## (COT)Fe(CO)<sub>3</sub> — Another Ring Whizzer

## Overview

In this project, you will convert cyclooctadiene to cycloctatetraene, prepare an iron complex from the tetraene, and obtain NMR spectra of the complex.

Although initial data suggested that all eight ring carbons in cyclooctadienetricarbonyliron (1) are bound to the metal, subsequent results showed that this is not the case. (Based on your knowledge of electron counting, how many carbons would you expect to be coordinated? Is it reasonable to expect all eight to coordinate?) Indeed, Cotton demonstrated that the cyclooctatetraene (COT) ring whizzes atop the iron at a rate that can be observed by NMR: at room temperature, a single carbon signal is observed for the COT ligand, but at -140 °C, four signals are observed.

The iron complex is readily prepared from COT and Fe(CO)<sub>5</sub>. COT, unfortunately, is quite expensive. The literature suggests, however, that it can easily be prepared from the diene: a fast search on ChemFinder turned up the three references listed below. The first two proceed by way of the aromatic COT dianion, and the third uses standard elimination chemistry. You are certainly welcome to search more thoroughly.

$$\mathsf{COD} \longrightarrow \mathsf{COT} \xrightarrow{\mathsf{Fe}(\mathsf{CO})_5} (\eta^{\mathsf{x}}\text{-}\mathsf{COT})\mathsf{Fe}(\mathsf{CO})_3$$

where

"COD" = 1,5-cyclooctadiene,

"COT" = 1,3,5,7-cyclooctatetraene,

" $\eta^x$  indicates that x of the 8 carbons are attached to the metal.

## References

With the exception of the Meier article, the references below may be obtained through the library's web-page. As the Meier article is on microfilm at the Library, it has been printed, scanned, and included in this directory.

Note that most of these preparations are on a very large scale. You should thus cut them back, to produce perhaps 2-3 g of COT and 1 g of the complex.

COT Preparation

Majumder, S; Odom, A.L. Tetrahedron Lett. 2008, 49, 1771-1772.

Gottfriedsen, J.; Miloslavina, A.; Edelmann, F.T. Tetrahedron Lett. 2004, 45, 3583-3584.

Detert, H.; Rose, B.; Mayer, W.; Meier, H. Chem. Ber. 1994, 127(8), 1529-1532.

(COT)Fe(CO)<sub>3</sub> Preparation

Manuel, T.A.; Stone, F.G.A. J. Am. Chem. Soc. 1960, 82, 366-372.

<sup>&</sup>lt;sup>1</sup> Manuel, T.A.; Stone, F.G.A. J. Am. Chem. Soc. 1960, 82, 366-372.

<sup>&</sup>lt;sup>2</sup> Dickens, B.; Lipscomb, W.N. J. Chem. Phys. 1962, 37, 2084-2093.

<sup>&</sup>lt;sup>3</sup> Cotton, F.A.; Hunter, D.L. J. Am. Chem. Soc. 1975, 98, 1413-1417.