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

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee WEE/2, Destructive Testing of Welds.

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Destructive tests on welds in metallic materials - Etchants for macroscopic and microscopic examination (ISO/TR 16060:2003)

Essais destructifs des soudures sur matériaux métalliques - Réactifs pour examens macroscopique et microscopique (ISO/TR 16060:2003)

Zerstörende Prüfung von Schweißverbindungen an metallischen Werkstoffen - Ätzungen für die makroskopische und mikroskopische Untersuchung (ISO/TR 16060:2003)

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Foreword

The text of ISO/TR 16060:2003 has been prepared by Technical Committee ISO/TC 44 “Welding and allied processes” of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TR 16060:2014 by Technical Committee CEN/TC 121 “Welding and allied processes” the secretariat of which is held by DIN.

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This document supersedes CR 12361:1996.

Endorsement notice

The text of ISO/TR 16060:2003 has been approved by CEN as CEN ISO/TR 16060:2014 without any modification.

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TECHNICAL REPORT **ISO/TR 16060:2003(E)**

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			
	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_2SO_4	1,84	98	—	
H_2O_2	—	6 % W/V [a]	—	Usually 20 volumes (i.e. 20 volume available O_2)
H_3PO_4	1,70	85	—	
CH_3COOH	1,05	99,1	—	glacial
HBF_4	1,23	35	—	
$C_2H_2O_4$	—	—	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_2H_5OH), denatured with methyl alcohol (CH_3OH) Also methyl alcohol or isoamyl alcohol [$(CH_3)_2CH(CH_2)_2OH$]
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* (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: 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 (C_2H_5OH) 1 ml hydrochloric acid (HCl) 4 g picric acid [$C_6H_2OH(NO_2)_3$]
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_2O) 10 g ammonium peroxodisulphate [$(NH_4)_2S_2O_8$]
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_2H_5OH) 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_2H_5OH.
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_2H_5OH) or methyl alcohol (CH_3OH) 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_2O) 25 ml ethyl alcohol (C_2H_5OH) 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.

Table A.10 — Magneso cuprochloric solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml ethyl alcohol (C_2H_5OH) 20 ml water (H_2O) 1 ml hydrochloric acid (HCl) 4 g magnesium (II) chloride ($MgCl_2$) 1 g copper (II) chloride ($CuCl_2$)
Safe shelf life: 2 h
Surface preparation: 3 µm diamond or finer
Etching temperature: Ambient
Etching time: 60 s
Additional precautions/requirements: Salts should be dissolved in the smallest amount of hot water, then diluted with ethyl alcohol. A slight polishing (1 µm) after the etching furnishes the best contrast.
Comments: Reveals phosphorus and related segregations. The copper deposits first of all on those areas poorest in phosphorus.

Table A.11 — Adler's etchant

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 25 ml water (H_2O) 3 g ammonium tetra chloro diaquo cuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$] 50 ml hydrochloric acid (HCl) 15 g iron (III) chloride ($FeCl_3$)
Safe shelf life: Months
Surface preparation: 320 grit or finer
Etching temperature: Ambient
Etching time: 5 s to 10 s
Additional precautions/requirements: Add [$(NH_4)_2CuCl_4 \cdot 2H_2O$] to H_2O (a). Add $FeCl_3$ to HCl (b). Mix both then add (b) to (a).
Comments: nil

Table A.12 — Heyn's etchant

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 120 ml water (H_2O) 10 g ammonium tetra chloro diaquo cuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$]
Safe shelf life: Months
Surface preparation: 240 grit or finer
Etching temperature: Ambient
Etching time: 10 s to 1 min
Additional precautions/requirements: Add [$(NH_4)_2CuCl_4 \cdot 2H_2O$] to H_2O.
Comments: Copper deposit shall be removed.

Table A.13 — Ferric chloride solution

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 70 ml water (H_2O) 30 ml iron (III) chloride ($FeCl_3$) (60 % W/V)
Safe shelf life: Indefinite.
Surface preparation: 1 000 grit or finer
Etching temperature: Ambient
Etching time: A few seconds
Additional precautions/requirements: Usual precaution for handling and disposal of acids.
Comments: 1) Swab the surface; 2) Rinse with water; 3) Swab again the surface; 4) After etching, water rinse, alcohol rinse, dry.

