

ENVIRONMENTAL ISOTOPE LABORATORY
DEPARTMENT OF EARTH SCIENCES
UNIVERSITY OF WATERLOO

Technical Procedure 21.0

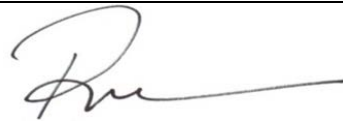
PHOSPHORIC ACID PREPARATION

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APPROVAL:



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2005-04-08

Date

Issue Date: 2005-04-07

Supersedes: Technical Procedure 21.0, Revision 02

Revision: 02

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1.0 INTRODUCTION

This technical procedure provides instruction for the preparation of 100% phosphoric acid (H_3PO_4). The method is a revision of that described by McCrea (1950) for reaction with carbonates to produce CO_2 .

1.1. Precautions

The precautions listed are not comprehensive. Use in conjunction with the relevant Material Safety Data Sheets (MSDS). Generic MSDSs from Aldrich Chemical Company are available on any computer with internet access. Follow acceptable safety procedures including the wearing of safety glasses at all times and gloves if necessary.

Whenever feasible, keep waste separate and retain for processing in the Environmental Safety Facility (ESF) in ESC 150. Acids and bases not containing toxic substances may be poured down the drain provided they are between pH 5.5 and 9 (or can be brought to pH 5.5-9 by dilution with water in a maximum ratio of 5:1 [water to solution]). **WHEN IN DOUBT, RETAIN ALL WASTE IN SEPARATE LABELED CONTAINERS AND TAKE TO THE ESF.**

1. Chromium Trioxide (CrO_3) is a odourless dark red deliquescent solid. **DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE A FIRE. CORROSIVE. CAUSES SEVERE BURNS TO EVERY AREA OF CONTACT. HARMFUL IF SWALLOWED OR INHALED. AFFECTS THE RESPIRATORY SYSTEM, LIVER, KIDNEYS, EYES, SKIN AND BLOOD. MAY CAUSE ALLERGIC REACTION. CANCAER HAZARD. CAN CAUSE CANCAER.** Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to environmental safety facility.
2. Hydrogen Peroxide (H_2O_2) is a clear liquid with slight odour. It decomposed slowly to release O_2 and H_2 . Avoid excess heat above 300°C , ignition sources, light, metals and reducing agents. Wear protective eyewear, gloves and clothing to prevent skin exposure. Let evaporate in small quantities in the fume hood.
3. Phosphoric Acid (H_3PO_4) is corrosive with moderate health risk. It gives off flammable vapours and may form an explosive mixture with air. Wear safety glasses and gloves in a well-ventilated area.
4. Phosphorous Pentoxide (P_2O_5) reacts violently with water. It is corrosive. Prepare in the fume hood with gloves and safety glasses. Keep away from water.

2.0 REAGENTS

Phosphoric Acid (H_3PO_4) 85% — 1.5 L

Phosphorous Pentoxide (P_2O_5) — 1 Kg

Chromium Trioxide (Cr_2O_3) — 7 - 15 grains

Hydrogen Peroxide (H_2O_2) 30% — 10 ml

3.0 PREPARATION

1. In a clean dry 3500 ml beaker, mix 1.5 L H_3PO_4 (85%) and 1 Kg P_2O_5 (phosphorous pentoxide).
2. Add 7-8 grains of Cr_2O_3 (chromium trioxide).

NOTE: Normally the solution is yellowish. If the solution turns green add 4-5 more grains (maximum).

3. Heat to approximately 180°C (maximum 200°C) for 6 hours. Monitor temperature with a metal encased thermometer placed inside a glass tube sealed at one end.
4. Slowly stir in 10 ml of H_2O_2 (hydrogen peroxide).

CAUTION: H_2O_2 reacts VIOLENTLY with HOT acid. Be very careful!!!

5. Heat to 220°C for 2 hours.
6. Cool to < 100°C before transferring the acid to the stock bottle. To prevent breakage of the stock bottle, preheat in the drying oven for 10 minutes prior to transfer.

4.0 REFERENCES

McCREA J.M., 1950. On the isotopic chemistry of carbonates and a Paleo-temperature scale. J. Chem. Phys. 18,849