

STANDARD OPERATING PROCEDURE:

Diluting concentrated nitric acid

Note: To be undertaken only by trained personnel in conjunction with a current Safety Data Sheet (SDS) and site-specific risk assessment.

1. Introduction

Concentrated nitric acid is a clear, colourless to pale yellow liquid with a strong, sharp odour. It is both a very strong acid and a powerful oxidising agent, which must be handled with great care. The concentrated acid oxidises metals and organic material with the formation of nitrogen oxides, including nitrogen dioxide (NO₂), a reddish brown, acid-smelling, highly toxic gas. In diluted solutions (<2M), nitric acid behaves as a strong acid in its reaction with metals; however, only in the reaction of the dilute acid with magnesium is hydrogen gas itself liberated.

'Concentrated nitric acid' refers to a 70% solution of nitric acid. Concentrations above 90% are called 'fuming nitric acid' and should not be used in schools.

Nitric acid is used industrially in the manufacture of ammonium nitrate, in nitration reactions with organic compounds, as a pickling agent for stainless steel, in the etching of metals, and in the production of explosives.

Due to its highly corrosive and oxidising properties, all handling of concentrated nitric acid must be carried out in a fume cupboard. The dilution of nitric acid is an exothermic (heat producing) reaction.

Synonyms: hydrogen nitrate, aqua fortis, spirit of niter, engraver's acid, azotic acid.

2. Context

- These instructions are for the use of experienced teachers and technicians only.
- Do not make up a dilution for the first time without seeking practical advice from an experienced colleague.
- Students must not be asked to make up dilutions from concentrated nitric acid.

3. Safety notes

- This activity may only be carried out with appropriate facilities available i.e. running water, fume cupboard, chemical safety/eyewash station and relevant Personal Protective Equipment (PPE).
- Avoid contact with skin and eyes, and avoid breathing fumes. Concentrated and high molarity nitric acid fumes and liquid cause severe burns and eye damage. Fumes of concentrated nitric acid are irritating to the respiratory system.
- Always carry large bottles of concentrated acid either in an approved carrier or by firmly grasping the body of the bottle with one hand and placing the other hand underneath the bottle. Do not carry by the neck or lid. Do not rush.
- Always make up dilutions in a fume cupboard.

- Ensure that glassware is free from chips and cracks before use.
- For first aid, accident and spill procedures refer to SDS before performing the dilution.
- Exposure to light can cause decomposition of nitric acid with formation of nitrogen oxides, which colour the acid yellow to brown. The concentrated acid should be stored in amber bottles away from light, in bunding (secondary containment), segregated from combustible material such as glacial acetic acid. See the SDS for further details of incompatibilities.
- **Always add concentrated acid to water (never water to acid).**

4. Regulations, licences and permits

Not applicable.

5. Equipment

- Fume cupboard
- PPE (lab coat, safety glasses or face shield, nitric acid resistant gloves e.g. neoprene, butyl rubber or neoprene/latex blend gloves, closed in shoes). Note that nitrile and latex gloves are not recommended for use with concentrated nitric acid, but have good resistance to dilute nitric acid. Nitrile gloves may be used for 'splash protection' but if contaminated with concentrated acid, the gloves should be removed and hands should be washed.
- Concentrated nitric acid
- Distilled/de-ionised water and wash bottle
- Large (2L) borosilicate glass beaker for diluting the acid
- Small glass measuring cylinder
- Large (1L) glass measuring cylinder or volumetric flask
- Glass stirring rod or magnetic stirrer and magnetic stirring bar
- Pre-labelled storage bottle

Note: For laboratory 'Stock' solutions use measuring cylinders. For greater accuracy use volumetric flasks and pipettes.