Annex B
(informative)

Etchants for stainless steels

See Tables B.1 to B.12.

Table B.1 — Oxalic acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml water (H_2O) 10 g oxalic acid (ethanedioic acid) ($C_2H_2O_4$)
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: Electrolytic 2 V to 6 V. Can reveal sensitivity to inter-crystalline corrosion. Reveals general structure. Reveals carbides at the grain boundaries.

Table B.2 — Thiocyanate solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 80 ml water (H_2O) 20 ml sulphuric acid (H_2SO_4) 10 g ammonium thiocyanate (NH_4SCN)
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: Electrolytic 1,5 V to 2,0 V. Good general purpose etchant.

Table B.3 — Acidified ferric chloride solution

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 480 ml water (H_2O) 120 ml hydrochloric acid (HCl) 50 g iron (III) chloride ($FeCl_3$)
Safe shelf life: Indefinite
Surface preparation: 600 grit 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: Good general purpose macroscopic etchant, for austenitic chromium nickel and other stainless steels.

Table B.4 — Modified Murakami's etchant

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 60 ml water (H_2O) 30 g potassium ferricyanide [$K_3Fe(CN)_6$] 30 g potassium hydroxide (KOH)
Safe shelf life: Fresh solution
Surface preparation: 1 µm diamond or finer
Etching temperature: Temperature of freshly prepared solution (hot)
Etching time: 20 s to 40 s
Additional precautions/requirements: Usual precautions for handling and disposal of caustic solutions.
Comments: Reveals σ phase from δ ferrite and carbides. Austenite matrix is not revealed.

Table B.5 — Cuprochloric solution 2

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml water(H_2O) 100 ml ethyl alcohol (C_2H_5OH) 100 ml hydrochloric acid (HCl) 5 g copper (II) chloride ($CuCl_2$)
Safe shelf life: 2 h
Surface preparation: 1 µm diamond or finer
Etching temperature: Ambient
Etching time: 10 s to a few minutes
Additional precautions/requirements: After etching, the test specimen should be washed in order to remove copper deposits. Usual precautions for handling and disposal of acids.
Comments: Especially for ferritic and martensitic stainless steels, an alternative to electrolytic etching. Less effective for austenitic grain boundaries that can be attacked using longer etching times. Not effective for carbides.

Table B.6 — Chromic acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml water (H_2O) 10 g chromium (VI) oxide (CrO_3)
Safe shelf life: A few days
Surface preparation: 1 µm diamond or finer
Etching temperature: Ambient
Etching time: 10 s to 1 min
Additional precautions/requirements: Usual precautions for handling and disposal of acids.
Comments: Austenitic grain boundaries. Distinguishes σ phase and carbides. Electrolytic etching (3 V) for general purpose etchings. For the analysis of carbides, the etching is carried out in two steps: first with 1 V and afterwards with 3 V.

Table B.7 — Alcoholic hydrochloric solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml ethyl alcohol (C_2H_5OH) 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: Add HCl to C_2H_5OH. Usual precaution for handling and disposal of acids.
Comments: nil

Table B.8 — Hydrochloric nitric acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 39 ml water (H_2O) 52 ml hydrochloric acid (HCl) 9 ml nitric acid (HNO_3), (concentration 65 %)
Safe shelf life: Indefinite
Surface preparation: 3 µm diamond or finer
Etching temperature: Ambient
Etching time: A few seconds to a few minutes
Additional precautions/requirements: Always add HCl and HNO_3 to H_2O. Usual precautions for handling and disposal of acids.
Comments: nil

Table B.9 — Adler's etchant

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 25 ml water (H_2O) 3 g ammonium tetra chloro diaquo cuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$] 50 ml hydrochloric acid (HCl) 15 g iron (III) chloride ($FeCl_3$)
Safe shelf life: Months
Surface preparation: 320 grit or finer.
Etching temperature: Ambient
Etching time: 5 s to 10 s
Additional precautions/requirements: Add [$(NH_4)_2$ $CuCl_4 \cdot 2H_2O$ to H_2O] (a). Add $FeCl_3$ to HCl (b). Mix both then add (b) to (a).
Comments: nil

Table B.10 — Fluonitric acid solution 1

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 50 ml nitric acid (HNO_3) 50 ml hydrofluoric acid (HF)
Safe shelf life: Do not store after using.
Surface preparation: 2 µm diamond or finer
Etching temperature: Ambient
Etching time: 5 min to 30 min by immersion
Additional precautions/requirements: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Reveals general structure.

