Canadian Conservation Institute
Institut canadien de conservation

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The Diphenylamine Spot Test for Cellulose Nitrate in Museum Objects

Introduction

In his paper on the instability of cellulose nitrate adhesives, Koob (1982) describes the diphenylamine spot test for the detection of cellulose nitrate. Diphenylamine is a very common reagent, and there are many recipes for it in the chemical literature. A slight modification of Koob's reagent is recommended in this Note for the rapid detection of cellulose nitrate, not just in adhesives but in any museum object or in any material proposed for museum or conservation use.

Preparing the Reagent

The reagent is a solution of 0.5% diphenylamine in 90% sulphuric acid. To prepare the reagent, slowly add 90 ml of concentrated sulphuric acid to 10 ml of water while stirring continuously, and then add this, in successive small portions, to 0.5 g of diphenylamine. Exercise extreme caution while adding the acid to the water. A substantial amount of heat is generated during this dilution. **Always add acid to water slowly while stirring to prevent splattering.** Carry out this operation at a sink or near an abundant source of running water to clean up equipment or any spills. Flood spills with water immediately.

The resulting test solution is very corrosive both to humans and to artifacts. It can be stored for several years, and remains effective even after it develops a slight brown or blue tinge. The quantity prepared (100 ml) is sufficient for several hundred tests. Store the test solution in glass bottles bearing the labels "0.5% Diphenylamine in 90% Sulphuric Acid" and "Corrosive". Glass bottles with plastic (not metal) screw caps are acceptable **only if** the cap liners are resistant to 90% sulphuric acid. Paper, cork, rubber, and metal foil liners are useless. Polyethylene and polypropylene liners and caps are suitable.

Procedure for the Spot Test

Take a tiny chip or scraping (smaller than a pin-head) from an unobtrusive part of the object to be tested, ensuring that the reagent does not come in contact with the object itself. If the reaction is to be carried out under a microscope, the sample can be microscopic in size. Place the sample on a glass surface (such as a microscope slide) or on a white glazed porcelain spot plate. Place a single drop of the reagent on the sample, using a pipette, an eye-dropper, or a glass rod. A blue-violet stain appearing within seconds on the sample and then diffusing into the test drop indicates the presence of cellulose nitrate. No colour change, or the appearance of other colours such as orange, yellow, brown, or green, is a negative result, indicating that cellulose nitrate is absent. Once the test is complete, wash all materials used during the test with water and discard them carefully. Remember, the reagent is corrosive.

1030 Innes Road
Ottawa ON K1A 0M5 Canada
Tel.: (613) 998-3721
Fax: (613) 998-4721

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To become familiar with the test, and to provide a reference for colour and speed of reaction, test commonly available cellulose nitrate products simultaneously with the specimen. Collodion and some glues such as DUCO Cement, UHU All-Purpose Clear Adhesive, and HMG Adhesive test positive with the reagent and can be used as reference materials.

Interferences with the Test

This test is based on the oxidation of colourless diphenylamine to quinoid-type blue dye by oxidizing nitrogen oxide ions liberated from the cellulose nitrate through the reaction with sulphuric acid (Maloney and Thornton 1982). The presence of certain other oxidizing ions could cause a similar reaction. In fact, this property has been exploited in other analytical chemical procedures (Tomicek 1951). Materials pigmented with compounds of oxidizing ions such as chromates (possible components of yellow, orange, or green pigments) could be a problem. There have also been reports of suppression of the test reaction by admixed resins including ester gums, copal, and other natural resins (Vollman 1961). These admixtures usually occur only in varnishes and coatings, not in plastic objects.

This spot test was conducted on 30 samples from random museum objects, coatings, and adhesives. Each sample was also examined by infrared spectrophotometry (IR). All samples that were shown by IR to contain cellulose nitrate gave positive results with the spot test. All samples shown by IR not to contain cellulose nitrate tested negative. No problems with interferents occurred.

The test is very sensitive. It can be applied to any type of object that can be sampled to give a microscopic chip or scraping for testing. Tiny amounts of cellulose nitrate admixture or contamination (such as remaining traces of coatings or adhesives) can yield a positive test. The consequences of these "false positive" tests may be unnecessary, overly cautious treatment of some objects.

Suppliers

Diphenylamine and sulphuric acid are both readily available from standard chemical suppliers. Microscope slides and spot plates are available from standard laboratory equipment suppliers.

Diphenylamine:
Fisher Scientific Ltd., Cat. No. 0-2611
J.T. Baker Chemical Co., Cat. No. 1944

Sulphuric Acid:
Fisher Scientific Ltd., Cat. No. A-300
J.T. Baker Chemical Co., Cat. No. 9681

Microscope Slides:
Fisher Scientific Ltd., Cat. No. 12-500

Spot Plates:
Fisher Scientific Ltd., Cat. No. 13-745;
Cat. No. 13-748B

References

Koob, Stephen P. "The Instability of Cellulose Nitrate Adhesives," *The Conservator*, no. 6 (1982), pp. 31-34.

Maloney, R.S. and J.I. Thornton. "Color Tests for Diphenylamine Stabilizer and Related Compounds in Smokeless Gunpowder," *Journal of Forensic Sciences*, vol. 27, no. 2 (1982), pp. 318-329.

Tomicek, O. *Chemical Indicators*. Trans. by A.R. Weir. London: Butterworths Scientific Publications, 1951, p. 171.

Vollman, H.F. "Detection of Nitrocellulose," *Journal of the Oil and Colour Chemist's Association*, vol. 44 (1961), pp. 308-310.

by R. Scott Williams, Conservation Processes Research Division

Copies are also available in French.

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