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

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



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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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ISO/TR 16060 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 5, *Testing and inspection of welds*.

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

1 Scope

This Technical Report gives a non-exhaustive list of etchants that can be used for the macroscopic and microscopic examination of welds in accordance with ISO 17639 for the following groups of materials:

- carbon steels 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

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 (including any amendments) applies.

ISO 17639, *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 is applicable. These values should be confirmed by the suppliers of each etchant.

4 Etchants for carbon steels and low-alloy steels

The etchants for carbon and low alloyed 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.

Table 1 — Characteristics of components

Components	Characteristics			Remarks
	Specific gravity g/cm ³	Concentration %	Hydrate	
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 ^a	—	Usually 20 volumes (i.e. 20 volume available O ₂)
H ₃ PO ₄	1,70	85	—	
CH ₃ COOH	1,05	99,1	—	glacial
HF 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	
^a W/V means weight by volume.				

Annex A (informative)

Etchants for carbon steels 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 (C ₂ H ₅ OH), denatured with methyl alcohol (CH ₃ OH) Also methyl alcohol or isoamyl alcohol [(CH ₃) ₂ CH(CH ₂) ₂ OH]
Safe shelf life: Indefinite
Surface preparation: 600 grit or finer (macroetching ≈ 5 % of nitric acid) 3 µm diamond or finer (microetching ≈ 2 % of nitric acid)
Etching temperature: Ambient
Etching time: A 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 (C_2H_5OH) 4 g picric acid [$C_6H_2OH(NO_2)_3$] [+ wetting agent (sodium dodecyl benzene sulphate) ($C_{18}H_{29}NaSO_4$) if required]
Safe shelf life: Indefinite
Surface preparation: 3 μm diamond or finer
Etching temperature: Ambient
Etching time: A 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 sulphate) ($C_{18}H_{29}NaSO_4$)
Safe shelf life: Indefinite
Surface preparation: 3 μm diamond or finer
Etching temperature: 55 °C to 60 °C
Etching time: A 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* ($\text{C}_2\text{H}_5\text{OH}$) 15 g picric acid [$\text{C}_6\text{H}_2\text{OH}(\text{NO}_2)_3$] *Also methyl alcohol (CH_3OH)
Safe shelf life: Indefinite
Surface preparation: 2 μm diamond or finer
Etching temperature: Ambient
Etching time: A few seconds to one minute — check by eye
Additional precautions/requirements: Usual precautions for handling and disposal of acids.
Comments: Reveals general structure. The composition given saturates 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 ($\text{C}_2\text{H}_5\text{OH}$) 1 ml hydrochloric acid (HCl) 4 g picric acid [$\text{C}_6\text{H}_2\text{OH}(\text{NO}_2)_3$]
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 peroxodisulphate solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml water (H ₂ O) 10 g ammonium peroxodisulphate [(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: nil
Comments: Reveals extent of HAZ. Microscopic features of multipass welds.

Table A.7 — Alcoholic hydrochloric 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 to 5 ml hydrochloric acid (HCl)
Safe shelf life: Indefinite
Surface preparation: 3 µm diamond or finer
Etching temperature: 40 °C to 50 °C
Etching time: A few seconds to one minute
Additional precautions/requirements: Usual precautions for handling and disposal of acids. Add HCl to C ₂ H ₅ OH.
Comments: nil

Table A.8 — 120/10/30 etchant

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 120 ml ethyl alcohol (C ₂ H ₅ OH) or methyl alcohol (CH ₃ OH) 10 ml iron (III) chloride (FeCl ₃) (60 % W/V) 30 ml hydrochloric acid (HCl)
Safe shelf life: Indefinite
Surface preparation: 2 µm diamond or finer
Etching temperature: Ambient
Etching time: A few seconds by immersion — check by eye
Additional precautions/requirements: Usual precautions for handling and disposal of acids.
Comments: Good general-purpose etchant.

Table A.9 — Cuprochloric solution 1

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 30 ml water (H ₂ O) 25 ml ethyl alcohol (C ₂ H ₅ OH) 40 ml hydrochloric acid (HCl) 5 g copper (II) chloride (CuCl ₂)
Safe shelf life: 2 h
Surface preparation: 1 000 grit or finer
Etching temperature: Ambient
Etching time: 10 s to 20 s
Additional precautions/requirements: After the etching, the specimen should be washed in order to remove copper deposits. Usual precautions for handling and disposal of acids.
Comments: Reveals cold working strain lines.