

STANDARD OPERATING PROCEDURE:

Diluting concentrated sulphuric 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 sulphuric acid (H_2SO_4) is a hazardous, highly corrosive chemical, which must be handled with great care. It is a clear, colourless, viscous (thick, oily) liquid when pure, but has a yellowish to brown tinge when contaminated with impurities such as organic material. It has a higher density than water and is hygroscopic, which means that it absorbs water from the air.

The dilution of sulphuric acid is a highly exothermic (heat generating) reaction.

Synonyms: hydrogen sulphate, oil of vitriol.

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 sulphuric 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 sulphuric acid causes severe burns and eye damage.
- 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.
- **Dilution of sulphuric acid liberates much heat and can cause a glass beaker to crack so must be conducted using a water bath to cool the solution.**
- **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, sulphuric acid resistant gloves e.g. neoprene or PVC gloves, closed in shoes). Note that nitrile and latex gloves have poor resistance to concentrated sulfuric acid, but good resistance to dilute sulphuric acid. Nitrile gloves may be used for 'splash protection' but if contaminated with concentrated acid, the gloves should be removed and hands washed.
- Concentrated sulphuric 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
- Large trough or bucket containing cold water to act as a water bath to cool the diluted acid
- 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. Place the beaker into a cold water bath and ensure that it will not tip. Ensure the cold water in the bath comes about equal to the level of the water inside the beaker.
4. 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.
5. Very slowly add the concentrated acid to the water with constant stirring. It is important to stir whilst adding the acid to the water to avoid a layer of concentrated acid forming at the bottom of the beaker creating a temperature gradient.
6. Rinse out the small measuring cylinder with distilled water using the wash bottle.
7. When the acid is well mixed leave the diluted acid in the water bath until it has cooled to room temperature. Note: Do not handle the diluted mixture until cooled. The cold water in the water bath may have to be changed.

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8. When the solution has cooled to room temperature, transfer it to a 1 litre measuring cylinder or volumetric flask. Rinse the large beaker with distilled water. Make up the volume to 1 litre.
9. Pour this solution into the pre-labelled bottle.
10. 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, which may be contaminated with concentrated acid, should be rinsed with water BEFORE removing it from the fume cupboard.

Table: Volume of concentrated H₂SO₄ required to prepare 1L of dilute solution

Molarity required	Volume of concentrated H ₂ SO ₄ (mL) ^a
0.01M	0.55 ^b
0.1M	5.5 ^b
0.5M	27.5 ^b
1M	55
2M	110

^a Based upon a 98% solution, approximately 18.2M

^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 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:** If spilt on skin or clothes quickly wipe off with a dry cloth to absorb as much liquid as possible. Remove contaminated clothes 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.
- To neutralise concentrated acid, first dilute the acid by adding it carefully to a larger volume of water. Neutralise waste with sodium bicarbonate 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.
- Larger quantities will 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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