



Technical Process Bulletin

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ALODINE® 1600
 Conversion Coating Process for Aluminum

1. Introduction:

ALODINE 1600 is a concentrated liquid two-package chemical used to produce a chromate conversion coating on aluminum and all its alloys. The color ranges from light iridescent gold to tan. ALODINE 1600 can be applied by immersion or spray method and it does not contain complex cyanides. Alodine 1600 is approved for use under Mil-DTL-81706A, formally Mil-C-81706, Classes 1A and 3, Form, I, Method C and BAC-5719.

2. Operating Summary:

<u>Chemical</u>	<u>Bath Preparation per 100 gallons:</u>
ALODINE 1600	0.65 gallons (2.5 liters)
ALODINE 1660 Additive	1.3 - 2.0 gallons (5.9 - 7.6 liters)
ALODINE® 22 TONER	1 to 1½ gallons (3.8 to 5.7 liters)
<u>Operation and Control:</u>	
ALODINE 1600 (Concentration)	7.4 to 11.4 ml
Relative Fluoride (Orion)*	-70 to -85 (mV*)
pH	1.5 to 2.1
Temperature, °Fahrenheit	60° to 130° (15 to 55°C)
Time, minutes	1 to 5

*NOTE: The relative fluoride value stated above is not a bath control and it should only be monitored for trouble shooting purposes.

3. The Process:

The complete process sequence for the ALODINE 1600 treatment normally consists of the following steps:

- A. Non-Etch Cleaning
- B. Water rinsing
- C. Deoxidizing for Mil-C-5541, Class 3; optional for Class 1A
- D. Water rinsing
- E. Treating with the ALODINE 1600 processing solution
- F. Water rinsing
- G. Post treatment (optional)
- H. Drying

4. Materials:

ALODINE 1600
ALODINE 1660 Additive
ALODINE 22 TONER
Nitric Acid, dilute
Sodium hydroxide, dilute
Testing Reagents and Apparatus

5. Equipment:

Process tank and housing should be fabricated from stainless steel, such as 316L or 304L. The 316L being preferred for maximum tank life. In all cases, approved welding techniques must be used. Polyvinyl chloride (PVC) or CPVC lined mild steel can be used. In spray applications, nozzles fabricated from 300 series stainless steel are preferred.

Process piping and pumps should be constructed of 316 or 304 stainless steel alloys. Various formulations of plastic pipe may be used with recommended support spacing, Schedule-80 being generally recommended. PVC Type I is limited to maximum process temperatures of 140° Fahrenheit. CPVC and PP may be used up to a maximum process temperature of 190° Fahrenheit. PVDF may be used for all expected operating temperatures.

Heat exchanger plates should be polished 316 stainless steel. Gas fired burner tubes are not recommended. All process circulation pump seals, valve seats, door seals, etc., which come into contact with the process solution and occasional acid equipment cleaners, should be EPDM, Viton™ or Teflon™.

Chemical feed pump parts and other elastomers which may come into contact with the concentrated replenishing chemical should be Viton or Teflon.

Support equipment available from Henkel Surface Technologies for this process includes: chemical feed pumps, level controls, transfer pumps and bulk storage tanks.

Our sales representative should be consulted for information on Henkel Surface Technologies automatic process control equipment for this process and any additional questions. In addition, the "Henkel Surface Technologies Equipment Design Manual" may be consulted.

6. Surface Preparation:

Cleaning:

All metal to be treated with the processing solution must be free from grease, oil and other foreign matter before the treatment. A complete line of cleaners is available and our representative should be consulted.

Water Rinsing:

After cleaning, the metal must be thoroughly rinsed with water. The rinse should be overflowed continuously at a rate which will keep it clean and free from scum and other contamination.

Deoxidizing (optional):

When aluminum to be treated with ALODINE 1600 processing chemical for Class 3 coatings or the surface has corrosion products or heavy oxides, it should be conditioned by installing two additional stages between the post cleaner rinse and the ALODINE 1600 treatment step. One is for deoxidizing with a deoxidizing chemical and one is for an additional cold water rinse.

