

ENE Electropolish NC-3

The ENE Electropolish NC-3 is a proprietary electrolyte to which chrome is added to. It is formulated and designed to process a wide variety of steels when utilized under proper conditions and in conjunction with suitable equipment. The ENE Electropolish NC-3 is a stable, easily controlled and economic solution readily adaptable to obtain the many advantages inherent in the electropolishing process.

Features & Benefits

High quality raw materials	Product consistency and performance
Rigorous quality assurance	Product consistency and performance

Physical Data

Specific gravity	1.09
Solubility in water	Complete
Appearance and odor	Light amber, clear, mild odor
pH 10% solution	1.0 – 2.0
pH concentrate	< 1.0

Operating Conditions

For use in electropolishing of carbon steel substrates chromic acid must be added to ENE Electropolish NC-3 in the following ratio:

Concentration of ENE Electropolish NC-3	93.56% by weight Note: ENE Electropolish NC-3 weighs 14.21 pounds per gallon.
Concentration of Chromic Acid	6.44% by weight

In electropolishing, the work piece is made anodic in the strongly acidic ENE Electropolish NC-3. As current is passed through the part, metal is dissolved from the surface of the part at a rate dependent on the current density applied and the temperature of the electrolyte. In the electropolishing process, metal is removed more rapidly from the high points of the metal surface and this produces the effect of smoothening and brightening the metal surface. This preferential



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metal removal rate also permits the electropolishing process to be effectively utilized for the deburring of metals. Electropolishing by removing surface inclusions, surface skin and through the formation of anodic oxide film, increases the corrosion resistance and passivity of the metal, as well as serving to reduce surface stresses and strains. Similarly, electropolishing will produce a clean, uncontaminated surface well suited for subsequent plating or for applications where exceptionally clean surfaces are required.

Electropolishing, being both a surface and anodic phenomenon, does not cause hydrogen embrittlement nor does electropolishing change the basic composition or properties of the metal or alloy being processed.

The amount of surface metal which must be removed to obtain the desired effect from electropolishing is determined by initial surface quality of the part, the metallurgy of the part and the intended application for the electropolishing process.

Generally, fine grained metals or alloys having a clean, scale-free surface will electropolish best. Parts which have deep scratches, gouges, dents or are fabricated from non-homogenous or multiphase alloys will not electropolish well or may require extended electropolishing times.

A typical cycle in electropolishing is as follows:

1. Degrease.
2. Descale.
3. Electropolish.
4. Water rinse.
5. Water rinse.
6. Neutralize if necessary.
7. Water rinse.
8. Dry.

Cleaning Before Electropolishing

Cleaning is necessary to remove surface soils such as grease, oil, fingerprints, drawing compounds, etc. Surface residues must be removed; otherwise they will interfere with the subsequent electropolishing and may cause an etched, pitted or non-uniform surface finish.

Vapor degreasing is very satisfactory method of cleaning. Degreasing units commonly used for cleaning prior to electroplating are suitable. Alkaline cleaners, used with or without current, emulsion or solvent type cleaners or detergent type cleaners are also suitable systems for cleaning prior to electropolishing. Where necessary, thorough water rinsing after cleaning should be provided to ensure that no cleaning material is dragged into the electropolishing solution. Likewise, excessive drag-in of water into the electropolishing electrolyte is detrimental and is to be avoided. Therefore, where aqueous systems are being used for cleaning, care must be taken to provide adequate drain and run-off time so that excessive water will not be dragged into the electropolishing electrolyte.

For most applications in the electropolishing of steels, descaling prior to electropolishing will not be required. However, where parts to be processed have heavy heat-treating scale, are scorched, have heavy weld scale or other tenacious surface oxides; it may be necessary to descale such parts



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prior to electropolishing to insure consistent high-quality results. Suitable descaling solution will depend on the metals or alloys being processed and may be based on nitric, hydrochloric, hydrofluoric or sulfuric acids. Molten salt descales, alkaline descaling systems, or other suitable methods may also be utilized. The descaling method chosen should not produce excessive attack on the basis metal. Parts being descaled in aqueous systems should be thoroughly rinsed after the descaling operation and contamination of the electropolishing electrolyte with descaling solution must be avoided.

