

Additions and Corrections

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An Efficient, Inexpensive, and Shelf-Stable Diazotransfer Reagent: Imidazole-1-sulfonyl Azide Hydrochloride

Page 3797. A number of safety issues regarding the preparation, storage and use of imidazole-1-sulfonyl azide hydrochloride **1·HCl** have become apparent. These concerns are listed below and are followed by several recommendations.

Concentration of the mother liquors from which **1·HCl** crystallized has resulted in an explosion. This solution may contain sulfonyl diazide and/or hydrazoic acid byproduct, which are both extremely sensitive, explosive substances.

Solid **1·HCl** is hygroscopic and reacts slowly with water to produce hydrazoic acid; care must be taken to prevent this from occurring.

The heterogeneous reaction of crystalline **1·HCl** with other substances can generate enough localized heat to initiate explosive thermal decomposition.

Impact sensitivity tests, as performed by J. Stierstorfer, N. Fischer, and T. M. Klapötke of the Department of Chemistry at the University of Munich, indicate that

1·HCl has an impact sensitivity of 6 J. In accordance with the *UN Recommendations on the Transport of Dangerous Goods*, **1·HCl** should be considered a “sensitive” material.

It is recommended that:

- the mother liquors from which **1·HCl** crystallizes, or any other solutions that contain **1·HCl**, should *not* be concentrated but rather disposed of as one would a solution containing hydrazoic acid.
- anhydrous acetonitrile be used to prepare **1·HCl** and that careful attention is paid to the stoichiometry and temperature of the reactions involved in the preparation.
- the reagent should always be added to and used in a solution to aid in heat dissipation.
- if **1·HCl** discolors in any way and/or appears wet, it should be diluted in water and disposed of per the first recommendation.

Finally, recent investigations (soon to be published) indicate that other salts of imidazole-1-sulfonyl azide are “insensitive” materials and efficient diazotransfer reagents.

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