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**Fine bubble technology — Cleaning applications —**

Part 2:

**Test method for cleaning machine-oil stained surfaces of machined metal parts**

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 281, *Fine bubble technology*.

A list of all parts in the ISO 21256 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

Mineral oil is used in manufacturing processes of industrial products, for applications such as cutting, lubrication and cooling. Oil remaining on the surface of products has to be cleaned during operation and at the end of the process. The main cleaning solvent fluids used in these industries are solvent based (e.g. hydrocarbon detergent liquids, bromine-containing detergents, and alkali detergents), despite it being well known that such detergents can cause environmental pollution such as ozone layer depletion, water pollution, etc.

As an ecological alternative, fine bubble technology, which does not use detergents, is becoming popular. Use of fine bubble technology improves resource sustainability, energy saving and safety, as it uses less water and no chemical substances.

This document is intended to provide a procedure that can help demonstrating the cleaning performance of such technology, so that different methodologies can be compared.

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# Fine bubble technology — Cleaning applications —

## Part 2:

# Test method for cleaning machine-oil stained surfaces of machined metal parts

## 1 Scope

This document specifies a test method for removal of machine oil stain from a noncorrosive metal surface using fine bubble water. A test is provided to show the comparative cleaning advantage of adding fine bubbles to the water.

## 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 edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 20480-1, *Fine bubble technology — General principles for usage and measurement of fine bubbles — Part 1: Terminology*

ISO 20480-2, *Fine bubble technology — General principles for usage and measurement of fine bubbles — Part 2: Categorization of the attributes of fine bubbles*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20480-1 and ISO 20480-2 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 3.1

#### oil cleaning index

number representing the residual oil removed from the test surface after cleaning

## 4 Principle

Evaluate the performance of test oil removal by fine bubble water by measuring the amount of residual oil with fine bubble water applied and not applied. Evaluation of the performance is made by comparing the oil cleaning index of test oil on the surface of the test piece with fine bubble water applied to a control sample using ordinary water.

The residual oil is measured using an oil content meter based on infrared spectroscopy.

## 5 Reagents and materials

5.1 **Water**, which usually has been treated and conforms to grades of water given in ISO 3696.

5.2 **Test oil**. The test oil used to coat the test surface shall be triolein or an oil agreed between the parties concerned. An example of infrared spectrum of the test oil applied for oil content meter is given in [Annex B](#).