Electropolishing

The operating conditions for the ENE Electropolish NC-3 are relatively wide and the most preferred parameters will depend on the metal or alloy being processed, the nature and geometry of the part and/or application required. It is advisable to experimentally determine these optimum operating parameters on trial samples prior to the design and installation of any electropolishing system. For most applications, and generally, the following operating conditions will apply to the processing of steels with the ENE Electropolish NC-3 and they may be used as a guide:

Temperature	130°F –180°F
Current densities	150 – 450 Amps/ft ²
Operating voltages	6 – 18 volts
Electropolishing time	3 – 20 minutes
Maximum current input	5 Amps/gallon
Cathode to anode ratio	10:1 to 1:1
Metal removal rate	0.00005 – 0.0001 inches per exposed surface per minute of electropolishing time; dependent on operating current densities
Distance of work from cathode	2 – 6 inches
Distance of work from tank bottom	> 6 inches

Current loads more than 5 amperes per gallon causes an overheating of the electrolyte. Electropolishing time is dependent on the initial condition of the surface, the finish and results desired and the dimension tolerances for the part. The electropolishing time is also dependent on operating current densities. Higher operating current densities will generally produce the desired results in shorter electropolishing times that when low operating current densities are utilized.

Equipment for Electropolishing

Processing tanks for electropolishing should be constructed of steel, chemical lead lined. The thickness of the lead lining should be the same as that generally utilized in chromium plating tanks (8 lb Lead). The weight of the ENE Electropolish NC-3 is approximately 14 lbs per gallon. Tanks



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should be constructed to handle this weight. Pyrex glass tanks or molded plastics tanks within a stainless-steel shell can also be used. In choosing any plastic, extreme care must be used to ensure that the plastic or synthetic will tolerate the maximum temperature which the bath may reach.

Heating facilities of enough capacity to heat the electropolishing solution to operating temperature in a reasonable time may utilize steam, electric heat or external gas fired heat exchangers. Steam heating coils should be constructed of chemical lead or Teflon and equipped with steam traps, strainers, as well as safety devices to prevent possible solution back-up into boiler; electric immersion heaters should be fabricated from graphite, quartz, or other suitable acid resistant materials. Temperature may be manually or thermostatically controlled. Sensing elements used with automatic and thermostatic control systems should be encased in Teflon or chemical lead.

A DC power source capable of providing the necessary amperage and working voltage is required. Rectifiers or DC generators should be provided with variable voltage control and capable of producing the needed amperage per workload at 6 to 18 volts at the electropolishing tank.

Cathodes should be made of chemical lead or 316 stainless steel. It is advantageous to have the cathodes mounted on sliding cathode bars so that cathodes can be positioned as close to or as far from the work as might be required by the shape of the work piece. Provision should also be made for easy removal of the cathodes from the cathode bar. This will simplify maintenance and cleaning of the cathodes when necessary.

Anode and cathode bars, bus bars and auxiliary electrical connections should be made of copper, brass or the other electrically conductive material. Care should be taken to ensure that adequate cross-sectional area is available in the electrical system to carry the intended current loads. As a rule, one square inch of copper will conduct 1000 amperes of current. All electrical connections and joints should be kept clean and tight to insure proper conductivity.

Agitation, either air or work bar, will in some applications, be advisable to prevent gas streaking or to increase the electropolishing effect. Work bar agitation can be accomplished by a tank mounted agitator motor. The motor should be capable of producing a 1 to 3-inch stroke at 15 to 30 cycles per minute. Air agitation of the solutions is helpful to prevent solution stratification and to maintain uniform bath temperature. Air agitation may be achieved by means of an air blower or by compressed air fed through a U or H shaped chemical lead or suitable plastic air coil mounted on the bottom of the electropolishing tank. The air entering the electropolishing solution should be filtered and oil free. A regulating valve should be installed on the air pattern. Air agitation may be used in conjunction with work bar agitation.

The electropolishing tank should be ducted and exhausted so that the oxygen and hydrogen, as well as the slight acid spray produced during electropolishing are drawn off. Exhausting requirements for electropolishing are like those necessary in chromium plating. Two- or three-sided duct castings or slotted hood type ducts may be used. Exhaust capacities required are in the order of 1800 to 2500 cubic feet per minute. Ducts, blowers and exhaust fans should be constructed of acid resistant material such as 300 series stainless steel, PVC, fiberglass, asphalt covered steel, rubber, etc.



