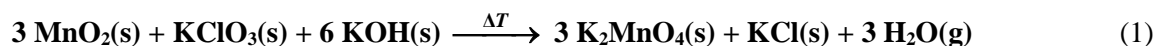


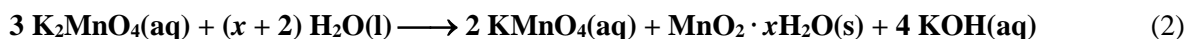
## Preparation of potassium permanganate

Permanganates themselves are very strong oxidizing agents in both acidic and basic solutions. Therefore, only the strongest oxidizers can oxidize manganese in lower oxidation states to permanganates. In the laboratory, potassium permanganate is always prepared by the two-step synthesis.

In the first step, manganese(IV) oxide is oxidized in a alkaline melt at high temperature to manganate(VI), according to the reaction 1



In the second step, the manganate(VI) undergoes disproportionation by the instrumentality of acids or acidic solutions. Manganate(VI) are unstable even in neutral solutions.



Potassium hydroxide must be neutralized, for example with acetic acid. Precipitated manganese(IV) oxide may then be separated by filtration.

When heated over 240 °C, potassium permanganate decomposes, and gaseous oxygen is excluded.



### Work

Prepare potassium permanganate from 4.00 g of manganese(IV) oxide.

### Chemicals

- manganese(IV) oxide,  $\text{MnO}_2$ , black insoluble powder,
- potassium chlorate,  $\text{KClO}_3$ , white crystalline substance,
- potassium hydroxide,  $\text{KOH}$ , white hygroscopic substance, flakes or granules.

### Procedure

❶ Put finely powdered manganese(IV) oxide into a cleaned steel dish and anneal it in the strongest flame for 10 minutes to eliminate oxidizable impurities (Fig. 1). Then let it cool down and add calculated amounts of potassium chlorate in a 50 % excess and potassium hydroxide in a 40 % excess. Stir the mixture carefully with a steel rod to homogenize it. Under continuous stirring, heat the mixture gently until hydroxide is molten. In few minutes the liquid mixture solidifies. Then turn on the strongest burner flame and anneal the dish until the mixture changes its colour to dark green. The colour is caused by potassium manganate(VI) formed. Then stop heating.

❷ Add water in small portions to the cooled mixture in the steel dish. Under continuous stirring bring the mixture to a boil. The hot solution often contains also insoluble manganese(IV) oxide. Pour the mixture into a bigger beaker and add in portions the calculated amount of 98.0 wt% acetic acid to neutralize the highly alkaline solution. After each adding put one drop of the solution on a filter paper and check the colour of the mixture. If the original dark green colour is still visible, add another portion of acid. In the fully neutralized solution, the colour should be deep purple caused by potassium permanganate.

❸ Heat the neutralized solution almost to the boiling point and let it settle at the bottom. Pour the purple solution carefully on a Büchner funnel and filter it to a clean suction flask. All hydrated manganese(IV) oxide should remain in the beaker. Pour the filtered solution from the suction flask into an evaporating dish on a water bath to saturate it (Fig. 2). Pour the saturated solution of potassium manganese into a crystallizing dish and place it in the coolest part of the laboratory. Once cooled to room temperature, filter out the crystallized dark purple product on a filter with glass frit and dry it between two sheets of filter paper.

❹ Wash the insoluble hydrated manganese(IV) oxide in the beaker by decantation with water until the solution is colourless. Then pour off water, transfer wet hydrated manganese(IV) oxide into the Petri dish and dry it up at temperature over 105 °C.

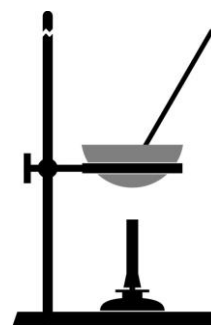


Fig. 1 Apparatus for alkaline oxidation.

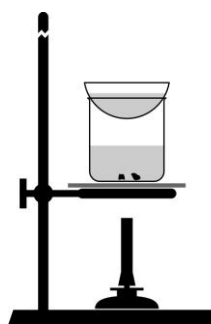


Fig. 2 Saturation on a water bath.

## *Safety instructions*

### Manganese(IV) oxide – MnO<sub>2</sub>

- R20** Harmful by inhalation.
- R22** Harmful if swallowed.
- S25** Avoid contact with eyes.

### Potassium chlorate – KClO<sub>3</sub>

- R9** Explosive when mixed with combustible material.
- R20** Harmful by inhalation.
- R22** Harmful if swallowed.
- R51/53** Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
- S13** Keep away from food, drink and animal feedingstuffs.
- S16** Keep away from sources of ignition – No smoking.
- S27** Take off immediately all contaminated clothing.
- S61** Avoid release to the environment. Refer to special instructions/safety data sheet.

### Potassium hydroxide – KOH

- R35** Causes severe burns.
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
- S37/39** Wear suitable gloves and eye/face protection.

### Carbon dioxide – CO<sub>2</sub>

- R61** May cause harm to the unborn child.
- R23** Toxic by inhalation.
- R48/23** Toxic: danger of serious damage to health by prolonged exposure through inhalation.
- S14** Keep away from .. (incompatible materials to be indicated by the manufacturer).
- S45** In case of accident or if you feel unwell seek medical advice immediately (show the label where possible).
- S23** Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer).
- S36/37/39** Wear suitable protective clothing, gloves and eye/face protection.

### Acetic acid – CH<sub>3</sub>COOH

- R10** Flammable.
- R35** Causes severe burns.
- S23** Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer).
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

### Potassium permanganate – KMnO<sub>4</sub>

- R8** Contact with combustible material may cause fire.
- R22** Harmful if swallowed.
- R50/53** Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
- S60** This material and its container must be disposed of as hazardous waste.
- S61** Avoid release to the environment. Refer to special instructions/safety data sheet.