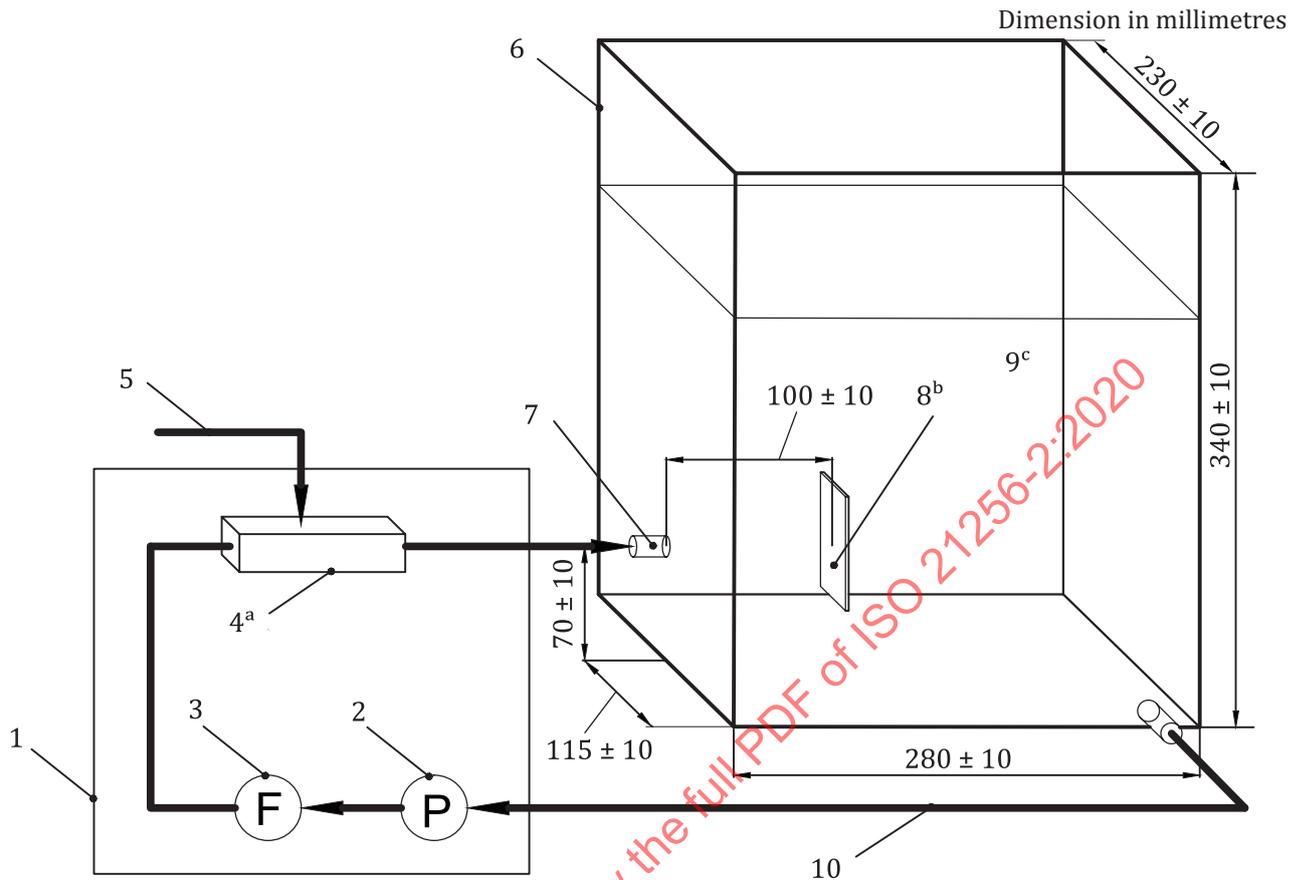
5.3 **Extraction solvent**, containing 65 % to 75 % (mass fraction) of extracted chloro-tri-fluoro-ethylene dimer ( $\text{Cl}(\text{CF}_2\text{CDCl})_2\text{Cl}$ ) and 25 % to 35 % (mass fraction) of extracted chloro-trifluoro-ethylene trimer chloro-tri-fluoro-ethylene tetramer [ $\text{Cl}(\text{CF}_2\text{CFCl})_3\text{Cl}$ ]. See Reference [1].

## 6 Apparatus

### 6.1 Test equipment

[Figure 1](#) shows a typical example of equipment, where the difference in result between the operation with fine bubble generating system on and off can be compared. The dimensions of the cleaning tank shall be as given in [Figure 1](#).

- a) The inside dimension of the cleaning tank shall be  $(280 \pm 10)$  mm  $\times$   $(230 \pm 10)$  mm  $\times$   $(340 \pm 10)$  mm.
- b) The inside diameter of the cleaning stream outlet shall  $(13 \pm 0,1)$  mm.
- c) The centre of the test piece shall be placed at  $(100 \pm 10)$  mm distance from the cleaning stream outlet, and  $(115 \pm 10)$  mm distance from the side wall of the tank.
- d) The centre of the test piece shall be placed on the axis of the outlet.
- e) The position of the test piece shall be such that the cleaning stream hits normal to its surface.
- f) The streamline at the outlet shall be parallel to the axis of the outlet.
- g) The material of the tank shall be a transparent heat-resistance resin.
- h) Test equipment and accessories shall be cleaned off residual oil after test for each test piece. Fresh water shall be used for testing the subsequent test piece.



**Key**

- 1 fine bubble supply system
- 2 pump
- 3 filter
- 4 fine bubble generating system ON/OFF
- 5 flow rate of suction air
- 6 cleaning tank
- 7 cleaning stream outlet
- 8 test piece
- 9 cleaning water
- 10 flow rate of water
- a The fine bubble generator shall be able to turn ON/OFF.
- b The position of the test piece shall be such that the cleaning stream hits normal to its surface.
- c Quantity of water shall be 15 l.

