



Guidance document for the paints industry on the determination of Semi-Volatile Organic Compounds (SVOC) using ISO 11890-2 (2013) (extending its scope)

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Recommandations de l'industrie de la peinture pour l'utilisation de la norme ISO 11890-2 (2013) afin d'identifier les Composés Organiques Semi Volatiles (COSV) (par extension du cadre d'application)

Background

The EU Ecolabel criteria for paints and varnishes (Commission decision 2014/312/EU and as amended by Commission Decision 2015/886/EU) refer to the use of the ISO 11890-2 testing standard for SVOC measurement in Criterion 4, Content of Volatile and Semi-volatile Organic Compounds (VOCs, SVOCs):

The applicant shall provide for the SVOC content of the ready to use product either a test report using the method given in ISO 11890-2 or a declaration of compliance supported by calculations based on the paint ingredients and raw materials. The test shall be carried out using the analytical system as identified in the Criteria User Manual. At the request of a Competent Body applicants may be required to validate calculations using the specified test method.

The scope of ISO 11890-2 does not currently cover SVOC measurement, meaning that laboratories contacted by applicants that decide to use SVOC measurement may interpret SVOC content testing in a different manner. This will lead to systematically different test results, which in turn will create problems of comparability between statements of compliance.

ISO 11890-2 is now proposed to be amended for its scope by ISO/TC 35 to include SVOC determination. As the standardisation process can take some time, this paper aims at providing recommendations to laboratories delivering SVOC test data based on ISO 11890-2, but extending the scope of applicability of that standard, until the standard has been modified to include SVOC measurement. These test data could then be used in the application dossier for the EU Ecolabel.

Scope:

This guidance document interprets the specifications of ISO 11890-2 to allow the running of a test to quantify paint SVOC content, either alone or in one run together with an ISO 11890-2 VOC test, so as to evaluate compliance with the requirements of the EU Ecolabel. This guidance should therefore be read alongside ISO 11890-2, but with the modified sample preparation method, apparatus and parameters specified taking precedence.

Sample preparation:

An organic solvent suitable for diluting the sample shall be used. It shall have a purity of at least 99% by mass. The recommended dilution solvent is methanol 100%. If necessary, the sample can be stirred during 30 minutes with application of ultrasound in order to achieve a

homogenous liquid phase, or by mechanically stirring during two hours followed by centrifugation or a filtration step using a PTFE filter type for paints containing large, undissolved particles. In the case that a homogenous liquid phase cannot be achieved using methanol 100% then another suitable dilution solvent, such as acetonitrile or tetrahydrofuran, shall be used.

Note:

The marker compounds to be used are n-tetradecane (n-C14) and n-Docosane (n-C22). It may be necessary to prepare a marker solution containing these compounds in acetone due to the limited solubility of n-Docosane in acetonitrile.

Apparatus:

Capillary column:

- The preferred choice of column shall be one made of fused silica coated with 5% phenyl / 95% dimethyl polysiloxane (slightly polar type, DB5 or equivalent).
- A column coated with 100% dimethyl polysiloxane (non-polar type, DB1 or equivalent) may be used if it can be shown to perform better for predominantly non-polar paint ingredients.

Note:

A suitable combination of column length (30m or 60m), diameter and temperature programme shall be selected such that compounds in the sample and the markers elute in the order of their increasing boiling points. A column length of 60m may be used to improve the elution order for the slightly polar column type.

Oven:

- Oven initial temperature: between 40 and 100°C
- Isothermal holding time: between 2 and 5 min
- Heating rate: between 3 and 20°C/min
- Oven final temperature: between 280 and 325°C
- Isothermal holding time: >2min
- Flow in the column: between 1 and 2 ml/min

Detector:

- Identification by mass spectrometer
- Quantification by flame ionization detector (FID)
- FID detector temperature: Final oven temperature or higher

Carrier gas:

- helium

Hot injection system:

- injector temperature : between 250 and 280°C
- injection volume: between 1 and 2 µl



Calibration:

- the preferred internal standard for quantification of SVOC peaks shall be n-tetradecane (n-C14)
- An alternative internal standard, 1,2-diethoxyethane (also named ethylene glycol diethyl ether) can be used in order to achieve improved recovery values when analysing water-based paints.

Note:

If the calibration procedures are run in an appropriate manner the selection of the internal standard should have no impact on the test result. However, it is important to ensure that the internal standard does not overlap or hide any peaks arising from the sample itself. It must therefore show a complete separation from other peaks in the chromatogram. A large choice of internal standards is thus possible but internal standards having very low boiling points (e.g. acetone...) or very high boiling points (C22 and more...) must be excluded to avoid any discriminatory phenomenon in the injector.

- All SVOCs shall be identified as far as achievable, and then quantification shall be performed with their authentic calibration standards, as specified for VOCs in ISO 11890-2, or via their relative response factors.
- Remaining unknown SVOC peaks shall be quantified using the response factor of diethyl adipate, expressed in diethyl adipate equivalents.