Important: Read Before Using Rieke® Zn

Guide to Handling Rieke Zinc® in THF

Introduction

Rieke Zinc® is highly reactive and is moisture and oxygen sensitive. Therefore, it is important to exclude atmospheric moisture and oxygen from the reactions. This guide contains the bare minimum amount of knowledge required to use Rieke Zinc®. If one is unfamiliar with inert atmosphere techniques, we strongly advise consulting a definitive source¹ on the manipulation of air sensitive reagents before using this metal. Once inert atmosphere techniques are mastered, Rieke Zinc® can be successfully and easily applied to many new synthetic strategies.²,³

Inert Atmosphere Manipulations

The minimum equipment necessary for using Rieke Zinc® includes an argon or nitrogen source, (best results are obtained using high quality nitrogen or argon), a flask equipped with a sidearm to which a septum can be attached, and cannula needles. It is desireable but not necessary that a manifold with both an argon (or nitrogen) and vacuum source be used. However, a tank of nitrogen or argon with a regulator are sufficient to utilize the active zinc.

Cannula needles should be kept in an oven until needed. All equipment should be predried in an oven (125°C overnight) and removed just before use. If a manifold with inert gas and vacuum sources is available, achieving an inert atmosphere is simple. The assembled glassware is attached to the manifold and vacuumed and refilled with argon or nitrogen three times. The system will then be under a suitable inert atmosphere. If no source of vacuum is available, assemble the glassware and purge it for at least 15-20 min with inert gas. To purge the system, use a long needle attached via rubber tubing to the inert gas source, (argon or nitrogen from a cylinder equipped with a regulator adjusted at three to five psi.) pierce the septum to add inert gas to the reaction vessel. Insert another needle in the same septum to allow an outlet for the gas.

Cannula Transfer of Rieke Zinc® in THF

To perform a cannula transfer from a reagent bottle, purge the flushing needle with inert gas. The bottle should be inverted and shaken vigorously several times and pressurized by inserting the flushing needle. The cannula needle is inserted into the cap and the inert gas is allowed to flush the cannula for 1-2 min. Always make sure the cannula is stored in an oven until just prior to use. Once the cannula has been flushed with inert gas, the other end of the cannula needle can be inserted through a septum on the reaction vessel.

Two methods may be used to lower the inert gas pressure on the reaction vessel, allowing
the reagents to be transferred. One method is to use the vacuum line on the manifold to decrease the system pressure slightly. The second method is to close the inert gas source on the manifold and pierce a second needle through the septum on the reaction flask, thus reducing the gas pressure. Once the gas pressure in the reaction vessel has been slightly reduced, the cannula needle can be lowered into the reagent. This should start the flow of the Rieke Zinc® slurry into the reaction vessel. During the transfer, gently swirl the reagent bottle to allow the Rieke zinc® powder to become uniform in the THF. The recommended size of cannula for the transfer of Rieke zinc® is 14 gauge or larger. Measuring the amount of reagent in a cannula transfer is easily accomplished by reading the volume mark on the reagent bottle. After the required amount of the reagent has been transferred, pull the cannula needle above the reagent level, but do not remove it entirely. Once the reagent is no longer coming out of the cannula, remove the cannula first from the reaction flask, then from the reagent bottle, and finally remove the inert gas flushing needle and place the reaction flask under positive inert gas pressure.

**Clean-up of excess Rieke Zinc®**

Extra care must be taken when cleaning equipment containing TRACE AMOUNTS of active zinc. All operations must be performed in an efficient fume hood. Wash with isopropanol first, followed by water, and then carefully add dilute (3M) HCl. For destroying LARGE AMOUNTS of active zinc, isopropanol is added and stirred overnight followed by water, and then dilute HCl.

**Preparation of Organozinc Reagents Using Rieke Zinc® and Halides**

Most primary alkyl bromides react with Rieke Zinc® in 3-4 hrs. This can be shortened considerably by refluxing. In most cases, 1.05-1.2 equiv of Rieke Zinc® to 1.0 equiv of alkyl halide is sufficient to achieve complete conversion to the organozinc. Secondary and tertiary alkyl bromides react with Rieke Zinc® quickly at r.t. using 1.0 equiv of halide to 1.05-1.1 equiv of Rieke Zinc®. Aryl iodides are also readily converted to the organozinc reagents. Generally, 1.5 equiv of Rieke Zinc® will effect complete conversion overnight at r.t. or 1-2 h under refluxing conditions. Aryl bromides are more difficult and generally require 2-3 equiv of zinc and refluxing for 2 to 3 h. A typical reaction experimental procedure is enclosed.

**References**


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**Rieke Metals, Inc.**
**1001 Kingbird Lane**
**Lincoln NE, 68521**
**402-434-2775**