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Racking

The parts to be electropolished must be firmly and securely fixtured on suitable contacts for introduction into the electropolishing tank. The design of these fixtures should provide for adequate electrical conductivity, provide for proper positioning in the electropolishing tank should be designed to permit adequate draining of the electropolishing solution.

Racks may be constructed of titanium or plastisol coated copper with titanium contact tips. Titanium is unaffected by the electropolishing solution and titanium racks give exceptionally long service life. However, because of the relatively low electrical conductivity of titanium and because of its cost, the initial costs for this type rack will be high. Copper splined racks, plastisol coated with titanium contact points, are excellent both in terms of initial cost and service life.

In the construction of the racks, attention must be given to the total current and contact points will carry and the necessary cross-sectional area provided; 0.001 square inches of copper will carry approximately one ampere. The number of parts to be mounted on rack should be considered in terms of the eventual weight of the rack and the geometry of the part. Parts should be positioned on the rack so that they do not shield or overlap one another, so that they do not offer areas for gas pockets or gas streaking; and so that the electropolishing solution can readily be rinsed and drained from the part.

Rinsing After Electropolishing

After the work is removed from the electropolishing bath, it should be rinsed free of the electrolyte as quickly as possible. Running cold water rinses with or without spray attachments are suitable. Rinse water turbulence, either through air agitation of the rinse water, countercurrent water flow or by introduction of the rinse water through a multi-outlet pipe on the rinse tank bottom, will improve the effectiveness of the rinsing cycle. Where required, rinse tanks should be equipped with safety devices to prevent siphoning of the rinse back into the water line in the event of an accidental drop in water pressure. Rinsing must be complete and thorough to remove all acid residues, since these residues may cause subsequent staining and/or corrosion. Complex shaped parts having blind hole, folds or similar areas for solution entrapment may require, in addition to water rinsing, an additional neutralization in a 3% to 5% solution of soda ash or proprietary alkaline neutralizer. Following this neutralization, the parts must again be thoroughly rinsed in running water.

Rinse tanks should be constructed of acid resistant materials, such as PVC, polyethylene, polypropylene, 300 series Stainless Steel, fiber glass etc.

Drying After Electropolishing

After water rinsing, the parts may be dried by any of the common methods usually employed in plating, such as:

1. Dipping in hot water and air drying.
2. Drying in sawdust.
3. Centrifuging.
4. Forced hot or compressed air.
5. Solvent vapor drying.
6. Dipping in water displacing oils and degreasing.

Operation and Control

The ENE Electropolish NC-3 is designed to produce a lustrous leveled finish on carbon steels. The electropolished surfaces so produced have increased corrosion resistance and improved surface wear characteristics. The ENE Electropolish NC-3 will operate effectively under a wide range of operating conditions and with little maintenance and control. Usual operating cycles run from 130°F to 180°F and current densities of 150 to 400 amps per square foot.

ENE Electropolish NC-3 is a non-sludging type bath and maintenance and control is accomplished by decantation, specific gravity control and chromic acid control.

During initial operation, the specific gravity will increase approximately according to the amount of iron dissolved as follows:

Dissolved Iron %	Specific Gravity ± 0.01	
	@ 150°F	@ 180°F
0.0	1.71 – 1.72	1.68 – 1.69
1.0	1.72 – 1.73	1.69 – 1.70
2.0	1.73 – 1.74	1.70 – 1.71
3.0	1.74 – 1.75	1.71 – 1.72

Smoothing and brightening will diminish after the concentration of dissolved iron exceeds about 3.0%. It is therefore advisable to operate the electropolishing bath at iron concentration below 3.0%. This may be accomplished by one of two methods. The bath may be used with no decantation until the iron reaches 3.0% and then a large portion, 25% to 50% by volume, of the bath is decanted or the dissolved iron may be permitted to increase to 2.2% and a continuous decantation schedule based on ampere-hour production instituted. The choice of procedure will depend on convenience of control and handling and will vary with different installations.

