

## Non-Destructive Test Field Metallography

### Procedure

This Standard replaces and cancels its previous revision.

The CONTEC - Authoring Subcommittee provides guidance on the interpretation of this Standard when questions arise regarding its contents. The Department of PETROBRAS that uses this Standard is responsible for adopting and applying the sections, subsections and enumerates thereof.

**Technical Requirement:** A provision established as the most adequate and which shall be used strictly in accordance with this Standard. If a decision is taken not to follow the requirement ("non-conformity" to this Standard) it shall be based on well-founded economic and management reasons, and be approved and registered by the Department of PETROBRAS that uses this Standard. It is characterized by imperative nature.

**Recommended Practice:** A provision that may be adopted under the conditions of this Standard, but which admits (and draws attention to) the possibility of there being a more adequate alternative (not written in this Standard) to the particular application. The alternative adopted shall be approved and registered by the Department of PETROBRAS that uses this Standard. It is characterized by verbs of a nonmandatory nature. It is indicated by the expression: **[Recommended Practice]**.

Copies of the registered "non-conformities" to this Standard that may contribute to the improvement thereof shall be submitted to the CONTEC - Authoring Subcommittee.

Proposed revisions to this Standard shall be submitted to the CONTEC - Authoring Subcommittee, indicating the alphanumeric identification and revision of the Standard, the section, subsection and enumerate to be revised, the proposed text, and technical/economic justification for revision. The proposals are evaluated during the work for alteration of this Standard.

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## CONTEC

Comissão de Normalização  
Técnica

## SC - 27

Non-Destructive Tests

### Introduction

*PETROBRAS Technical Standards are prepared by Working Groups - WG (consisting specialized of Technical Collaborators from Company and its Subsidiaries), are commented by Company Units and its Subsidiaries, are approved by the Authoring Subcommittees - SCs (consisting of technicians from the same specialty, representing the various Company Units and its Subsidiaries), and ratified by the Executive Nucleus (consisting of representatives of the Company Units and its Subsidiaries). A PETROBRAS Technical Standard is subject to revision at any time by its Authoring Subcommittee and shall be reviewed every 5 years to be revalidated, revised or cancelled. PETROBRAS Technical Standards are prepared in accordance with PETROBRAS Technical Standard [N-1](#). For complete information about PETROBRAS Technical Standards see PETROBRAS Technical Standards Catalog.*

## Foreword

This Standard is the English version (issued in 08/2012) of PETROBRAS N-2484 REV. C 04/2011. In case of doubt, the Portuguese version, which is the valid document for all intents and purposes, shall be used.

## 1 Scope

1.1 This Standard specifies the required conditions for the field metallography test using field microscopy or metallographic replica. Conditions for macrographic examination are also defined.

1.2 This Standard applies to the non-destructive metallographic test in process equipment and metallic structures.

1.3 This Standard applies to procedures started as from the date of its edition.

1.4 This Standard includes only Technical Requirements.

## 2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document applies.

INMETRO VIM:2008 – Vocabulário Internacional de Metrologia (First Brazilian Edition of VIM 2008);

ASTM [E 3:2001](#) - Standard Guide for Preparation of Metallographic Specimens;

ASTM [E 7:2003](#) - Standard Terminology Relating to Metallography;

ASTM [E 340:2000](#) - Standard Test Method for Macroetching Metals and Alloys;

ASTM [E 407:2007](#) - Standard Practice for Microetching Metals and Alloys;

ASTM [E 1351:2001](#) - Standard Practice for Production and Evaluation of Field Metallographic Replicas;

ASTM [E 1558:2009](#) - Standard Guide for Electrolytic Polishing of Metallographic Specimens.

**NOTE** For documents referred in this Standard and for which only the Portuguese version is available, the PETROBRAS department that uses this Standard should be consulted for any information required for the specific application.

## 3 Terms and Definitions

For the effects of this document, terms and definitions of ASTM [E 7:2003](#), supplemented or changed by 3.1 to 3.6 and INMETRO [VIM:2008](#).

