

SHORT COMMUNICATION

THE FORMATION OF TRIFLUOROACETYLIRON TETRACARBONYL IODIDE

H.G.ANG and (Mrs) D.CHEONG

Chemistry Department, University of Singapore, Bukit Timah Road, Singapore 10, Republic of Singapore

Trifluoroacetic anhydride has been found to react with sodium salts of various substituted metal carbonyls of Cr, Mo, W, Mn, Fe and Co to afford the corresponding trifluoroacetyl derivatives [1-3]. These reactions proceed along the same course as those with perfluoroacyl fluorides and chlorides [2,4-6]. On the other hand, reactions of trifluoroacetyl halides with iron pentacarbonyl have failed to yield the trifluoroacetyl derivatives. Instead the chloride is converted to the fluoride together with a small amount of carbonyl fluoride; and its reactions with trifluoroacetyl fluoride at elevated temperatures yield perfluoroethane and carbonyl fluoride [7].

We now report that on slow addition of iodine into a mixture of iron pentacarbonyl and trifluoroacetic anhydride at room temperature carbon monoxide is released almost instantaneously, and an orange-brown solid which can be formulated as $\text{CF}_3\text{COFe}(\text{CO})_4\text{I}$ (I) precipitates. It is stable in vacuum at room temperature, but decomposes slowly in air. It melts with decomposition at 105°C . In solution it undergoes decomposition if air is not completely excluded.

The infrared spectrum of the complex (I) in chloroform gives three distinct strong peaks located at 2140, 2080 and 2060 cm^{-1} , which are due to the C-O stretching vibrations. The positions and intensities of these bands are comparable to those reported for $\text{C}_3\text{F}_7\text{Fe}(\text{CO})_4\text{I}$, which are located

at 2145(m), 2111(vw), 2088(vs) and 2054(m) cm^{-1} [6]. The absence of the fourth peak is presumably because it is degenerate with its adjacent peak. The data suggest that the iodine atom and the trifluoroacetyl group are in cis positions in the octahedral complex. The presence of the trifluoroacetyl group is confirmed by the peak located at 1680 cm^{-1} due to the carbonyl stretching frequency, and two strong peaks at 1200 and 1150 cm^{-1} which are characteristic of the C-F stretching frequency.

The complex (I) provides the first example of a metal carbonyl complex containing both iodine and a trifluoroacetyl group. The synthetic route is analogous to that leading to the formation of trifluoromethyliron tetracarbonyl iodide [6,8].

EXPERIMENTAL

The i.r. spectrum was recorded on a Perkin Elmer 337 using potassium bromide optics. All manipulations were carried out in dry apparatus using dry nitrogen.

Reaction of iodine with iron pentacarbonyl in trifluoroacetic anhydride

Iodine (1.04 g., 4.09 mmole) was introduced into a mixture of trifluoroacetic anhydride (40.0 g., 19.0 mmole) and iron pentacarbonyl (1.2 g., 4.1 mmole). Slow effervescence occurred and the solution turned dark brown. The solution was allowed to continue overnight under nitrogen. On filtration, an orange-brown solid (1.2 g.) was obtained. It melted at 105°C with decomposition, and analysis gave : I, 33.9, Fe, 14.1 %; and calculated for $\text{C}_6\text{F}_3\text{O}_5\text{FeI}$: I, 32.4, Fe, 14.3 % .

Attempts to recrystallise the orange-brown solid from acetone and pentane resulted in some decomposition.

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