**Figure 1 — Test equipment**

**6.2 Oil content meter**

Measuring instrument based on infrared spectroscopy, which is commercially available. Refer to the manufacturer instructions for the sampling procedure, sample preparation and detection limit<sup>[1][2]</sup>.

The oil content meter should preferably be calibrated.

An example of infrared spectrum of the test oil applied for oil content meter calibration is given in [Annex B](#).

## 7 Preparation of test pieces

### 7.1 Test pieces

The dimensions of test pieces shall be length of  $(75 \pm 1)$  mm and width of  $(20 \pm 1)$  mm with thickness of  $(1,5 \pm 0,5)$  mm for easy treatment. Material of the test pieces shall be 4301-304-00-I that is prescribed in ISO 15510, and lapped surface with roughness  $Ra$  50 nm to 100 nm.

### 7.2 Method for depositing oil stain on the test pieces

The method for depositing test oil on the test pieces shall be as follows.

- a) Clean all surfaces of the test pieces using a waste cloth with propanol-2-ol as recommended by ISO 6353-3<sup>[5]</sup>.
- b) Then, clean all surfaces of the test pieces using clean waste cloth with extraction solvent (5.3).
- c) Dry the surface of the test pieces completely.
- d) Deposit the test oil on the front surface of test pieces. The surface density of the test oil shall be from  $50 \mu\text{g}/\text{cm}^2$  to  $150 \mu\text{g}/\text{cm}^2$  with uniformity less than  $20 \mu\text{g}/\text{cm}^2$ . An example of a method for oil depositing is given in Annex A.
- e) Fix the test oil on the front surface by heating at a temperature of  $50 \text{ }^\circ\text{C}$  for 30 min.

## 8 Procedure

### 8.1 Test procedure

Three test pieces shall be prepared according to the process described in Clause 7, with the uniformity of surface density of test oil ensured.

Wearing rubber gloves is necessary, whenever handling the test pieces, in order not to attach any sebum to the test piece.

- a) Dissolve away the test oil on the front surface of the first test piece by using extra solvent and then measure the quantity of oil deposited using the oil content meter, according to the procedure described 8.3. This provides a base line of the oil deposition.
- b) Clean the test oil on the front surface of the second test piece with plain water using the equipment and the conditions described in 8.2. Then, dissolve away the residual test oil and measure the residual quantity with the oil content meter according to the procedure described in 8.3.
- c) Clean the test oil on the front surface of the third test piece with fine-bubble enhanced water using the equipment and procedure described in 8.2. Then, dissolve away the residual test oil and measure the residual quantity with the oil content meter according to the procedure described in 8.3.
- d) Record the three surface concentrations of test oil given by normalizing the quantities of test oil by the surface area of the test pieces.

### 8.2 Conditions of cleaning test

The following conditions shall apply for cleaning the test pieces, using the equipment described in 6.1.

- a) Water temperature shall be less than  $(40 \pm 1) \text{ }^\circ\text{C}$ .
- b) The flow rate of cleaning liquid at the outlet shall be  $(8,0 \pm 0,2) \text{ l}/\text{min}$ .
- c) Duration of the cleaning operation shall be  $(5,0 \pm 0,1) \text{ min}$ .

- d) For the duration of the cleaning time, a continuous flow of water shall be circulated with a pump to impact upon the test plate.
- e) The flow of cleaning liquid shall be in a steady state.
- f) The tank is to be emptied and the walls wiped to remove any traces of oil prior to re-filling with the prescribed quantity of water. The water is the same type of water as that used for fine bubble generation.
- g) The clean tank shall be filled with the prescribed volume of water. Volume of water shall be  $(15,0 \pm 0,5)$  l.

### 8.3 Measurement of test oil on the test piece

The measurement method of the total test oil quantity on the front surface of the test piece is as follows.