### 3.1

#### **macrography**

method for observation of metallographic structures after adequate preparation and attack, with the naked eye or using an optical instrument with up to 50X amplification

**3.2**
**field metallography**

a non-destructive procedure to evaluate the microstructure of metallic materials in available surfaces of equipment or accessories at the site of installation

**3.3**
**replica metallography**

non-destructive procedure to register and maintain the metallographic sample print on a negative plastic film

**3.4**
**field microscopy**

a method used for observation of a metallographic structure using a portable microscope, after adequate preparation of the surface, with or without attack

**3.5**
**mechanical polishing**

a procedure used to get a flat, polished and specular surface, with the help of a mechanical apparatus

**3.6**
**metallization**

covering the metallographic replica film with the help of a vacuum chamber to get a sharper contrast for optical microscopic (OM) observation and conductivity for Scanning Electron Microscope (SEM) observation

**4 General conditions**
**4.1 Tools and Consumables**
**4.1.1 Surface Preparation:**

- a) roughhewing tool (grinding machine, micro-milling machine, etc.);
- b) variable rotation sander;
- c) 50, 80, 120, 220, 320, 400, 600 and 1 200 grit sandpaper;
- d) felt or cloth for mechanical polishing;
- e) polishing paste or suspension: alumina or diamond;
- f) polishing lubricant;
- g) electrolytic polishing tool;
- h) electrolytic polishing reagent, according to Table 1, considering Health, Safety and Environment (HSE) precautions.

**Table 1 - Metallographic Reagent for Metal and Metal Alloys Electrolytic Polishing Used in Oil Industry**

<b>General use reagent</b>	78 mL perchloric acid (70%); 120 mL distilled water; 700 mL ethanol; 100 mL butylglycol or glycerol.
<b>Specific use reagents</b>	As per ASTM E 1558:2009.
<b>NOTE</b>	For safety reasons, when preparing the general use reagent, first dilute the acid in distilled water. Mix the butylglycol with the ethanol separately. Finally, dilute the acid solution in the ethanol with butylglycol or glycerol mix.

4.1.2 For inspection:

- a) reagents with chemical and electrolytical etching, according to 4.5;
- b) material for replica acquisition, when applicable;
- c) bench metallographic microscope for replica analysis;
- d) portable metallographical microscope for *in situ* analyses;
- e) SEM for special analyses;
- f) stereoscopic magnifying glass for macrographic analyses;
- g) metallization chamber for replica coating;
- h) photography camera for registration.

## **4.2 Execution Conditions**

4.2.1 The procedure for test execution shall be prepared according to 4.7.

4.2.2 Special care shall be taken when handling electrolytic polishing and etching solutions, due to their corrosive potential, toxicity and explosion hazard. Use Safety Data Sheets for Chemicals (SDS) when handling chemical substances. These precautions shall be considered in the procedure prepared in accordance with 4.2.1.

4.2.3 During the test, keep the surroundings free from dust and protected from strong air currents. In case the test is performed in open air, adequate protection shall be provided for those involved.

## **4.3 Selection of place for test execution**

4.3.1 Areas for the metallographic non-destructive test shall be mapped beforehand, taking into consideration the test purpose.

4.3.2 The tests should be executed preferably in places selected according to other preliminary tests performed (magnetic particle, macrography, liquid penetrant).

## **4.4 Surface Preparation**

### **4.4.1 Initial roughing**

Performed with the abrasive tool and / or coarse sandpapers, for example, 50, 80, and 120 grit, for the removal of decarbonized layers, oxides and for surface smoothing.

### **4.4.2 Sanding**

Performed with fine grit sanders, with 150, 220, 320, 400 e 600 grit, in this order. The sanding orientation in each stage shall differ in 90° in relation to the previous stage, and the surface shall be flushed clean after each stage.

### **4.4.3 Polishing**

Surface preparation for the etching can be performed by mechanical or electrolytic polishing, described in 4.4.3.1 and 4.4.3.2.

#### 4.4.3.1 Mechanical Polishing

This shall be performed as below:

- a) polishing with a 15  $\mu\text{m}$ , 6  $\mu\text{m}$ , 3  $\mu\text{m}$  and 1  $\mu\text{m}$  or 5  $\mu\text{m}$ , 1  $\mu\text{m}$ , 0.3  $\mu\text{m}$  and 0.05  $\mu\text{m}$  alumina;
- b) the polishing orientation shall differ 90° in each stage, in relation to the previous stage;
- c) the surface shall be washed clean with alcohol or acetone and dried with hot air blower after each stage.