Table B.11 — Fluonitric acid solution 2

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 100 ml water (H_2O) 100 ml nitric acid (HNO_3) 100 ml hydrofluoric acid (HF)
Safe shelf life: Do not store after using.
Surface preparation: 2 µm diamond or finer
Etching temperature: Ambient
Etching time: 1 min to 15 min by immersion
Additional precautions/requirements: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Reveals grain boundaries.

Table B.12 — Nitric acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 50 ml water (H_2O) 50 ml nitric acid (HNO_3)
Safe shelf life: Indefinite
Surface preparation: 2 µm diamond or finer
Etching temperature: Ambient
Etching time: A few seconds
Additional precautions/requirements: Usual precautions for handling and disposal of acids.
Comments: Electrolytic at 3 V to 6 V. Rinse in the solution to remove the film present on the surface.

Annex C
(informative)

Etchants for nickel and nickel alloys

See Tables C.1 to C.4.

Table C.1 — Alcoholic hydrochloric acid solution with hydrogen peroxide

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 65 ml ethyl alcohol (C_2H_5OH) 35 ml hydrochloric acid (HCl) 4 ml hydrogen peroxide (H_2O_2) (20 volumes)
Safe shelf life: Do not store after using.
Surface preparation: 2 µm diamond or finer
Etching temperature: Ambient
Etching time: A few seconds — check by eye
Additional precautions/requirements: Add H_2O_2 just before using. Usual precautions for handling and disposal of acids.
Comments: Good general-purpose etchant.

Table C.2 — Thiocynate solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 80 ml water (H_2O) 20 ml sulfuric acid (H_2SO_4) 10 g ammonia thiocyanate (NH_4SCN)
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: Electrolytic 1,5 V to 2,0 V. Good general-purpose etchant.

Table C.3 — Nitric acetic acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 30 ml acetone [$(CH_3)_2CO$] 30 ml nitric acid (HNO_3) 30 ml acetic acid (CH_3COOH)
Safe shelf life: Do not store after using.
Surface preparation: 3 µm diamond or finer
Etching temperature: Ambient
Etching time: few seconds — check by eye
Additional precautions/requirements: Keep cool. Nitrous oxide given off. Usual precautions for handling and disposal of acids.
Comments: Good general-purpose etchant.

Table C.4 — Adler's etchant

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 25 ml water (H_2O) 3 g ammonium tetra chloro diaquo cuprate (II) [$(NH_4)_2CuCl_4 \cdot 2H_2O$] 50 ml hydrochloric acid (HCl) 15 g iron (III) chloride ($FeCl_3$)
Safe shelf life: Months
Surface preparation: 320 grit or finer
Etching temperature: Ambient
Etching time: 5 s to 10 s
Additional precautions/requirements: Add [$(NH_4)_2CuCl_4 \cdot 2H_2O$] to H_2O (a). Add $FeCl_3$ to HCl (b). Mix both then add (b) to (a).
Comments: nil

Annex D
(informative)

Etchants for titanium and titanium alloys

See Tables D.1 and D.2.

Table D.1 — Keller's etchant

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 950 ml water (H_2O) 25 ml nitric acid (HNO_3) 15 ml hydrochloric acid (HCl) 10 ml hydrofluoric acid (HF)
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: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Good general-purpose etchant.

Table D.2 — Fluonitric acid solution 3

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 880 ml water (H_2O) 100 ml nitric acid (HNO_3) 20 ml hydrofluoric acid (HF)
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: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Good general-purpose etchant.

Annex E
(informative)

Etchants for copper and copper alloys

See Tables E.1 to E.3.