To maintain the dissolved iron at 2.2% on an ampere-hour basis, experience has shown that for every 2400 ampere hours, 4 gallons of the electropolishing bath is decanted and replaced with 4 gallons of fresh electropolishing bath.

The ENE Electropolish NC-3 contains chromic acid. The chromic acid concentration should be between 5.5% to 6.5%. As the bath is operated, there is a reduction of the chromic acid from the hexavalent state (Cr⁶⁺) to the trivalent state (Cr³⁺). A good indication of the oxidation state of the chromium is the color of the electropolishing solution. As the chromic acid is reduced, the color of the electropolishing solution will change from red to red brown to brown-green and finally green. Additions of chromic acid should be made to maintain the hexavalent chromium content between 5.5% to 6.5% by weight, red to red-brown color.

Analytical procedures for the analysis of iron and chromium are included.



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Quality of Steel

Certain metal characteristics regarding grain size, cleanliness, temper, etc., cannot be tolerated if good blanking and cold-forming results are to be obtained. Similarly, there are certain characteristics which act against obtaining optimum appearance that can be expected by electropolishing.

There are structural characteristics that can vary in steel without appreciably affecting the nominally specified properties, yet which will influence electropolishing results. In general, fine-grained (Size 9, ASTM Standard) clean steel gives “excellent” appearance. When the grain size is 7 to 8, the appearance of the electropolished steel generally will be only “fair” and might be “good”. With grain size 6, results are usually poor. Pitting is sometimes associated with steel having nonmetallic inclusions.

Comments on the Appearance of Plane Surfaces

Certain appearance defects are not associated with the quality of the metal but relate to conditions not being “just right” in the tank.

If streaks appear on the polished surface, work placement is improper for the agitation being used. Horizontal surfaces, particularly facing down, are to be avoided. Place the work so that all surfaces slant into the direction of motion insofar as is possible.

“Pebbly” or pitted appearance on some areas might result from too low current density due to poor current distribution. Higher average current density, or higher specific gravity, or both, may be the correction. Also, too high specific gravity causes a loss in luster and an “unfinished” appearance, due to loss in brilliance.

If the cathodes are too far away from the work, and if the sides, ends, and bottom of tank are in the cathode circuit, poor current distribution to the work will result. This situation can be avoided by installing glass-sheet inserts at the sides, ends, and bottom of the tank, to act as barriers to stray currents.

If the articles on the bottom of the rack come out better than those at the top, one of several conditions exists and should be corrected:

1. Stratification in the bath; mix with mild air bubbling or stirrer to provide uniform temperature and solution composition.
2. Higher current density because of too long cathode placement.
3. Top articles on rack are too near the surface; provide at least 3 inches of bath above the top work row on the rack.
4. Stray currents are robbing the work.

Generally, 5 to 10 minutes will produce acceptable luster and smoothness on stampings from a good no. 2 or better finish for sheet, or the equivalent or better in strip. Orange peel from forming, and score marks from dies (or drawing in the case of wire), should be slight, otherwise a longer treating time will be required.



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After maximum luster is obtained in 5 to 10 minutes, continued treatment improves the luster very little except as benefited by any added smoothing that is accomplished. The smoothest surface by electropolishing will be obtained with the finest grained and "cleanest" metal. Fine grain size is required for avoiding "orange-peel" condition in cold formed sheet or strip stock and for avoiding the excessive "texture effect" visible in the surface after electropolishing.

If electropolishing and test results indicate too low a specific gravity and water is to be evaporated out, remove stainless steel cathodes before raising the tank temperature. At specific gravity below about 1.67, types 316 and 317 stainless steel cathodes may be severely attacked at temperatures exceeding 200°F.

If electropolishing and test results indicate too high a specific gravity, then water is carefully added in small increments to the bath to bring the specific gravity into the proper range. The water addition should not exceed 5% by weight.

Titration Method

The following methods are intended for routine bath control. In special cases where speed and simplicity are of secondary importance, more precise methods may be desirable. Some interference that may be encountered due to the build-up of foreign ions in the bath is discussed in notes at the end of the control procedures.

