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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 42357-2023

**Water-insoluble dyes - Determination of purity - Liquid
chromatography**

非水溶性染料 纯度的测定 液相色谱法

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Foreword

This document was issued on 17 March 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 October 2023.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 42357-2023 determines the purity of dyes that do not dissolve in water, disperse dyestuffs among them, by liquid chromatography. The principle of the method is plain: the sample is dissolved in a solvent such as methanol, acetone or dioxane, the components are separated chromatographically, and quantification is done by peak area normalisation at a suitable wavelength. The test method clause is the body of the document, running from the general provisions - analytical reagents only, and grade 3 water to GB/T 6682-2008 - through preparation and the chromatographic conditions to the determination itself, and precision and a test report clause close it. Area normalisation is the reason the method works in practice: a pure reference of the dye itself is often not to be had, and normalising within the chromatogram lets a laboratory state a purity without one. Purity is what a dyehouse is actually buying. Unreacted intermediates and by-products change the depth of shade a nominal dosage gives and can shift the hue between deliveries, so a batch that meets its strength claim on paper can still force a re-dye. Dye manufacturers, textile dyehouses, traders and testing laboratories are the users.

This document describes a liquid chromatography method for the determination of purity of water-insoluble dyes.

This document applies to the determination of purity of disperse dyestuffs, water-insoluble fluorescent whitening agents, and solvent dyes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 6682-2008, Water for analytical laboratory use - Specification and test methods

GB/T 8170-2008, Rules of rounding off for numerical values & expression and judgment of limiting values

3 Terms and definitions

No terms and definitions need to be defined in this document.

4 Principle of the method

After dissolving the dye sample by using a solvent (such as methanol, acetone or dioxane), use liquid chromatography to separate the components in the dye, and use the peak area normalization method for quantification at a suitable wavelength.

5 Test method

5.1 General provisions

Unless otherwise specified, only use reagents confirmed to be analytical reagents and grade 3 water specified in GB/T 6682-2008. The determination of test results shall be carried out according to the comparison method of rounded values in 4.3.3 of GB/T 8170-2008.

5.2 Reagents

5.2.1 Methanol: chromatographic pure.

5.2.2 n-hexane: chromatographic pure.

5.2.3 Dioxane: chromatographic pure.

5.2.4 Acetonitrile: chromatographic pure.

5.2.5 Trichloromethane.

5.2.6 Acetone.

5.2.7 Acetic acid.

5.2.8 Ammonium acetate.

5.3 Instruments and equipment

5.3.1 Liquid chromatograph: infusion pump - flow range 0.1 mL/min ~ 5.0 mL/min, within which the flow stability is $\pm 1\%$; with ultraviolet/visible light detector (VWD) or diode array detector (DAD).

5.3.2 Electronic balance: accuracy 0.1 mg.

5.3.3 Chromatographic work station or integrator.

5.3.4 Flat-top microsyringe or autosampler.

5.3.5 Ultrasonic generator.

5.3.6 Chromatographic columns, which shall meet the following requirements:

a) Chromatographic columns for normal phase systems: stainless steel columns of stationary phase Rx-SIL, length 150 mm ~ 250 mm, inner diameter 3.9 mm ~ 4.6 mm, particle size 3.5 μm ~ 5.0 μm ;

b) Chromatographic columns for reversed-phase systems: stainless steel columns of stationary phase C18, length 150 mm ~ 250 mm, inner diameter 3.9 mm ~ 4.6 mm, particle size 3.5 μm ~ 5.0 μm .

5.4 Liquid chromatography analysis conditions

5.4.1 Liquid chromatography separation system mode

5.6 Determination

The optimum analysis conditions can be selected based on different instruments and equipment and different types of samples.

In the reversed-phase chromatography system mode, the organic phase of the mobile phase can be methanol or acetonitrile, and the water phase can be pure water or added with a certain proportion of acetic acid or ammonium acetate. According to the different

dye structures, the ratio of acetic acid and ammonium acetate in the water phase and the volume ratio of the organic phase and the water phase can be adjusted to achieve the best separation effect. The mobile phase shall be shaken well and then degassed using an ultrasonic generator.

Turn on the chromatograph. After the instrument runs stably, use a flat-top microsyringe or autosampler to inject the sample solution. After the components have finished flowing out, use a chromatographic work station or integrator to process the results. When measuring water-insoluble fluorescent whitening agents, the room shall be properly protected from light to avoid sunlight irradiating the test sample. The measurement shall be performed continuously and shall not be left for too long to prevent the sample solution from being exposed to light and affecting the measurement results.

5.7 Calculation of results

Using the peak area normalization method, the purity of water-insoluble dyes is based on w_i and calculated according to Formula (1):

Where:

A_i - the peak area value of the main component of water-insoluble dyes in the sample solution;

ΣA_i - the sum of the peak area values of the main component of water-insoluble dyes and their organic impurities in the sample solution.

Keep the calculation result to two decimal places.

5.8 Chromatogram

Typical chromatograms for determination of purity of water-insoluble dyes by liquid chromatography are shown in Appendix A.

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