Table E.1 — Alcoholic acidified ferric chloride solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 960 ml industrial methylated spirits* 20 ml hydrochloric acid (HCl) 50 g iron (III) chloride ($FeCl_3$) *ethyl alcohol (C_2H_5OH) denatured with methyl alcohol (CH_3OH)
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: Good general-purpose etchant, especially for high copper content alloys.

Table E.2 — Ammonium peroxodisulfate solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 90 ml water (H_2O) 10 mg ammonium peroxodisulfate [$(NH_4)_2S_2O_8$] 10 ml ammonium hydroxide (ammonia solution) (NH_3 in H_2O) specific gravity 0,880 g/cm^3.
Safe shelf life: Use fresh. Do not store after using.
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 caustic solutions.
Comments: Good general-purpose etchant.

Table E.3 — Nitric acid with ammonium and ferric nitrate solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 500 ml water (H_2O) 2 ml nitric acid (HNO_3) 2 g ammonium nitrate (NH_4NO_3) 20 g iron (III) nitrate [$Fe(NO_3)_3$]
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: Good general-purpose etchant.

Annex F
(informative)

Etchants for aluminium and aluminium alloys

See Tables F.1 to F.7.

Table F.1 — Sodium hydroxide solution

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 100 ml water (H_2O) 15 g sodium hydroxide (NaOH)
Safe shelf life: Do not store after using.
Surface preparation: 600 grit or finer
Etching temperature: Ambient
Etching time: A few seconds — check by eye
Additional precautions/requirements: Usual precautions for handling and disposal of caustic solutions. Use in a fume cupboard.
Comments: Good general-purpose etchant. Can be used in various dilutions.

Table F.2 — Keller's etchant

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 950 ml water (H_2O) 25 ml nitric acid (HNO_3) 15 ml hydrochloric acid (HCl) 10 ml hydrofluoric acid (HF)
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: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Good general-purpose etchant. Warning: grain boundary attack can look like cracks.

Table F.3 — Hydrochloric nitric hydrofluoric acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 350 ml water (H_2O) 300 ml hydrochloric acid (HCl) 300 ml nitric acid (HNO_3) 50 ml hydrofluoric acid (HF)
Safe shelf life: Is indicated by a change of colour to greenish brown and sluggish reaction.
Surface preparation: 3 µm diamond or finer
Etching temperature: Ambient
Etching time: 30 s to 60 s after start of reaction.
Additional precautions/requirements: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Warning: Grain boundary attack can look like cracks.

Table F.4 — Hydrochloric nitric orthophosphoric acid solution

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 480 ml water (H_2O) 200 ml hydrochloric acid (HCl) 200 ml nitric acid (HNO_3) 120 ml orthophosphoric acid (H_3PO_4)
Safe shelf life: Is indicated by a change of colour to greenish brown and sluggish reaction.
Surface preparation: 3 µm diamond or finer
Etching temperature: 30 °C to 40 °C
Etching time: 60 s to 120 s after start of reaction.
Additional precautions/requirements: Usual precautions for handling and disposal of acids.
Comments: Warning: grain boundary attack can look like cracks

Table F.5 — Barker's etchant

Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 940 ml water (H_2O) 60 ml fluorobaric acid (HBF_4)
Safe shelf life: Normally is stable for an unlimited period only.
Surface preparation: 3 µm diamond of finer
Etching temperature: Ambient
Etching time: 40 s to 60 s
Additional precautions/requirements: See in Clause F.1 the description of the "Barker" procedure.
Comments: Warning: grain boundary attack can look like cracks. For examination of grain and/or fibre structures of aluminium and aluminium alloys, the examination of the "Barker" anodized samples under polarized light proved to be successful and produced the required result

Table F.6 — Poulton's etchant

Type of etchant: Macroscopic etchant
Composition in volume and in order of mixing: 50 ml water (H_2O) 600 ml hydrochloric acid (HCl) 300 ml nitric acid (HNO_3) 50 ml hydrofluoric acid (HF)
Safe shelf life: Do not store after using.
Surface preparation: 1 000 grit or finer
Etching temperature: Ambient
Etching time: A few seconds — check by eye
Additional precautions/requirements: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Reveals general macroscopic structure.

