International Standard 6713

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Paints and varnishes — Preparation of acid extracts from liquid paints

Peintures et vernis — Préparation des extraits acides des peintures liquides

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Foreword

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International Standard ISO 6713 (formerly ISO/DIS 3856/1) was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in April 1978.

It has been approved by the member bodies of the following countries :

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France
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Paints and varnishes — Preparation of acid extracts from liquid paints

0 Introduction

This document is for use in conjunction with ISO 3856, *Paints and varnishes — Determination of "soluble" metal content.*

1 Scope and field of application

This International Standard specifies methods for the preparation of acid extracts required as the test solutions for the determination of the "soluble" metal contents of liquid paints and related products.

2 References

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

ISO 1512, *Paints and varnishes — Sampling.*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 3856, *Paints and varnishes — Determination of "soluble" metal content.*

3 Definition

"soluble" metal content of paint : The metal content of the pigments and extenders soluble in a dilute acid of defined concentration plus the total metal content present in the liquid portion of the solvent-diluted paint.

4 Principle

Dilution of a sample of the liquid product to be tested, followed by centrifugal separation of the solid matter in the sample. Three methods, dependent on the medium of the paint being examined, are described for the separation of the solid matter.

Extraction with 0,07 mol/l hydrochloric acid solution of the separated solid matter (consisting of pigments and extenders).

Evaporation to dryness of the liquid portion of the product tested, obtained by centrifuging. Preparation of the residue for analysis by dry ashing and extraction of the ash with acid.

NOTE — 0,07 mol/l hydrochloric acid solution is chosen as an approximation to the acid strength in the stomach. It has been agreed that the mass ratio between the pigments and the hydrochloric acid solution shall be 1 : 15, except in the case of soluble lead where it shall be 1 : 1 000.

5 Sampling

Take a representative sample of the product to be tested by the method specified in ISO 1512.

6 Separation of the pigments and extenders from the paint sample

6.1 Preparation of the test sample

Examine and prepare the sample for testing as specified in ISO 1513. If any skin is present, remove it as far as possible. Thoroughly stir the sample and, if necessary, pass it through a sieve of nominal aperture 150 µm to remove any remaining skin and other extraneous matter.

6.2 Reagents

Select a solvent that effects the optimum separation of the pigments and extenders.

WARNING — In selecting the solvent to be used, possible toxic and other hazards and necessary precautions should be taken into consideration.

Examples of suitable solvents or solvent mixtures are as follows :

— for solvent-thinned paints, printing inks and similar products (method A) :

Toluene/ethanol (4 + 1) (for air-drying paints)
Xylene/1-butanol (9 + 1) (for stoving paints)
Toluene (for chlorinated rubber paints)
Butanone (for nitrocellulose paints)

— for emulsion paints based on aqueous polymer dispersions (method B) :

Acetone
Chloroform, freshly distilled
Tetrahydrofuran

— for plastisols and organosols based on polyvinyl chloride (PVC) and its copolymers and for paints based on non-aqueous polymer dispersions (method C) :

Tetrahydrofuran
Cyclohexanone
Cyclopentanone

6.3 Apparatus

Ordinary laboratory apparatus and glassware, and in particular

6.3.1 Suitable laboratory centrifuge, with tubes of inert material of capacity 50 or 100 ml. A centrifuge, capable of imparting a relative centrifugal acceleration of 100 km/s^2, is preferred.

6.3.2 Air-ventilated oven, capable of being maintained at 105 ± 2 °C.

6.4 Procedure

6.4.1 Method A (for solvent-thinned paints, printing inks and similar products)

Weigh, to the nearest 0,01 g, a number (see note 1) of centrifuge tubes (6.3.1). Add 10 to 20 g (see note 2) of the prepared sample to each tube, taking care to avoid contamination of the walls and lip of the tube. *Immediately* weigh the tubes and contents to the nearest 0,01 g. Approximately half fill the tubes with the selected solvent (6.2) and stir thoroughly using a glass rod. Wash the glass rods thoroughly with the solvent, adding all washings to the appropriate tube. Balance the opposing centrifuge tubes to within 0,1 g by adding further solvent, taking care to preserve an adequate working level. Centrifuge until there is a complete separation into a clear liquor and a pigment cake. Decant the supernatant liquor from all the tubes comprising a "set" (see note 1) into a stoppered glass container of approximate capacity 1 litre.

Add further solvent to each tube and mix thoroughly as specified above, taking care to completely disperse the pigment cake. Repeat the centrifuging and transfer of liquor. Repeat the addition of solvent, centrifuging and transfer of liquor for a third time, taking special care, as before, to disperse the pigment cake. As a final treatment for the pigment cake, use acetone in place of the selected solvent, to assist rapid drying. Add the acetone and mix, taking special care to disperse the whole pigment cake. Centrifuge and transfer the liquor as before to the stoppered glass container. Retain the container with the combined extracts for the procedure specified in clause 8.

After ensuring the evaporation of excess acetone, place the centrifuge tubes in the oven (6.3.2), maintained at 105 ± 2 °C, for a minimum period of 3 h. Remove, transfer to a desiccator, allow to cool to ambient temperature and weigh each tube and contents to the nearest 0,01 g. Return the tubes and contents to the oven for a minimum period of 1 h, allow to cool to ambient temperature in the desiccator and re-weigh. Repeat the heating, cooling and weighing operations until constant mass is reached; that is, until the results of two consecutive weighings do not differ by more than 0,01 g.

Calculate the pigment content of the paint as a percentage by mass of the paint sample (see note 3).

NOTES

1 Subsequent acid extractions should be carried out in duplicate and therefore sufficient paint and tubes should be taken so that at least 11 g of pigment is obtained. The number of tubes (usually four) required for each sample is referred to as a "set".

2 The mass of paint taken is dependent upon the capacity of the centrifuge tubes employed and on the expected pigment content of the paint.

3 If the dried pigment cake cannot be broken easily at the end of the extraction, this indicates that the binder has not been completely extracted.

6.4.2 Method B (for emulsion paints based on aqueous polymer dispersions)

Carry out the separation as specified in 6.4.1, but use acetone for the first and final treatment and the selected solvent for three intermediate treatments of the pigment cake.

6.4.3 Method C (for plastisols and organosols based on polyvinyl chloride (PVC) and its copolymers and for paints based on non-aqueous polymer dispersions)

Carry out the separation as specified in 6.4.1 but using a mass of sample such that it may be diluted with solvent in the ratio of 1 : 10. This ratio is necessary to obtain a practical sedimentation rate. Stir very thoroughly for sufficient time to convert the polymer from the dispersed to the dissolved state. Gentle heating may also be necessary.