

Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination

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National foreword

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English version

Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination

Essais destructifs des soudures sur matériaux
métalliques — Réactifs pour examen
macroscopique et microscopique

Zerstörende Prüfung von Schweißnähten an
metallischen Werkstoffen — Ätzungen für die
makroskopische und mikroskopische Untersuchung

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CEN

European Committee for Standardization
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Foreword

This technical report was prepared by the technical committee CEN/TC 121, Welding, of which the secretariat is held by DS.
The technical committee agreed to publish this technical report.

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1 Scope

This technical report gives a non-exhaustive list of etchants which can be used for the macroscopic and microscopic examination of welds in accordance with EN 1321 for the following materials:

- carbon and low alloy steels;
- stainless steels;
- nickel and nickel alloys;
- titanium and titanium alloys;
- copper and copper alloys;
- aluminium and aluminium alloys.

2 Normative references

This European technical report incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European technical report only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1321 *Destructive tests on welds in metallic materials — Macroscopic and microscopic examination of welds*

3 General

Where details of concentration or waters of crystallization of reagents are not defined in the annexes, table 1 applies. These should be confirmed by the suppliers of each etchant.

Table 1. Characteristics of components

Components	Characteristics			
	Specific gravity g/cm ³	Concentration %	Hydrate	Remarks
HCl	1,18 1,16	35 to 38 31,5 to 33	—	
HF	1,13	40	—	
HNO ₃	1,42	69	—	
H ₂ SO ₄	1,84	98	—	
H ₂ O ₂	—	6 % W/V ¹⁾	—	Usually 20 volumes (i.e. 20 volumes of available O ₂)
H ₃ PO ₄	1,70	85	—	
CH ₃ COOH	1,05	99,1	—	Glacial
HBFe ₄	1,23	35	—	
C ₂ H ₂ O ₄	—	—	2	
FeCl ₃	—	—	6	
CuCl ₂	—	—	2	
MgCl ₂	—	—	6	
Fe(NO ₃) ₃	—	—	9	

¹⁾ W/V means weight by volume.

4 Etchants for carbon and low alloy steels

The etchants for carbon and low alloy steels are given in annex A.

5 Etchants for stainless steels

The etchants for stainless steels are given in annex B.

6 Etchants for nickel and nickel alloys

The etchants for nickel and nickel alloys are given in annex C.

7 Etchants for titanium and titanium alloys

The etchants for titanium and titanium alloys are given in annex D.

8 Etchants for copper and copper alloys

The etchants for copper and copper alloys are given in annex E.

9 Etchants for aluminium and aluminium alloys

The etchants for aluminium and aluminium alloys are given in annex F.

10 Designation

Etchants should be designated either by names or by numbers of tables in accordance with annex G.

Annex A (informative)

Etchants for carbon and low alloy steels

See tables A.1 to A.13

Table A.1 Nital
Type of etchant Macroscopic and microscopic etchant
Composition in volume and in order of mixing 99 ml to 95 ml industrial methylated spirits* 1 ml to 5 ml nitric acid (HNO_3) * ethyl alcohol ($\text{C}_2\text{H}_5\text{OH}$), denatured with methyl alcohol (CH_3OH) Also methyl alcohol or isoamyl alcohol [$(\text{CH}_3)_2\text{CH}(\text{CH}_2)_2\text{OH}$]
Safe shelf life Indefinite
Surface preparation 600 grit or finer (macroetching $\approx 5\%$ of nitric acid) 3 μm diamond or finer (microetching $\approx 2\%$ of nitric acid)
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Can increase strength to 15 % for macroetching on ground surface; reveals ferrite boundaries; differentiates ferrite from martensite. Good general purpose etchant. Amyl alcohol is preferable for galvanized steel

Table A.2 Picral (4 %)
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol ($\text{C}_2\text{H}_5\text{OH}$) 4 g picric acid [$\text{C}_6\text{H}_2\text{OH}(\text{NO}_2)_3$] [+ wetting agent (sodium dodecyl benzene sulfate) ($\text{C}_{18}\text{H}_{29}\text{NaSO}_3$) if required]
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Little attack prior austenite boundaries; detects carbides; good resolution with fine pearlite, martensite, tempered martensite and bainitic structures

Table A.3 Picric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 1 l saturated aqueous picric acid [$C_6H_2OH(NO_2)_3$] 10 ml wetting agent (sodium dodecyl benzene sulfate) ($C_{18}H_{29}NaSO_3$)
Safe shelf life Indefinite
Surface preparation 3 μm diamond or finer
Etching temperature 55 °C to 60 °C
Etching time Few seconds, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Reveals prior grain boundaries and segregation

Table A.4 Picral (15 %)
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol* (C_2H_5OH) 15 g picric acid [$C_6H_2OH(NO_2)_3$] * Also methyl alcohol (CH_3OH)
Safe shelf life Indefinite
Surface preparation 2 μm diamond or finer
Etching temperature Ambient
Etching time Few seconds to a minute, check by eye
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Reveals general structure. Composition given will saturate the solution with picric acid

Table A.5 Hydrochloric picric acid solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml ethyl alcohol (C ₂ H ₅ OH) 1 ml hydrochloric acid (HCl) 4 g picric acid [C ₆ H ₂ OH(NO ₂) ₃]
Safe shelf life Indefinite
Surface preparation 3 µm diamond or finer
Etching temperature Ambient
Etching time 10 s to a few minutes
Additional precautions/requirements Usual precautions for handling and disposal of acids
Comments Microstructural characterization of HAZ, weld and parent metal. Especially effective for very fine structures. Less effective than Nital for the ferrite grain boundaries

Table A.6 Ammonium peroxodisulfate solution
Type of etchant Microscopic etchant
Composition in volume and in order of mixing 100 ml water 10 g ammonium peroxodisulfate [(NH ₄) ₂ S ₂ O ₈]
Safe shelf life Limited
Surface preparation 6 µm diamond or finer
Etching temperature Ambient
Etching time 2 min to 3 min
Additional precautions/requirements
Comments Reveals extent of HAZ and microscopic features of multipass welds