7. Treating with the ALODINE 1600/1660 Processing Solution:Buildup:

Acid proof equipment is required. Fill the tank about three-fourths full with water, temperature 60° to 130° Fahrenheit. For each 100 gallons (378.5 liters) of final solution volume, add 1.3 to 2.0 gallons (5.9 to 7.6 liters) of ALODINE 1660 Additive and circulate or thoroughly mix, add, 0.65 (2.5 liters) gallons of ALODINE 1600 and thoroughly mix; add 1 to 1 1/2 gallons (3.8 to 5.7 liters) of ALODINE 22 TONER while mixing. Finally, add sufficient water to bring the solution up to the working level and then heat, if necessary, to the desired operating temperature.

Operation:

Time: 1 to 5 minutes
Temperature: 60° to 130° Fahrenheit

Each alloy reacts with the ALODINE 1600/1660 solution to produce a coating that is characteristic of that alloy. The bath should produce a light, iridescent, gold to tan coating within the operating conditions selected.

Under most operating conditions **it is recommended to replenish with the ratio of ALODINE 1600 and ALODINE 1660 Additive as used in buildup** (i.e. for every 1 gallon (3.8 liters) of ALODINE 1600 add 2.0 to 3.1 gallons (7.5 to 11.7 liters) of ALODINE 1660).

ALODINE 22 Toner is usually only needed for initial buildup of a fresh ALODINE 1600/1660 bath.

When first charging a new bath it is desirable to process about 10 ft² per 100 gallons of bath of clean scrap aluminum or aluminum foil for 15 minutes to break in the bath.

If the Coating is Powdery:

- a. The pH of the bath may be too low for the bath concentration or the bath activity selected.
- b. The work may have been improperly cleaned and/or rinsed.
- c. The ALODINE 1600 bath may have become contaminated with phosphates, sulfates, or some other contaminant. In most cases, phosphate contamination will stop the coating formation completely.
- d. The bath temperature may be too high.
- e. The process time may be too long.
- f. The fluoride level ALODINE 1660 Additive may be too high
- g. The transfer time between the treatment and the water rinse may be too long.

If the Coating Weight is too low:

- a. The temperature of the ALODINE 1600 bath may be too low for the method or process time selected.
- b. The process time may be too short

- c. The concentration of the bath may be too low.
- d. The pH of the bath may be outside of the proper range.
- e. The fluoride level (ALODINE 1660 Additive) might be too low.

8. Testing and Control:

Never pipet by mouth, use a pipet filler.

ALODINE Titration:

Pipet a 5 ml sample of the ALODINE 1600 bath into an iodimetric flask and dilute to approximately 100 ml with water. Add approximately 1 gm (1/2 teaspoonful) of Reagent 2 and agitate the solution until the compound is dissolved. Add approximately 10 ml of Reagent Solution 49 in 5 ml increments to the lip of the flask, raising the stopper slightly after each addition to allow the acid to run into the flask. Rinse the lip several times with water and replace the stopper. Let sit for approximately one minute. Titrate with Titrating Solution 104 until a straw color is obtained. Do not rezero the buret. Add several milliliters of Indicator 10 to the sample and continue the titration until the blue-black color disappears. The milliliters of Titrating Solution 104 used is the ALODINE titration.

ALODINE titration range: 7.4 - 11.4 ml.

To increase the ALODINE titration value by 1.5 ml: add 0.1 (0.4 liters) gallons of ALODINE 1600 per 100 gallons of working bath.

pH Adjustment:

Always make any needed additions of water, Alodine 1600 and Alodine 1660 to the bath and allow for complete mixing before adjusting the pH.

If needed, dilute sodium hydroxide (caustic) or dilute ammonium hydroxide can be used to raise the pH of the solution. Dilute nitric acid can be used to lower the pH of the bath.

Fluoride Activity Measurement:

The method of measuring fluoride activity makes use of a standardized Orion Fluoride Ion electrode and an Orion Meter or an equivalent instrument capable of measuring relative Millivolts.

