

TESTING FOR THE PRESENCE OF PEROXIDES

Reference: Chem 13 News, March 1984, p. 3

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The following chemicals may form explosive peroxides after an extended period of time. They should be checked before using and discarded if dangerous levels of peroxides are found.

Benzyl alcohol
sec-Butyl alcohol (2-Butanol)
Cyclohexanol
Cyclohexene
Diethyl ether
Isoamyl alcohol (isopentyl alcohol)
Methyl isobutyl ketone
2-Octanol

One or more of the following tests should be done on any suspect chemicals. It must be noted that these tests are valid only for simple chemicals. Some organic compounds may also act as oxidizing agents and create false positive tests. When dealing with peroxidizable materials that may be insoluble in the test solution, it may be necessary to use a co-solvent such as peroxide-free isopropanol to complete the test.

METHOD A

Note: This method is the most rapid method of peroxide detection.

Add 0.5 to 1.0 mL of the material to be tested to an equal volume of glacial acetic acid to which has been added 0.1 g of sodium iodide or potassium iodide.

yellow colour = low peroxide concentration in sample
brown colour = high peroxide concentration in sample

Always prepare the iodide-acetic acid mixture at the time of the test to avoid false results due to oxidation.

METHOD B (Recommended by the Manufacturing Chemists Association)

Note: Colour development in this method may take up to 10 minutes.

Add 1 mL of a freshly-prepared 10% aqueous solution of potassium iodide to 10 mL of the compound being tested in a 25 mL glass-stoppered cylinder of colourless glass protected from strong light.

Barely discernable yellow colour = 0.001 to 0.005% peroxide as H₂O₂
Definite yellow colour (*) = 0.01 %
Brown colour (*) = ?

* A percentage of 0.01% or more is not safe.

METHOD C

Prepare a sodium ferrocyanide reagent by dissolving 9 g of FeSO₄•7H₂O in 50 mL of 18% HCl. Add 0.5 to 1.0 g of granulated zinc followed by 5 g of sodium thiocyanate. When the transient red colour fades, add 12 g of sodium thiocyanate and decant the liquid from the unused zinc into a clean stoppered bottle. Add 1 drop of this solution to 1 drop of the compound to be tested in a clear glass vial.

Barely discernable pink colour = 0.001% peroxide as H₂O₂
Pink to cherry red = 0.002%
Red (*) = 0.008%
Deep red (*) = 0.04 %

* A percentage of 0.01% or more is not safe.