- a) Wipe side and reverse surfaces (all surfaces except the front surface) using a clean waste cloth with extraction solvent.
- b) Pour the extraction solvent in a beaker with diameter larger than the size of the test piece, and half-fill the beaker. Weigh the amount of the extraction solvent and record the volume after normalizing with the density of the extraction solvent.
- c) Clean a pipet with extraction solvent and then dry it.
- d) Set a surface of the test piece vertical. Rinse out the vertical surface of the test pieces by down-flow of the extraction solvent from the pipet, with all extra solvent pouring into the beaker.
- e) Repeat the process c) for all surfaces of the test piece. Ensure no test oil is left.
- f) Sample the extraction solvent in the beaker, fill it to the measurement cell of the oil content meter and measure it.
- g) Calculate the total test oil quantity in the beaker from the reading of the oil content meter, the volumes of the measurement cell and the extraction solvent in a).

It is possible to use organic substances such as surfactant or detergent for assistance in the test procedure. Record the three surface concentrations of test oil given by normalizing the quantities of test oil by the surface area of the test pieces.

## 9 Calculation of oil cleaning index of oil stain

The oil cleaning indexes of oil stain,  $E_a$  and  $E_b$ , are calculated for 8.1 b) and 8.1 c) by [Formulae \(1\)](#) and [\(2\)](#).

$$E_a = \frac{\sigma_0 - \sigma_a}{\sigma_0} \quad (1)$$

$$E_b = \frac{\sigma_0 - \sigma_b}{\sigma_0} \quad (2)$$

where

- $\sigma_0$  is the surface concentration, [8.1 d)], of the test oil on the front surface of the test piece by the process in 8.1 a);
- $\sigma_a$  is the surface concentration, [8.1 d)], of the test oil on the front surface of the test piece by the process in 8.1 b) (using plain water);
- $\sigma_b$  is the surface concentration, [8.1 d)], of the test oil on the front surface of the test piece by the process in 8.1 c) (using fine bubble water).

## 10 Test report

The test report shall contain the following information:

- a) test date;
- b) temperature;
- c) test conditions e.g. flow rate of cleaning liquid and material of test piece, parameters of the fine bubble generating system [product name, flow rate of suction air, flow rate of circulation water, bubble size index and number concentration index (when they are available), etc.];
- d) test result (oil cleaning index of the test oil).

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## Annex A (informative)

### Example of a method for depositing oil stain on the test pieces

#### A.1 General

This annex describes a cleaning test procedure and its result on practical test pieces. In this example, the test oil is deposited by spraying a solvent containing the dissolved test oil over the test piece. See 7.2 d).

#### A.2 Preparation

##### A.2.1 Test oil

The test oil (5.2) is mixed with propanol-2-ol (isopropyl alcohol) to a concentration of 0,5 g soluble in 9 ml and fed to an air brush. The viscosity of the mixture becomes less than  $(10 \times 10^{-3})$  Pa·s.

Propanol-2-ol is a volatile reagent prescribed by ISO 6353-3<sup>[3]</sup>.

