

STANDARD OPERATING PROCEDURE:

Diluting concentrated acetic 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

Acetic acid is an organic acid with chemical formula CH_3COOH . Acetic acid containing less than 1% water is commonly referred to as 'glacial acetic acid', so-called as it resembles ice crystals when it freezes, which it does at just under 17°C . The concentrated acid is a colourless, corrosive liquid with a pungent odour, and should be handled with care.

The name 'acetic' is derived from the Latin *acetum* meaning 'sour wine'. The production of acetic acid dates back to at least 10000 BC with the emergence of the practice of winemaking. While industrial-grade acetic acid is synthesised from fossil fuels, food-grade vinegar, which typically contains 4-8% acetic acid, is still made through the fermentation of sugars by yeast to give ethanol, which is then oxidised to acetic acid by bacteria of the genus *Acetobacter*.

In senior school chemistry, concentrated acetic acid is used in the preparation of acetate esters. In dilute form in junior and senior school science, acetic acid is used as an example of a weak organic acid.

The concentrated acid is flammable and combustible and should be handled away from flames or sparks; it is also hygroscopic, which means that it absorbs water from the air.

Synonyms: ethanoic acid, glacial acetic acid, vinegar acid, methanecarboxylic acid, ethylic 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 acetic 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 acetic acid liquid can cause severe burns and eye damage. Fumes of concentrated acetic acid cause irritation to the eyes and 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.
- Note that the concentrated acid should be stored in bunding (secondary containment), segregated from strong bases and oxidising agents, including nitric acid, and segregated from other acids. See the SDS for further details of incompatibilities.
- Pure (glacial) acetic acid has a melting point of 16.7°C and may freeze in cool weather. The frozen acid can be melted by placing the bottle in a plastic bag in a bath of warm water.
- **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, acetic acid resistant gloves (butyl-rubber gloves are well-suited for the handling of concentrated acetic acid; nitrile, neoprene/latex or latex gloves also provide good splash protection against the concentrated acid), closed-in shoes.
- Concentrated acetic 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 acetic acid required to prepare 1L of dilute solution

Molarity required	Volume of concentrated acetic acid (mL) ^a	Volume of concentrated acetic acid (mL) ^b
0.01M	0.6 ^c	0.6 ^c
0.1M	5.8 ^c	6.4 ^c
0.5M	29	32
1M	58	64
2M	116	127

^a Based upon a 99% solution, approximately 17.3M.

^b Based upon a 90% solution, approximately 15.7M.

^c 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.

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 medical attention.
 - **Skin/clothes:** Remove contaminated clothing and drench the area with excess water under a safety shower. Seek medical attention.
 - **If inhaled:** Remove to fresh air and seek medical attention if symptoms persist.
 - 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 non-combustible material such as dry sand or vermiculite and placed in a labelled container for collection and disposal by a registered hazardous waste disposal company.
- 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 and 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 acetic 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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Cheryan, M. Acetic Acid Production, in Encyclopedia of Microbiology, (M. Schaechter, Editor-in-Chief) 3rd Edition, Elsevier Inc. (2009), Academic Press, pp. 145-149.

CLEAPSS, 2014, Hazcard 38A Ethanoic acid, methanoic acid and their salts, <http://www.cleapss.org.uk/attachments/article/0/HC-038A.pdf?Secondary/Science/Hazcards/> (Subscription required. Accessed June 2015)

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'Material Safety Data Sheet – Acetic acid 89-100%', January 2011, Chem-supply website <https://www.chemsupply.com.au/documents/AA0091CH0J.pdf> (Accessed June 2015)

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