ROHM AND HAAS

ELECTRONIC MATERIALS

PACKAGING AND FINISHING TECHNOLOGIES

INTERVIA™ 3D-P PHOTORESIST

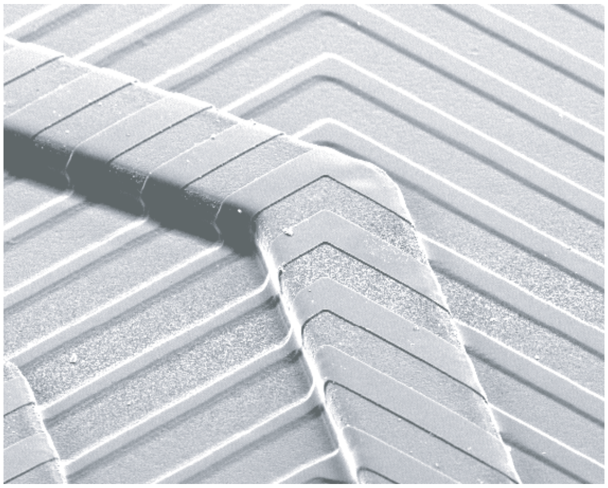
For Advanced Packaging Applications

Regional Product Availability

N. America	Japan/Korea	Asia	Europe
✓	✓	✓	✓

DESCRIPTION

InterVia 3D-P Photoresist is electrodeposited from an aqueous emulsion. It is anaphoretically deposited onto electrically-conductive substrates regardless of shape or geometric complexity. After coating, the substrate is baked to smooth the film, imaged and developed. After development, the part can be plated with most common plating chemistries or etched. After processing, the InterVia 3D-P Photoresist is easily stripped.



ADVANTAGES

- Applicable to a wide variety of substrate sizes and geometries
- Provides uniform, defect free coatings
- Aqueous emulsion
- Coating thickness capability of from 2–10 µm
- Exposure at *i*-line (365–405 nm) wavelength
- Development in common commercial developers
- Withstands common etch and plating chemistries
- Can be stripped in InterVia 3D-P Remover

PROCESS SEQUENCE

- Preclean
- Rinse
- InterVia 3D-P Photoresist
- Conservation rinse
- DI Rinse
- Forced air dry
- Bake
- Rehydrate coating
- Exposure
- Development
- Etch or plate
- Strip

INTERVIA 3D-P BATH MAKE UP

InterVia 3D-P Photoresist is packaged at 20% solids. It is recommended that the resist be diluted 1:1 with DI water to 10% solids using the procedure below. Prior to filling process-tanks with chemistry, the tanks should be thoroughly cleaned and rinsed. Please make sure all valves are closed. New 5 micron wound, unsized polypropylene filters should be used.

Add ingredients to a clean tank in the order listed below with constant mixing

InterVia 3D-P Photoresist:	50% by volume.
DI water <10 microS:	50% by volume

Turn on resist circulation system and allow to circulate for about four hours. Always analyze the bath for percent solids prior to operation and adjust solids if necessary. Analyze for TEA and adjust level as necessary. Check thickness. Heat to operating temperature.

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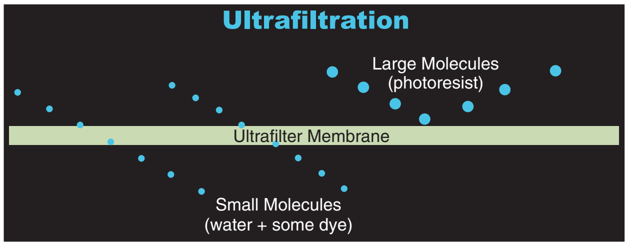
PERMEATE BATH MAKE-UP

If a conservation rinse is available on the equipment, the residual resist solids from dragout can be removed. *(Figure 1)* The resist solids are re-concentrated by the Ultrafilter and returned to the resist coating tank.

If a permeate bath is used for conservation after coating, fill clean tank with high quality DI water. Add NMP to 1.5% of the volume of the Permeate bath. Never add NMP directly into the InterVia 3D-P Photoresist working solution. Always make the addition to the Permeate bath.

Figure 1. Ultrafiltration of Residual Resist solids



INTERVIA 3D-P PHOTORESIST PRODUCT OPERATION

PRODUCTION SCALE COATING

Pre-Clean

Prior to entering the InterVia 3D-P Photoresist, parts must be thoroughly clean. Failure to use clean parts can result in resist contamination and uneven coatings. If the part has been freshly plated, make sure that it is thoroughly rinsed with DI water prior to entering the coating bath. If cleaning is necessary, the type of cleaning will depend on the surface metal. Your Rohm and Haas Electronic Materials representative can help you make the selection of chemistry for cleaning. Rinsing after cleaning is essential. The resist can become contaminated if the part is not rinsed thoroughly with DI water. Parts with complex 3-D structures will need longer rinsing.

Coating

InterVia 3D-P Photoresist is applied by anodic electrodeposition *(Figure 2)*. Upon application of a direct current, charged micelles containing all the components of the resist migrate to the conductive substrate to form a uniform coating *(Figure 3)*. Coating thickness is dependent on temperature as well as substrate as seen in *Figure 4*.