Reagents and Solutions Required

Hydrochloric Acid (1:3)	1 volume HCl (sp. gr. 1.19); 3 volumes H ₂ O
Chromic Acid Mixture	320 mL of H ₂ SO ₄ (1:1); 80 mL of H ₃ PO ₄ ; 600 mL of H ₂ O
Manganous Sulphate Solution, 5%	5g of MnSO ₄ dissolved in 100 mL of H ₂ O
Silver Nitrate Solution, 0.25%	2.5g of AgNO ₃ dissolved in 1000 mL of H ₂ O
Standard Ferrous Ammonium Sulphate (0.1N) Approx.	40g of FeSO ₄ (NH ₄) ₂ SO ₄ •6H ₂ O and 100 mL of H ₂ SO ₄ (1:1) diluted to 1000 mL
Standard Potassium Permanganate	3.3g of KMnO ₄ in 1000 mL of H ₂ O. Let stand several days and siphon into a clean container. Standardize against Bureau of Standards sodium oxalate.
Sulphur Acid (1:1)	1 volume of H ₂ SO ₄ (sp. gr. 1.84); 1 volume of H ₂ O
Ammonium Persulfate Solution	15g (NH ₄) ₂ S ₂ O ₈ dissolved in 85 mL H ₂ O



Standard Potassium Permanganate	3.2 g. KMnO ₄ per liter of solution. Standardize against Bureau of Standards Sodium Oxalate
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Analytical Procedures

1. Transfer approximately 15g of sample (weighed to the nearest milligram) to a 500 mL volumetric flask. Dilute to the mark and mix well.

Iron

1. Transfer a 100 mL aliquot to a 400 mL beaker.
2. Add 100 mL of hot water and 10 mL of H₂SO₄ (1:1).
3. Place a strip of aluminum in the solution and boil one-half hour.
4. Cool, remove the aluminum, and titrate immediately with 0.05N KMnO₄.
5. A reagent blank should be carried through all steps of the procedure.

Calculations

$$1 \text{ mL of } 0.05N \text{ KMnO}_4 = 0.00279 \text{ grams Iron}$$

$$(0.00279 \times \text{mL of KMnO}_4) / 3 \times 100 = \% \text{ Fe}$$

Chromium

1. Transfer an approximately 0.5g sample (taken as an aliquot from the dilution of a large sample) to a 600 mL beaker.
2. Add 60 mL chromium acid mixture, 10 mL 0.25% AgNO₃ solution, and 1 drop 5% MnSO₄ solution.
3. Dilute to 300 mL with hot water, heat to boiling and add 20 mL (NH₄)₂S₂O₈ solution.
4. Boil 10 minutes after the red MnO₄ color develops.
5. Add 5 mL HCl (1:3) and boil 5 minutes after the red color has disappeared.
6. Cool, add 25 mL titrating mix, and add standard 0.1N ferrous ammonium sulphate solution until the solution takes on a greenish coloration.
7. Add 3 drops Orth phenanthroline indicator and continue the addition of ferrous ammonium sulphate until the indicator assumes a red color, adding several mL in excess. It is convenient to add an amount of ferrous ammonium sulphate that is evenly divisible by 10.
8. Titrate the excess with standard KMnO₄ solution to a clear green endpoint.
9. Record the KMnO₄ required as titration A.
10. Add to the same solution the same amount of ferrous ammonium sulphate that was initially added and again titrate to the clear green endpoint with KMnO₄.
11. Record the KMnO₄ required as titration B.

Calculations

$$1 \text{ mL } 0.1N \text{ KMnO}_4 = 0.001734 \text{ grams Chromium}$$

$$\frac{(\text{Titration B} - \text{Titration A}) \times \text{Cr value of } 1 \text{ mL of KMnO}_4 \times 100}{\text{wt. of sample, grams}} = \% \text{ of Cr in the sample}$$

Hexavalent Chromium

1. Transfer an approximately 0.5g sample (preferably an aliquot from the dilution of a large sample) to a 600 mL beaker.
2. Add 600 mL chromium acid mixture, 300 mL cold water, and 5 mL HCl (1:3).



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3. Titrate as described in the determination of total chromium.

Trivalent Chromium

Calculation

$$\% \text{ Total Chromium} - \% \text{ Hexavalent Chromium} = \% \text{ Trivalent Chromium}$$

Caution

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