Table F.7 — Kroll's etchant

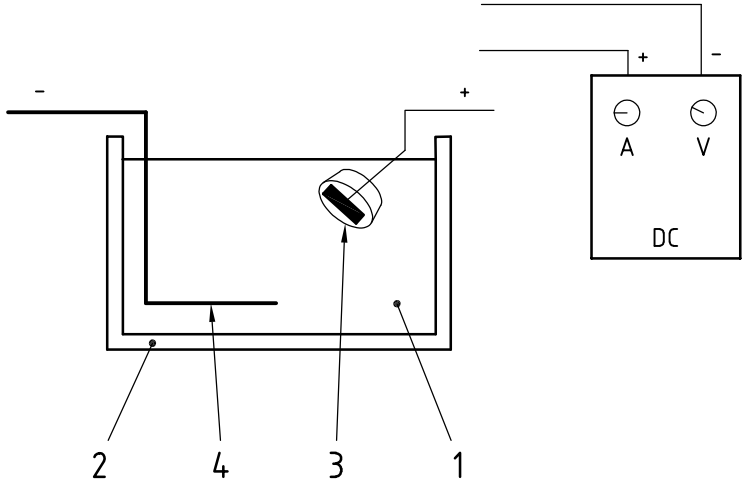
Type of etchant: Microscopic etchant
Composition in volume and in order of mixing: 960 ml water (H_2O) 30 ml nitric acid (HNO_3) 10 ml hydrofluoric acid (HF)
Safe shelf life: Indefinite
Surface preparation: 3 µm diamond or finer
Etching temperature: Ambient
Etching time: $<$ 5 s
Additional precautions/requirements: **CAUTION: When handling HF wear hand and eye protection. In the event of bodily contact, wash off skin immediately and seek medical advice.** Use receptacles in plastic. Usual precautions for handling and disposal of acids.
Comments: Warning: grain boundary attack can look like cracks.

F.1 Description of the “Barker” procedure

F.1.1 General

The Barker anodic oxidation procedure should be used (see Table F.5).

A schematic representation of the method is given in Figure F.1.



Key

1 electrolyte [6 % by volume HBF_4 (35 % by weight) in distilled water]
2 plastic container
3 anode (test specimen connected to an electrode by a good electrical conductor e.g. aluminium wire)
4 cathode (pure aluminium sheet)

Voltage: 25 V to 30 V (D.C)
Time: 40 s to 60 s
Maximum temperature: ambient temperature (stirring or cooling of electrolyte bath)

Figure F.1 — Schematic representation of the “Barker” procedure

F.1.2 Treatment

After the anodic oxidation, wash the test specimen under running water and remove the water using alcohol, dry in hot air.

Do not apply ultrasonic treatments.

F.1.3 Examination

Observe the oxidized surface with polarized light using a light microscope.

Annex G
(informative)

List of etchants

See Tables G.1 and G.2.

Table G.1 — List of etchants classified by groups of material

Name	Table	Type of etchant	Material
Adler's etchant	A.11	Macroscopic	Carbon steels and low-alloy steels
Alcoholic hydrochloric solution	A.7	Microscopic	
Ammonium peroxodisulfate solution	A.6		
Cuprochloric solution 1	A.9	Macroscopic	
Ferric chloride solution	A.13		
Heyn's etchant	A.12		
Hydrochloric picric solution	A.5	Microscopic	
Magneso cuprochloric solution	A.10		
Nital	A.1	Macroscopic and microscopic	
Picral (4 %)	A.2	Microscopic	
Picral (15 %)	A.4		
Picric acid solution	A.3		
120/10/30 etchant	A.8		
Acidified ferric chloric solution	B.3	Macroscopic	Stainless steels
Adler's etchant	B.9		
Alcoholic hydrochloric solution	B.7	Microscopic	
Chromic acid solution	B.6		
Cuprochloric solution 2	B.5		
Fluonitric acid solution 1	B.10		
Fluonitric acid solution 2	B.11		
Hydrochloric nitric acid solution	B.8		
Modified Murakami's etchant	B.4	Macroscopic	
Nitric acid solution	B.12	Microscopic	
Oxalic acid solution	B.1		
Thiocyanate solution	B.2		