##### A.2.2 Test piece

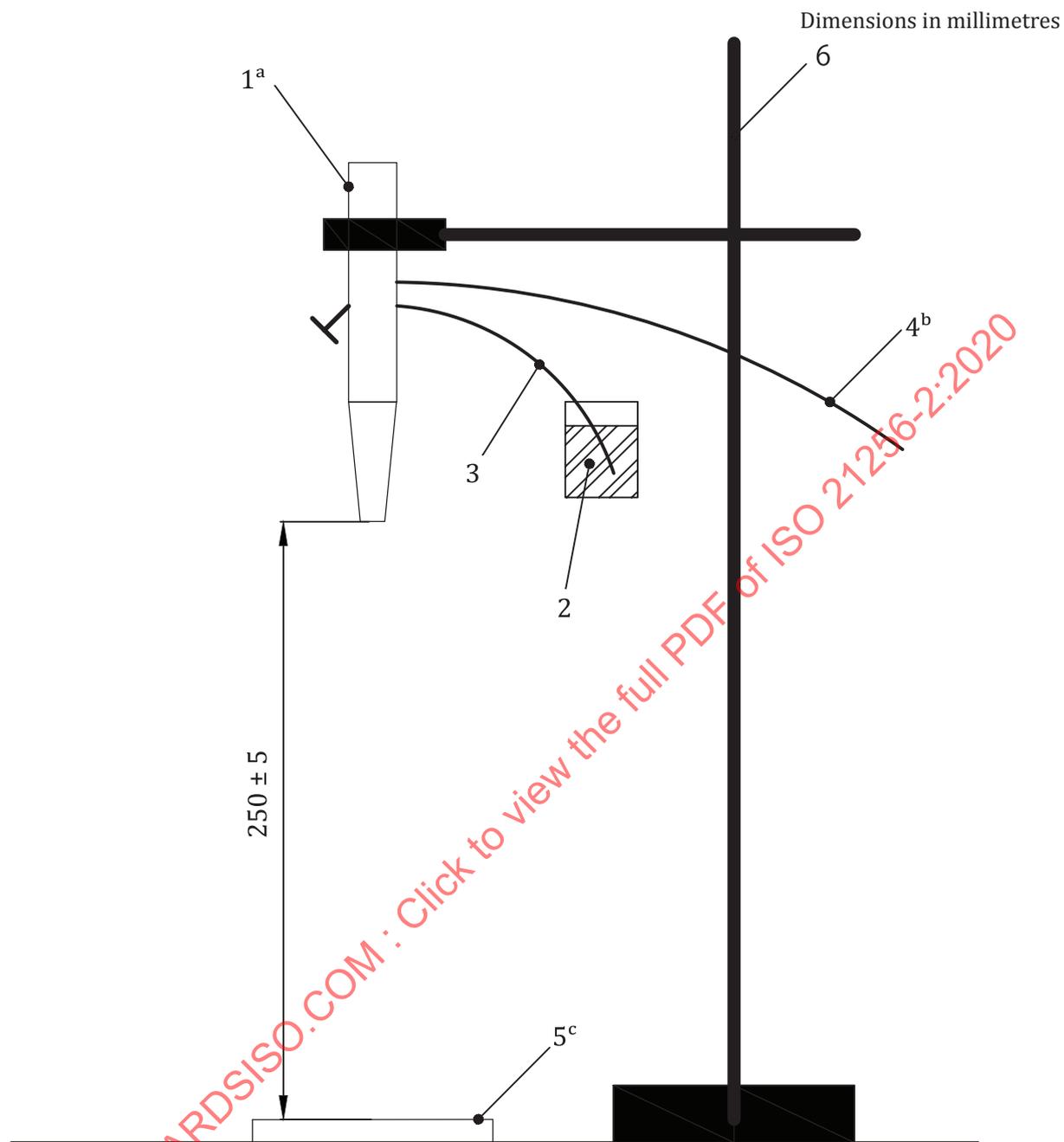
The test piece is made from material 4301-304-00-1 that is prescribed in ISO 15510, with surface roughness  $R_a$  65 nm. The dimensions of the test piece are  $(75,0 \times 20,0 \times 1,5)$  mm.

The procedure in 7.2 a), b) and c) is followed.

#### A.3 Method for depositing oil stain on the test pieces

- a) The front side of the test piece is placed on top. The outlet of the air brush is directed vertical down and set 250 mm right above the centre of the surface.
- b) The air brush is operated with pressure 60 kPa. The spraying time is subject to the surface concentration of the oil stain as specified in 7.2 d) and calibrated as shown in A.4.
- c) Maintain the test piece at 50 °C for 30 min as specified in 7.2 e), to fix the oil stain on the surface of the test piece.

Figure A.1 shows the process described in a) and b) above.



