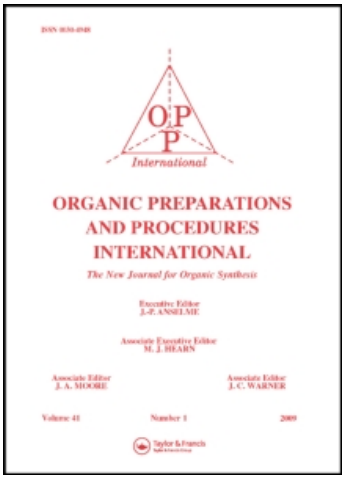

Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

A SIMPLE APPARATUS FOR THE SAFE HANDLING OF PALLADIUM BLACK CATALYST

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To cite this Article Jimenez, Manuel H.(1978) 'A SIMPLE APPARATUS FOR THE SAFE HANDLING OF PALLADIUM BLACK CATALYST', Organic Preparations and Procedures International, 10: 6, 295 — 297
To link to this Article: DOI: 10.1080/00304947809355055
URL: http://dx.doi.org/10.1080/00304947809355055

Anal. Calcd. for $C_6H_5N_3$: C, 60.49; H, 4.23; N, 35.28.

Found: C, 60.45; H, 4.34; N, 35.00.

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2. The chloroform insoluble compound is 3-amino-6-chloropyridazine (3.1 g).

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A SIMPLE APPARATUS FOR THE SAFE HANDLING OF PALLADIUM BLACK CATALYST

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A simple and inexpensive device made from readily available laboratory materials is described here for the safe handling of palladium black.[1,2] The device allows thorough rinsing of the catalyst (once generated from palladium chloride) with water, any suitable solvent, removal of most of the solvent and safe transfer of the catalyst to the reaction mixture with little or no danger of ignition or of moisture absorption.

This "syringe filter" device (Fig. 1) consists simply of a 10 milliliter Plastipak syringe[3] (Becton-Dickinson and Co., Rutherford, NJ) with the tip cut off at the end of the cylindrical barrel and of a porous polypropylene disc[4] forced-fitted in its place.

The syringe filter method for handling palladium black catalyst has been shown to be much safer than conventional manipulation. Numerous experiments using the syring filter have been performed in our laboratory

without incidences of fires, a potential hazard when working with palladium black.[6] We have also demonstrated that palladium black handled with the syringe filter is of superior quality to that generated by the conventional procedure, since it contains only trace amounts of solvent and can be transferred to the reaction vessel with little or no moisture absorption. The syringe filter has also been used for the safe handling of other dangerous catalysts including Raney Nickel.

EXPERIMENTAL

Generation and Handling of Palladium Black.[5]- A 5 ml portion of a $PdCl_2$ stock solution prepared by dissolving 1 g of $PdCl_2$ for every 5 ml of 2 N HCl (heating required), was added to 50 ml of boiling water contained in a 400 ml beaker. The mixture was brought to a boil and treated with 0.20 ml of 97-100% formic acid. While still boiling, the pH was adjusted to 7.5-8.0 by the addition of approximately 16 ml of 10% KOH. The pH of the boiling mixture was then brought back to 6-7 by the addition of formic acid. Boiling was continued for 5 minutes.

The palladium black was then transferred to the assembled syringe filter that has been fitted onto a 250 ml filter flask by means of a rubber tapered filter adapter (Fig. 2). With mild suction, the catalyst was washed thoroughly (6 to 7 times) with distilled water. Water was then removed from the catalyst by 6 or 7 successive washings with methanol. [CAUTION: Do not allow the catalyst to go dry]. After the final rinse, the plunger was reinserted into the syringe barrel, the end of the syringe filter placed against a rigid absorbant surface and the excess methanol squeezed out. Quick withdrawal of the plunger (approximately one inch) causes the catalyst "pellet" to be separated from the filter disc. This filter was then pried out cleanly with a sharp object (the disc can be cleaned for re-

use). The palladium black pellet was then ready for introduction into the reaction mixture.

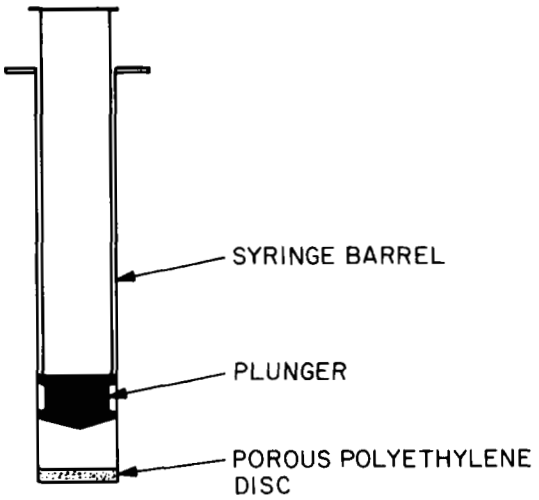

Fig. 1 Syringe Filter

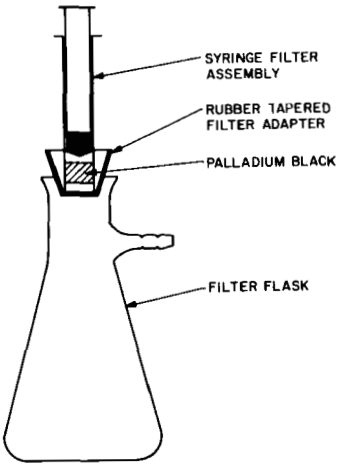

Fig. 2 Syringe Filtration Assembly

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1. H. Willstätter and E. Waldschmidt-Leitz, Ber., 54, 113 (1921).

2. H. Wieland, Ber., 45, 484 (1912).

3. For smaller scale reactions a 3 or 5 milliliter syringe may be used.

4. Cut from 70 micron porous polyethylene sheeting (Bel-Art Products, Pequannock, NJ). The filter disc is easily cut using a cork borer and final trim can be made using a piece of sand paper or emery cloth.

5. J. P. Greenstein and M. Winitz, "Chemistry of the Amino Acids," Vol. 2, p. 1233, John Wiley and Sons Inc., New York, 1961.

6. Although contact with trace sulfur-containing materials such as rubber stoppers is reported to poison palladium black,[5] we have never experienced this difficulty using the Plastipak syringes which contain rubber plungers. However, if this is a concern, a teflon barrier made from a single layer of 1 inch teflon thread tape (Crane Packing Company, Morton Grove, IL) may be used over the rubber plunger.