6. Operating procedure

To make 1 litre of stock solution:

1. Wear PPE and work at a fume cupboard.
2. Into the large beaker place about 650mL of distilled water (or an amount such that the volume of water combined with the volume of concentrated acid to be added does not exceed about 850mL).
3. Carefully measure the required volume of concentrated acid in the small measuring cylinder; see table below. (If your concentrated acid is stored in a large 2.5L Winchester bottle, firstly pour some into a smaller bottle or beaker to be able to safely pour into the measuring cylinder as handling liquids in smaller containers is safer and easier.) *Hint:* keep the label of the acid bottle uppermost when pouring and clean up any spilt liquid from the outside of the bottle.

(Operating procedure cont....)

4. Add the concentrated acid slowly to the water with stirring. Dissolution of the concentrated acid will generate heat.
5. With distilled water from the wash bottle, rinse the remaining acid from the small measuring cylinder into the solution in the large beaker.
6. When the solution has cooled to room temperature, transfer it to a 1L measuring cylinder or volumetric flask. Rinse the large beaker and stirring rod or magnetic stirring bar with distilled water and add the rinsings to the solution in the measuring cylinder or volumetric flask. Make up the volume to 1 litre.
7. Pour this solution into the pre-labelled bottle.
8. On completion of the activity, clean up spills or splashes with plenty of water and thoroughly clean all used equipment and fume cupboard. All glassware that may be contaminated with concentrated acid should be rinsed with water BEFORE removing it from the fume cupboard.

Table: Volume of concentrated HNO₃ required to prepare 1L of dilute solution

Molarity required	Volume of concentrated HNO ₃ (mL) ^a
0.01M	0.6 ^b
0.1M	6.4 ^b
0.5M	32 ^b
1M	64
2M	127

^a Based upon a 70% solution, approximately 15.7M.

^b 0.5M solution can be prepared by a 1 part in 2 dilution of the 1M solution, a 0.1M solution by a 1 part in 10 dilution of the 1M solution, and a 0.01M solution by a 1 part in 10 dilution of the 0.1M solution.

7. Trouble shooting/emergencies

- First aid: See latest SDS for more detailed information
 - **If swallowed:** Do not induce vomiting. Rinse mouth with water, and then give plenty of water to drink. Seek urgent medical attention.
 - **If in eyes:** Hold open and irrigate with copious quantity of water for at least 15 minutes. Seek urgent medical attention.
 - **Skin/clothes:** Immediately flush skin and clothes with excess water under a safety shower. Remove contaminated clothing. Seek medical attention.
 - **If inhaled:** Remove to fresh air. Seek medical attention.
 - For further advice contact the Poisons Information Centre on 131 126.
- Check plastic bottle caps for corrosion regularly and ensure bottles are tightly screwed closed.

8. Waste disposal

- Clean up any small spillages immediately with plenty of water (larger spills should be neutralised). When fuming stops, sweep/mop up. Spills that are too large to neutralise safely should be absorbed with dry sand or vermiculite and placed in a labelled container for collection and disposal by a registered hazardous waste disposal company. Do not absorb spill with combustible materials such as sawdust, paper or clothing as fire may result.
- To neutralise concentrated acid, first dilute the acid by adding it carefully to a larger volume of water. Neutralise the solution with sodium bicarbonate, sodium carbonate or a 50:50 mixture of sodium carbonate and calcium hydroxide so that the pH is in the range pH 6-8. Exercise care as heat & corrosive fumes are produced. Use an indicator such as Universal Indicator to determine the pH. Wash the neutralised diluted solution to waste down the sink with excess cold water.
- Large quantities of waste nitric acid need to be collected and disposed of by a registered hazardous waste disposal company.

9. Related material

- Manufacturer's Safety Data Sheet
- Risk Assessment

References:

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Greenwood,N.N. and Earnshaw,A. 1984, Chemistry of the Elements, Pergamon Press: Oxford, pp. 536-545.

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History of reviews:

Date	Version Number	Notes
Jun 2015	Version 1.0	
Nov 2016	Version 2.0	Added magnetic stirrer and magnetic stirring bar to equipment Additional instructions for rinsing the stirring rod/magnetic stirring bar