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# INTERNATIONAL STANDARD



# 3499

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## Plastics — Aqueous dispersions of homopolymers and copolymers of vinyl acetate — Determination of bromine number

*Matières plastiques — Dispersions aqueuses d'homopolymères et de copolymères d'acétate de vinyle — Détermination du nombre de brome*

First edition — 1976-06-01

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UDC 678.744.422 : 543.871.3

Ref. No. ISO 3499-1976 (E)

**Descriptors** : plastics, copolymers, polymers, vinyl acetate copolymers, chemical tests, determination, bromine number.

## FOREWORD

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International Standard ISO 3499 was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in September 1974.

It has been approved by the Member Bodies of the following countries:

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The Member Body of the following country expressed disapproval of the document on technical grounds:

Netherlands

# Plastics — Aqueous dispersions of homopolymers and copolymers of vinyl acetate — Determination of bromine number

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the bromine number of aqueous dispersions of homopolymers and copolymers of vinyl acetate.

NOTE — In some cases, and in particular when this method is applied to an **unmodified dispersion of vinyl acetate homopolymer**, it is possible to calculate the **residual monomer content** also (see note, clause 7).

## 2 DEFINITION

**bromine number**: The number of grams of free bromine (Br) consumed by 100 g of the sample under the conditions of test.

## 3 PRINCIPLE

Bromination of the residual vinyl acetate and of any other substances present which are capable of undergoing bromination, by treatment of the sample in acid solution with excess potassium bromate-bromide solution.

Determination of the excess bromine by addition of potassium iodide solution and titration of the liberated iodine with a standard volumetric solution of sodium thiosulphate.

Calculation of the bromine number.

## 4 REAGENTS

During the analysis, use only reagents of recognized analytical reagent quality, and only distilled water or water of equivalent purity.

### 4.1 Methanol.

### 4.2 Hydrochloric acid, $\rho$ 1,18 g/ml.

### 4.3 Potassium bromate-bromide solution.

Dissolve 5,6 g of potassium bromate and 25 g of potassium bromide in approximately 500 ml of water in a 1 000 ml volumetric flask. Dilute to the mark with water.

NOTE — Bromine water may be used instead of potassium bromate-bromide solution.

### 4.4 Potassium iodide, 200 g/l solution.

### 4.5 Sodium thiosulphate, 0,2 N standard volumetric solution.

## 5 APPARATUS

Ordinary laboratory apparatus, and in particular:

5.1 **Conical flasks**, capacity 500 ml, having ground glass stoppers.

5.2 **Burettes**, capacity 25 ml, graduated in 0,05 ml divisions, having ground glass taps.

5.3 **Balance**, accurate within 0,01 g.

## 6 PROCEDURE

### 6.1 Determination

From the test sample weigh, to the nearest 0,01 g, into a conical flask (5.1), a test portion of between 7 and 9 g.

Dilute with 50 ml of water, mix well and add 200 ml of methanol (4.1).

Add 10 ml of hydrochloric acid (4.2), insert the stopper and shake the flask well in order to mix the contents thoroughly.

Using a burette (5.2), add sufficient potassium bromate-bromide solution (4.3) to produce a permanent yellow colour; continue adding the solution until a whole number of millilitres has been added.

Stopper the flask and allow the contents to react for about 2 min.

Add 5 ml of potassium iodide solution (4.4) by loosening the stopper and pouring the solution down the side into the flask. Re-stopper the flask, in order to prevent loss of bromine, shake it and allow the reaction to proceed for 1 min.

Titrate the iodine produced with the standard volumetric sodium thiosulphate solution (4.5) until the colour disappears.

NOTE — The end-point may be determined by potentiometry, this method being of particular value if the end-point of the volumetric titration is not sharp.

Make a second determination under the same conditions.

### 6.2 Blank test

Carry out a blank test using the same quantities of reagents, particularly the same volume of potassium bromate-bromide solution (4.3) established during the determination, but omitting the test portion.