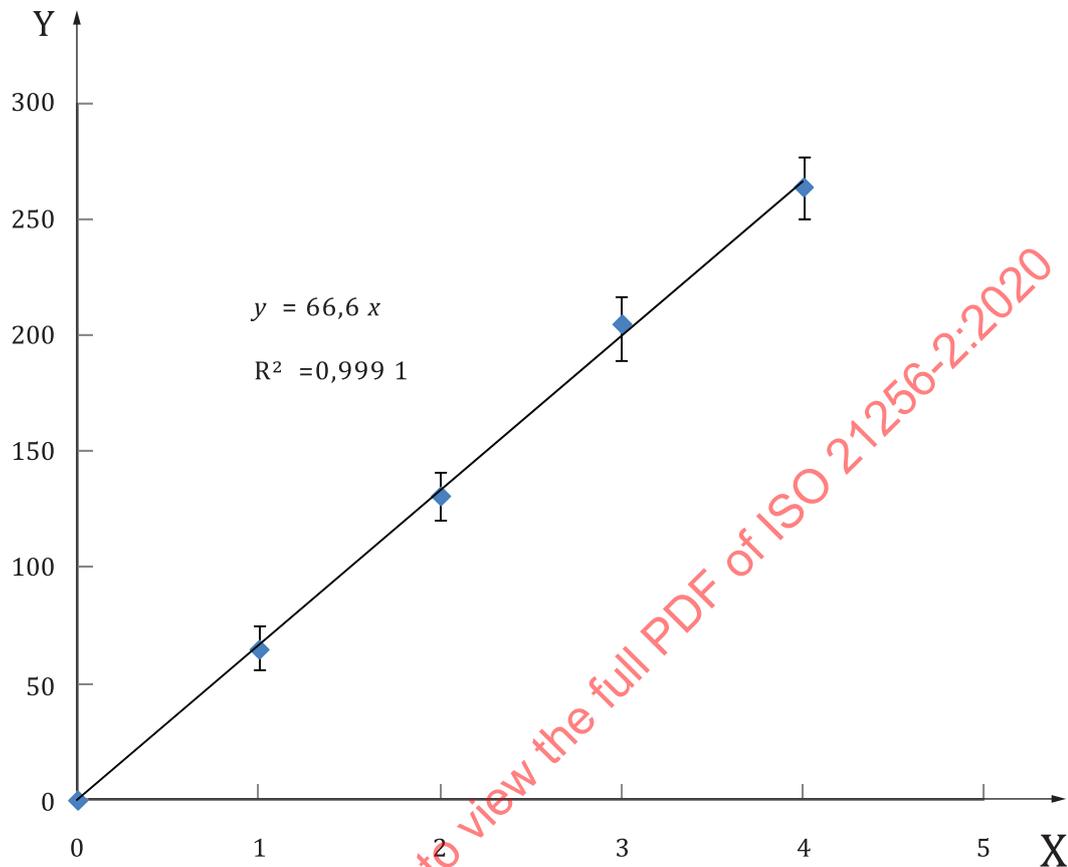
**Key**

- 1 air brush
- 2 oil stain
- 3 tube
- 4 tube
- 5 test piece
- 6 stand
- a The pressure of the air brush shall be 60 kPa and fixed to the stand.
- b The tube shall be connected to the compressor.
- c The test piece shall be set with target cleaning surface on top.

**Figure A.1 — Deposition of the test oil on the test piece**

## A.4 Calibration of spraying time

Figure A.2 shows an example of calibration result for determining the spraying time.



### Key

- X spraying time, expressed in seconds  
 Y oil density on the target surface of the test piece, expressed in  $\mu\text{g}/\text{cm}^2$

Figure A.2 — Example of calibration result

## A.5 Cleaning test

The procedure for cleaning test in 8.1 is applied to the test pieces. Test conditions are as follows.

- Water temperature is 40 °C.
- The flow rate of cleaning liquid at the outlet is 8 l/min.
- The flow rate of suction air of the fine bubble generating system is 0,8 l/min.
- Duration of the cleaning operation is 5 min.
- The test piece is fixed in the cleaning tank by a method and an appliance such as in Figure A.3.
- The test oil is triolein.
- The fine bubble generating system is a venturi tube or injector.

