

## 8.1: Part A- Synthesis of Wilkinson's Catalyst

### Safety Precautions

1. All parts of this experiment must be performed in the fume hood, except the gas chromatography.
2. Take care when adding solids to hot liquids; the liquids may "bump" (boil over violently).

### Note

1. Solutions or solids containing rhodium should not be discarded. They should be put into the bottle in the fume hood labeled "rhodium for recycling."
2. Don't use a septum that someone else has already used. Recycling septa is not wise because the leaks caused by damaged septa could cause the loss of expensive catalyst.
3. Review the sections on air sensitive compounds in this manual before you attempt this experiment.

In a fume hood, place a magnetic stir bar and 12 ml of absolute (100%) ethanol in a 25 mL round-bottom flask fitted with a 14/20 ground glass joint. Attach the condenser to the flask and the water lines to the condenser. Heat the flask in a sand bath on a hotplate so that the ethanol reaches boiling. Turn off the heat and wait till the boiling ceases. Add 450 mg of triphenylphosphine through the condenser. Use a stir rod to push most of the phosphine down the condenser. When almost all of the phosphine is dissolved, add 75 mg (0.36 mmol) of rhodium trichloride hydrate by the same method. Flush down any solids stuck on the condenser wall with up to 4 ml of ethanol. Resume the heating and refluxing.

During the reflux time, prepare the standard filtration apparatus (see Introduction) with a Hirsch funnel. After 30 min of reflux, red crystals should be observed in the flask. Turn off the heat. Suction filter the hot solution, then wash with ether (3 x 1 mL) and leave the solid to dry on the funnel without turning off the suction.

The filtrate (the liquid which passed through the filter) may contain extra rhodium, which should be recycled. Pour this into the bottle in the fume hood labeled "rhodium for recycling."

Report the theoretical, observed, and percentage yields. Acquire the  $^1\text{H}$  and  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of the complex in  $\text{CDCl}_3$ . Warning: Chloroform may be a carcinogen. Therefore do not touch, inhale or ingest chloroform. Use chloroform in a fume hood. Wear gloves which are resistant to chloroform (see chart earlier in this manual). The Viton and Silver Shield gloves are not disposable.

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