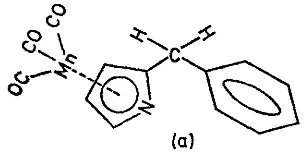
2-Benzyl-π-pyrrolylmanganese tricarbonyl was prepared by heating manganese pentacarbonyl bromide under reflux with the potassium salt of 2-benzylpyrrole in tetrahydrofuran. The resulting material was chromatographed on silica gel and the isolated complex recrystallized from petroleum (60—80°) to give light orange crystals (m.p. 72—73°). 2-(π-Benzylchromium tricarbonyl)pyrrole was prepared by allowing chromium hexacarbonyl and 2-benzylpyrrole to react in refluxing solvent. The solvents used were di-n-butyl ether, n-octane and hexane–decane mixtures with di-n-butyl ether giving the best yield. The resulting material was again chromatographed on silica gel and the isolated complex recrystallized from petroleum (60—80°) to give pale yellow crystals (m.p. 94—95°). Finally chromium hexacarbonyl, dimanganese decacarbonyl, and 2-benzylpyrrole were allowed to react in refluxing n-octane. The mixture was again chromatographed on silica gel and, apart from unreacted starting materials, three compounds were isolated and each was recrystallized from petroleum (60—80°). These were (I) 2-benzyl-π-pyrrolylmanganese tricarbonyl, (II) 2-(π-benzylchromium tricarbonyl)pyrrole, and a third compound (III) which was bright yellow in colour and melted at 103—104°. This compound proved to be 2-(π-benzylchromium tricarbonyl)-π-pyrrolylmanganese tricarbonyl. All these reactions were carried out in a nitrogen atmosphere. I.r. and mass-spectral data on these compounds are given in the Table.

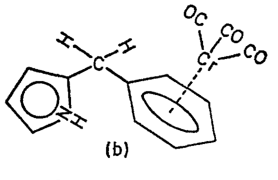
TABLE

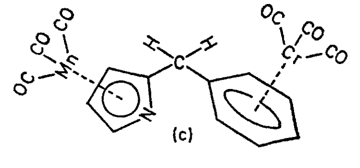
	I.r. cm^{-1} (CHCl$_3$)			Mass spectra m/e	
	CO stretch	N–H stretch	$M^{+\cdot}$	$M^{+\cdot} - n$(CO)	(Metal)$^+$
2-Benzylpyrrole	none	3471	157	none	none
(I)	2042, 1948	none	295	267, 239, 211	55
(II)	1968, 1888	3470	293	265, 237, 209	52
(III)	2044, 1969, 1950(sh), 1895	none	431	403, 375, 347	55
				319, 291, 263	52

For compound (I), the CO stretching vibrations are typical of the π-pyrrolylmanganese tricarbonyl complexes. For example, they occur at 2062 and 1980 cm^{-1} in π-pyrrolylmanganese tricarbonyl.[1] The absence of the N–H stretch shows that a pyrrolyl group is involved here. Furthermore, the mass-spectral data show a parent ion value in agreement with the formula suggested and the successive loss of three CO groups from the parent ion is typical of this type of compound. The suggested structure for this compound is shown as *a*.

Similar arguments lead us to structure *b* for compound (II). The CO stretching vibrations of this compound are typical of tricarbonyl-π-arenechromiums. For example in tricarbonyl-π-benzenechromium the bands at 1977 and 1910 cm^{-1} are assigned to CO stretching vibrations.[2] Furthermore, the parent ion peak for compound (II) is in agreement with the formula shown in *b* and the successive loss of three CO groups is observed. The presence of the N–H stretch in this compound shows that it contains a pyrrole ring and not a pyrrolyl system as in the manganese complex.


(a)


(b)


(c)

The CO stretching absorptions in compound (III) are almost a superposition of those of compounds (I) and (II), but the shift in the lowest band with respect to the chromium complex shows that this is not simply a mixture. The m.p. is also different from, and higher than, those of either of the single metal compounds. The mass-spectral data confirm the formula shown as *c*, and show the correct parent ion for our formulation together with ions which represent successive loss of six CO groups.

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[2] D. A. Brown and H. Sloan, *J. Chem. Soc.*, 1962, 3849.