NOTE For specific conditions regarding the mechanical preparation of hard or excessively soft surfaces, ASTM E 3:2001 shows specific procedures for each case.

#### 4.4.3.2 Electrolytic Polishing

This should be performed as below:

- a) electrolytic polishing using a specific construction tool, in compliance with 4.1.1;
- b) it is recommended that adjustment parameters for electrolytic polishing be previously tested with similar material, before their use in field. **[Recommended Practice]**.

NOTE 1 Mechanical or electrolytic polishing shall be performed so as to obtain a surface free of imperfections, scratches, corrosion pit and other defects that may mask the true characteristics of the microstructure.

NOTE 2 Electrolytic polishing is used preferably when a faster service is required and when environmental conditions are not in compliance with 4.2.3.

NOTE 3 Where the electrolytic polishing can result in incorrect interpretation of microstructure, mechanical polishing shall be used.

NOTE 4 It is recommended that the superficial finishing between stages and at the procedure's completion be inspected with a field microscope. A 100X amplification is enough to this end. **[Recommended Practice]**.

#### 4.5 Etching

Chemical etching shall be done using reagents according to ASTM E 407:2007 or ASTM E 340:2000, aiming at revealing the desired structural aspects, with due observation of HSE precautions.

#### 4.6 Precautions for Handling and Using Chemical Reagents

Additionally to HSE precautions recommended in ASTM E 407:2007, ASTM E 340:2000 and ASTM E 1558:2009, the following shall be considered:

- a) reagents shall not contain in its composition:
  - hydrofluoric acid - HF;
  - cyanides in general, such as: KCN, NaCN;  $\text{K}_3\text{Fe}(\text{CN})_4$ ;
- b) never dispose of concentrate perchloric acid directly with organic substances (alcohol, wood, paper, acetic acid,  $\text{H}_2\text{SO}_4$ ,  $\text{H}_3\text{PO}_4$ , and glycerol). Any perchloric acid mix in organic solutions shall be done with acid previously diluted in water;
- c) avoid the use of methanol.

#### 4.7 Procedure

4.7.1 The following items shall be performed in the following sequence:

- a) purpose:

- b) reference standards;
- c) HSE warnings and requirements;
- d) equipment or accessory to be inspected;
- e) tools and consumables used;
- f) polishing technique used, as well as the reagent used, when applicable;
- g) specification of the reagent used for etching;
- h) method used for metallographic structure observation;
- i) results registration system, including the report form;
- j) identification and traceability system;

4.7.2 The issuer's name (PETROBRAS unit or contractor's company), number of the procedure and revision indication and issuance date are required.

4.7.3 The description of a system for registration of results may not be needed in the inspection procedure, at the discretion of PETROBRAS, when the Contractor (PETROBRAS unit or the Company performing the services) has an established system meeting the requirements set forth in 5.3.1. in this quality assurance system.

## **5 Specific Conditions**

### **5.1 Metallographic Replica Testing**

All metallographic replicas shall be performed according to ASTM [E 1351:2001](#).

### **5.2 Field Microscopy Evaluation**

5.2.1 The area to be prepared for a field microscopy evaluation shall meet the inspection requirements, and a 12 mm x 18 mm prepared area is satisfactory.

5.2.2 Field microscopy evaluation shall be performed immediately after the surface preparation and the metallographic etching, to avoid surface contamination and oxidation.

### **5.3 Results Registration**

5.3.1 Results from the evaluation shall be registered using an identification and traceability system that allows a correlation of the tested point with the report, and vice-versa.

5.3.2 Shall be issued a report with the following:

- a) name of the issuer (PETROBRAS agency or company performing the service);
- b) numerical identification;
- c) equipment or accessory identification;
- d) number and revision of the procedure;
- e) preparation and etching technique used;
- f) registration of results, with indication of where the evaluation took place;
- g) a written report indicating the findings and recommendations;
- h) a photography registration of the typical structures and events, with amplification and / or scale used;
- i) date;
- j) identification, and signature by the inspector and the person who prepared the report.

## ÍNDICE DE REVISÕES

**REV. A**

Não existe índice de revisões.

**REV. B**

Partes Atingidas	Descrição da Alteração
	Revalidação

## REV. C

[illegible]