A Convenient Method for the Preparation of Red Ruthenium Carbonyl Chloride Solution*

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The 'red ruthenium carbonyl chloride solution' [1] is the starting material for the preparation of several ruthenium(II) carbonyl compounds such as Ru(1,10-phenanthroline)$(CO)_2Cl_2$, Ru(bipyridyl)-$(CO)_2Cl_2$, $Ru(PPh_3)_2(CO)_2Cl_2$, *etc.* [2–4]. The procedure mentioned in the literature for the initial carbonylation reaction involves bubbling of pure carbon monoxide gas for a period of 6–10 hr at atmospheric pressure.

During our synthetic studies with carbon monoxide at atmospheric pressure, we used the adsorption of carbon monoxide on coconut shell charcoal for storing small quantities of pure carbon monoxide gas. Active coconut shell charcoal adsorbs 4–5% by weight of carbon monoxide at atmospheric pressure and this adsorbed gas, in certain cases, is readily given out to form carbonyl complexes. This phenomenon has been utilized here for the rapid preparation of red ruthenium carbonyl solutions. The following simple experiment will illustrate the method.

Active coconut shell charcoal was specially made by the low temperature carbonisation of ripe coconut shells kept out of contact with air (500 °C). The charcoal thus obtained was washed with dilute hydrochloric acid (1:1 v/v) to remove any carbonate, kept in boiling water till free of chloride, and dried in an oven (150 °C) overnight. The product was crushed and coarse particles (30 to 100 mesh) were stored in air tight containers. Yield of the charcoal is roughly 20% based upon the weight of the dry raw shells taken.

Before subjecting to carbon monoxide, the charcoal powder (25 g; about 40 ml) was kept under vacuum at 200 °C for 15 minutes. It was then cooled, vacuum broken with dry carbon monoxide and kept for another 10 minutes in a carbon monoxide atmosphere. The adsorption of carbon monoxide caused an increase in weight of 4% (1 g). The carbon monoxide adsorbed on charcoal was now ready for use for carbonylation reactions. For making the red carbonyl chloride solution, ruthenium trichloride trihydrate (1.024 g; 4 mmol) was dissolved in 250 ml aldehyde-free ethanol, kept in contact with the above charcoal and warmed up to boiling and allowed to remain for 30 min., preferably in a slow current of carbon monoxide gas. The solution was then filtered, charcoal washed with fresh ethanol till colourless, and used for making ruthenium complexes with donor molecules. Physical examination showed that most of the adsorbed carbon monoxide was given out. The spent charcoal could be re-used after boiling with water, drying, and activation under vacuum.

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