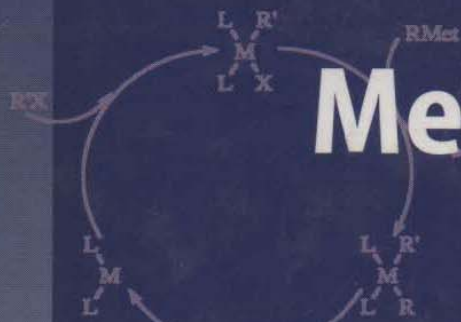


L. Brandsma  
S. F. Vasilevsky  
H. D. Verkruijsse

# Application of Transition Metal Catalysts in Organic Synthesis



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Although several transition metal catalysts are commercially available, one may prefer to make them oneself if larger quantities are needed. The procedures described in this chapter are taken from the literature, but in some of them modifications have been introduced in order to facilitate their performance.

## 1.1 Catalysts

### 1.1.1 Copper Halides

Copper(I) chloride and the corresponding bromide and iodide ( $\text{CuX}$  or  $\text{Cu}_2\text{X}_2$ ) are almost colorless compounds. Molecular weights for  $\text{CuX}$  are 98.9, 143.4 and 190.4, respectively. Due to oxidation a light-green or - in the case of  $\text{CuI}$  - light-brown colour appears during storage, but the small traces of  $\text{Cu(II)}$  present in most cases do not affect the intended result of a reaction in which the salts are used in catalytic amounts. The preparations of  $\text{CuBr}$  and  $\text{CuCl}$  are described in Vogel's *Textbook of Practical Organic Chemistry*, 5<sup>th</sup> ed., Langstein, London (1994) p. 428 and by G. S. Hammett and S. M. Mc Elroy in *Org. Synth., Coll. Vol. 1* (1941), 170, respectively.

For some reactions the use of the complex  $\text{Cu}(\text{DMS})_2$  (molecular weight 286.3) is recommended (e.g. by H. O. House, C.-W. Cho, J. M. Williams and M. J. Umen, *J. Org. Chem.* (1975) 40, 4160). Catalytic reactions are sometimes carried out in the presence of additional amounts of dimethyl sulfoxide, which serve to increase the solubility of the intermediary complex. A serious disadvantage is the stench of the sulfide liberated during the work-up.

#### 1.1.1.1 Solubilization of Copper(I) Halides

Copper(I) halides can be solubilized by shaking the powder with a solution of an excess of anhydrous lithium bromide in tetrahydrofuran. In this way (concentrated) solutions of the cuprous  $\text{LiCuXBr}$  can be prepared. These may have a rather dark green or brown colour, caused by the presence of small amounts of  $\text{Cu(II)}$ . The advantage of using these solubilized copper halides over solutions of the powder in a catalytic reaction with an organometallic reagent is that the catalyst is quickly and homogeneously distributed.