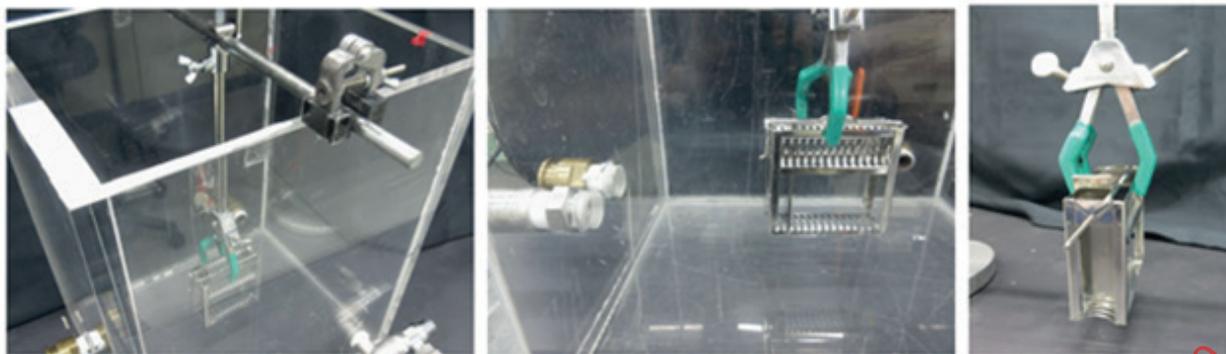


Figure A.3 — Method and appliance for fixing a test piece

## A.6 Result of the cleaning test

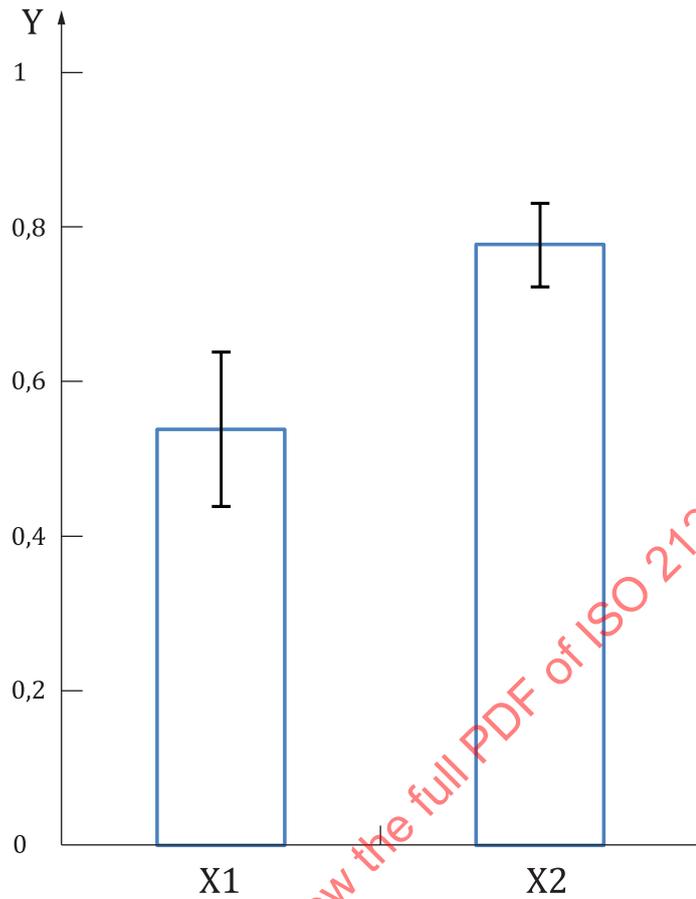
The surface concentration of test oil  $\sigma_0$ ,  $\sigma_a$ , and  $\sigma_b$  for the procedure in 8.1 a) 8.1 b) and 8.1 c), namely those for before cleaning, using plain water, and using fine bubble water, respectively are shown in Table A.1. The results for successive five readings on the output of oil content meter are averaged.

The oil cleaning indices are calculated according to Formulae (1) and (2) and illustrated in Figure A.4.

An example of test report (see Clause 10) is shown in Table A.2.

Table A.1 — Result of the cleaning test

	Before cleaning	Using plain water	Using fine bubble water
Surface concentration $\mu\text{g}/\text{cm}^2$	108,8	40,9	21,5
	118,7	55,5	27,3
	127,3	67,5	30,1
Average of surface concentration $\mu\text{g}/\text{cm}^2$	118,3	54,6	26,3
Oil cleaning index	—	0,538	0,778



The error bars on the bar graphs indicate a double value of the standard deviation.

**Key**

- X1 water
- X2 fine bubble water
- Y oil cleaning index

**Figure A.4 — Result of cleaning test**