

Department of Materials Engineering

Safe Preparation of Nital

Scope

This document covers safety considerations for preparation of Nital solution. It does not cover the numerous ways in which the reagent may be employed in research and testing.

Composition and Uses

Nital is the name given to an oxidizing etchant solution composed of aqueous nitric acid and ethanol; commercial preparations may also contain other organic compounds. It is used in metallography applications. Nital is made up of a powerful oxidizing acid (nitric acid) and an organic, and therefore reducing, alcohol. Commonly, the solution may contain up to 5% (by volume) concentrated nitric acid in ethanol. The solution must be made up carefully following the procedure outlined in this document.

Hazards

Concentrated nitric acid contains 68-70% HNO_3 by weight in water. It is a strong acid, meaning it fully dissociates. It is also a powerful oxidizing agent which can react violently with reducing agents, including organic compounds. Upon reduction, nitric acid may release highly toxic nitrogen oxide gases such as nitric oxide (NO) and nitrogen dioxide (NO_2). Red-brown fumes are indicative of NO_2 formation. Nitric acid itself is highly corrosive to skin and eyes and is toxic.

Ethanol is a flammable liquid. It is also toxic. Vapours have toxic effects.

Precautions

- See the MSDS for concentrated nitric acid, ethanol and Nital. MSDS are available on line from vendor websites or search, e.g. "Nital MSDS".
- Nital preparation must be performed in a properly functioning fumehood.
- Wear appropriate personal protective equipment, as per the MSDS.
- Have a tub of water available with twice the amount of water as the volume of Nital to be prepared. The tub must have more than enough residual capacity to contain the addition of the Nital solution as well. If it appears that the Nital solution is beginning to evolve toxic NO_2 gas etc., it can be poured into the tub of water to slow and quench the reaction. *This should not be done if it appears the reaction has become vigorous.* The quenched reaction mixture will need to be neutralized and disposed of. Ask your supervisor for assistance.

- Commercial Nital solutions contain up to 5% of concentrated nitric acid. More concentrated solutions will evolve gases and must not be stored in a closed container. Dispose of excess of such more concentrated Nital solutions when done (see below).
- Never add ethanol to nitric acid; always nitric acid to the ethanol. Otherwise vigorous or explosive reaction may occur and copious quantities of toxic gases will be evolved.
- Do not prepare solution with more than 5% concentrated nitric acid solution without the approval of your supervisor. Solutions containing more than about 10% concentrated nitric acid *by weight* may be explosive. (*The density of concentrated nitric acid is about 1.4 g/mL; ethanol is about 0.79 g/mL. Note that 5% concentrated nitric acid by volume in ethanol is already about 9% concentrated nitric acid by weight!*)

Procedure

- If you have not prepared the solution before you must be supervised until the person training you is satisfied you are competent to prepare the solution without supervision.
- Make only about as much of the solution as needed. Do NOT make up large volumes of the solution.
- Measure out the required volume of absolute ethanol, using a graduated cylinder, into a clean, dry beaker.
- Pour roughly the required volume of concentrated nitric acid (68-70%) into a clean dry beaker. From the beaker, measure out the required volume of the nitric acid into a *separate*, clean and dry graduated cylinder. (Avoid pouring directly from the bottle into a small graduated cylinder as spills can easily occur.)
- Pour the nitric acid very slowly into the ethanol with good stirring. If you notice a brown colouration developing and/or brown fumes evolving, cease the addition immediately. If safe to do so, pour the solution into the tub of water (see Precautions above). If vigorous gas evolution is occurring lower the fumehood sash completely and seek assistance from qualified personnel.

Storage

- Solutions up to 5% by volume in concentrated nitric acid may be stored in a cool dry place in a plastic bottle.
- Label appropriately. Make sure the date of preparation is on the label as well.
- Do not store solutions with >5% concentrated nitric acid!

- Do not store Nital solutions in glass bottles.
- Dispose of excess solutions within one week.

Disposal

Dispose of Nital as per MSDS instructions. For in-house treatment, seek advice from your supervisor. If you plan to neutralize the solution with base (if so instructed by your supervisor), dilute the solution with about two equal volumes of water. Add a solution of NaOH (e.g. about 1 mol/L) slowly with good stirring until all the HNO_3 is consumed. This can be indicated by an acid-base indicator. Seek advice from your supervisor about disposal, or from UBC Environmental Services Facility; a division of Risk Management. Used solutions may contain metal ions. Seek advice from your supervisor about proper disposal.