Immerse the Orion Fluoride Ion Electrode and the reference electrode into Standard Solution 120MC. Using the expanded relative millivolt scale, set the meter to zero.

Remove the electrodes from the activity Standard Solution 120MC, rinse with distilled water and dry.

Cool a sample of the bath to the same temperature as the Standard Solution 120MC used for the electrode standardization. Immerse the electrodes into the bath sample and record the relative negative Millivolts.

The product is designed so that as long as the relative millivolts is maintained between -70 and -85 excellent salt spray is observed. Fluoride activity is monitored only, and under normal bath operation it is **not a control point**.

9. After Treatment:

Water Rinsing:

After treating with ALODINE 1600 the work should be thoroughly rinsed in clean cold water. The rinse should be continuously overflowed such that the main body of the rinse never becomes excessively contaminated.

Post Treatment:

The ALODINE 1600 treated metal may be treated with an acidic post treatment solution. The post treatment adds to the overall quality of the coating and prevents the formation of hard water salts blistering. In some cases the post treatment stage may be heated to facilitate work drying.

Drying:

Parts coming from the final water rinse or post treatment stage can be dried in an indirectly fired oven or by any other means which will not contaminate the metal with fumes, oil, or partially burnt gases. Peak metal temperature should not exceed 120° Fahrenheit.

10. Storage Requirements:

No special storage requirements are required for ALODINE 1600, ALODINE 1660 Additive or for ALODINE 22 Toner. If any of these products were to freeze after extended storage at a low temperature, thaw in a warm place and very carefully stir thoroughly (until no solids are observed) before use.

11. Waste Disposal Information:

Applicable regulations covering disposal and discharge of chemicals should be consulted and followed.

Disposal information for ALODINE 1600, ALODINE 1660 Additive and ALODINE TONER 22 is given on the Material Safety Data Sheet for each product.

The processing bath is acidic and contains chromium and complex fluorides. Waste treatment and neutralization will be required prior to discharge. (Refer to Waste Treatment Information Bulletin WT1004, available on request.)

12. Precautionary Information:

When handling the chemical products used in this process, the first aid and handling recommendations on the Material Safety Data Sheet for each product should be read, understood and followed.

The processing bath is strongly acidic and can cause irritation of the skin and eyes. Do not get in eyes or on skin or clothing. In case of contact, follow the Emergency and First Aid Procedure recommendations on the Material Safety Data Sheets for ALODINE 1600 and ALODINE 1660 Additive.

The processing bath contains chromic acid in excess of 0.1% when prepared as recommended. The following statement, or one similar to it, should be included as part of the process tank labeling [29 CFR 1910.1200(f)(4)].

"POSSIBLE CANCER HAZARD, CONTAINS CHROMIC ACID WHICH MAY CAUSE CANCER BASED ON ANIMAL DATA. Risk of cancer depends on duration and level of exposure."

Refer to attached Material Safety Data Sheet for additional information.

Testing Reagents and Apparatus
 (Order only those items which are not already on hand.)

<u>Code</u>	<u>Quantity</u>	<u>Item</u>
205700	1	Buret Assembly, 25-ml Automatic, Glass
205897	2*	Flasks, iodimetric, 250-ml
205852	2*	Graduated Cylinder, 50-ml, Glass
205010	250 ml	Indicator 10 (0.5% starch solution)
205590	1	Indicator Dropping Bottle, 2 oz
205876	2	Pipet, 5-ml, plastic
205082	450 gm	Reagent 2 (KI)
205249	2.5 liters	Reagent Solution 49 (C.P. HCl)
205980	1	Thermometer, Floating
205104	1.0 liters	Titration Solution 104 (0.1N Na ₂ S ₂ O ₃)
--	1	Orion #9409BN, solid state F- electrode
--	1	Orion #900100, reference electrode
--	1	Orion #900011, reference filling solution
--	1	Orion Model 250A portable pH/mV meter (or equivalent)

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