Table G.1 (*continued*)

Name	Table	Type of etchant	Material
Adler's etchant	C.4	Macroscopic	Nickel and nickel alloys
Alcoholic hydrochloric acid with hydrogen peroxide	C.1	Microscopic	
Nitric acetic acid solution	C.3		
Thiocyanate solution	C.2		
Keller's etchant	D.1		Titanium and titanium alloys
Fluonitric solution 3	D.2		
Alcoholic acidified ferric chloride solution	E.1		Copper and copper alloys
Ammonium peroxodisulfate solution	E.2		
Nitric acid with ammonium and ferric nitrate	E.3		
Barker's etchant	F.5		Aluminium and aluminium alloys
Hydrochloric nitric hydrofluoric acid solution	F.3		
Hydrochloric nitric orthophosphoric acid solution	F.4		
Keller's etchant	F.2		
Kroll's etchant	F.7		
Poulton's etchant	F.6	Macroscopic	
Sodium hydroxide solution	F.1		

Table G.2 — List of etchants by English alphabetical order

Name	Table	Type of etchant [a]	Material
Acidified ferric chloride solution	B.3	A	Stainless steels
Adler's etchant	A.11		Carbon steels and low-alloy steels
	B.9		Stainless steels
	C.4		Nickel and nickel alloys
Alcoholic acidified ferric chloride solution	E.1	I	Copper and copper alloys
Alcoholic hydrochloric acid with hydrogen peroxide	C.1		Nickel and nickel alloys
Alcoholic hydrochloric solution	A.7		Carbon steels and low-alloy steels
	B.7		Stainless steels
Ammonium peroxodisulfate solution	E.2		Copper and copper alloys
	A.6		Carbon steels and low-alloy steels
Barker's etchant	F.5		Aluminium and aluminium alloys
Chromic acid solution	B.6		Stainless steels
Cuprochloric solution 1	A.9	A	Carbon steels and low-alloy steels
Cuprochloric solution 2	B.5	I	Stainless steels
Ferric chloride solution	A.13	A	Carbon steels and low-alloy steels
Fluonitric acid solution 1	B.10	I	Stainless steels
Fluonitric acid solution 2	B.11		
Fluonitric acid solution 3	D.2		Titanium and titanium alloys
Heyn's etchant	A.12	A	Carbon steels and low-alloy steels

[a] A = macroscopic etching and I = microscopic etching in accordance with ISO 17639.

Table G.2 (*continued*)

Name	Table	Type of etchant [a]	Material
Hydrochloric nitric acid solution	B.8	I	Stainless steels
Hydrochloric nitric hydrofluoric acid solution	F.3		Aluminium and aluminium alloys
Hydrochloric nitric orthophosphoric acid solution	F.4		
Hydrochloric picric acid	A.5		Carbon steels and low-alloy steels
Keller's etchant	D.1		Titanium and titanium alloys
	F.2		Aluminium and aluminium alloys
Kroll's etchant	F.7		
Magneso cuprochloric solution	A.10		Carbon steels and low-alloy steels
Modified Murakami's etchant	B.4	A	Stainless steels
Nital	A.1	A and I	Carbon steels and low-alloy steels
Nitric acetic acid solution	C.3	I	Nickel and nickel alloys
Nitric acid with ammonium and ferric nitrate	E.3		Copper and copper alloys
Nitric acid solution	B.12		Stainless steels
Oxalic acid solution	B.1		Stainless steels
Picral (4 %)	A.2		Carbon steels and low-alloy steels
Picral (15 %)	A.4		
Picric acid solution	A.3		
Poulton's etchant	F.6	A	Aluminium and aluminium alloys
Sodium hydroxide	F.1		
Thiocyanate solution	B.2	I	Stainless steels
	C.2		Nickel and nickel alloys
120/10/30 etchant	A.8		Carbon steels and low-alloy steels

[a] A = macroscopic etching and I = microscopic etching in accordance with ISO 17639.

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