Figure 2. Electrodeposition of the Negatively Charged Micelle

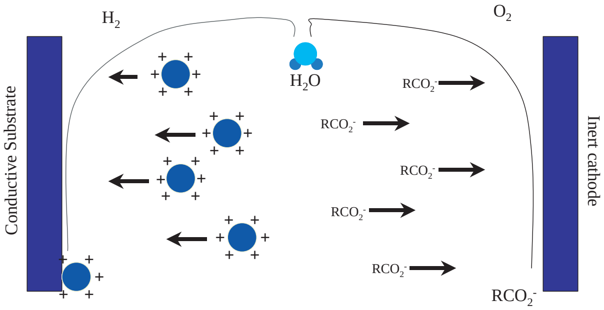


Figure 3. InterVia 3D-P Photoresist Micelles coating onto a part

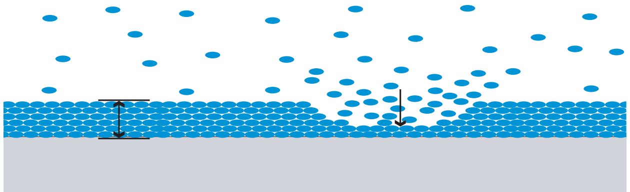
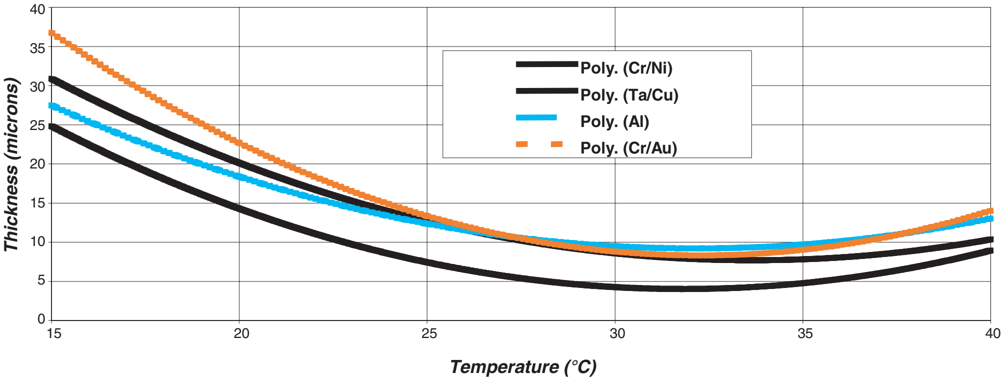


Figure 4. InterVia 3D-P Photoresist Temperature vs. Thickness on Spurttered Metal



Operating Instructions

It is important to maintain solution level with DI water.

Operating Conditions

Solids content:	9.5–10.5 %
Conductivity:	400–500 microS/cm
Voltage:	100–250 Volts
Current:	1.0 A/dm^2 (10 A/ft^2) peak
Circulation:	Recommended Good circulation while parts are submerged is one method to prevent pinholes
Temperature:	Thickness is dependent on temperature *(see Figure 1)* Temperature control should be within ±1 degree C
Wetting time:	40 seconds
Coating time:	Potential is applied for 20 seconds.
Ventilation:	Required
Filtration:	A 5 micron wound, unsized polypropylene filter should be used prior to the ultrafilter

Monitor resist thickness using the supplied procedures. Resist thickness is controlled by additions of InterVia 3D-P TC. Make all replenishments into the resist overflow sump very slowly (drop-wise) with the circulation on. The favored method of addition is automatic. A consistent need to make large additions of InterVia 3D-P TC indicated that the operation of the system needs to be checked or adjusted.

InterVia 3D-P TC and NMP should be analyzed daily. This will help establish an automatic replenishment schedule. TEA should be monitored once a week.

Rinse

The final rinse is a DI rinse.

Dry

To ensure uniform baking, all drops of water should be blown off before baking. Air knives or an air gun can be used. Ensure the air used is water and oil free. Water spots when baked form areas which develop slower than surrounding resist.

Bake (Convection or hotplate)

The coating is dry once the water is blown off, but needs to bake to coalesce the micelles.

This can be done in a convection oven or on a hotplate. Since the purpose of the bake step is to smooth the micelles, there is not a time advantage to baking on a hotplate.

Convection

Ensure the ovens are free from foreign matter and debris. Verify the calibration of the temperature controllers on a monthly basic. Verify the temperature uniformity of the oven on a monthly basis.

Bake Temperature:	90–105°C
Bake Time:	8–15 minutes
Ventilation:	Required

Hotplate

Temperature:	90°C
Bake time:	8–15 minutes

Exposure

The coatings must be equilibrated in 40–60% humidity before exposure. Exposure under conditions of less than 40% will result in an inability to develop an image.

InterVia 3D-P Photoresist is sensitive at 365–405 nm. Mercury lamps are suited for this wavelength. Many different exposure devices can be used. Printer Intensity should be >20 mΩcm^2.

Exposure time is dependent on the exposure unit used. On a typical 7kW printer 10–25 seconds can be expected. Exposure dose is about 400 mJ/cm^2. Radiometers can be used to estimate exposure dose and monitor the output.

Development

Development should be done in yellow light conditions.

Operating Conditions

InterVia 3D-P Developer at 35°C. Under normal circumstances, the exposed resist will clean in 60–90 seconds. Additional time may be required for smaller features to be cleared. Rinsing after develop should be thorough. Ventilation is required. It is suggested that the developer be filtered with a 10 micron filter. If used in batch mode, the developer should be replaced when the clear time goes over 2 minutes.

Stripping

Operating Conditions

InterVia 3D-P Remover at 55°C. Under normal circumstances strip time should be 30–60 seconds. Ventilation is required. It is suggested that the stripper be filtered with a 10 micron filter. The stripper should be replaced when the clear time goes over 2 minutes.

Clamp Strip

To ensure good electrical contact during coating, the clamps must be kept clean by stripping off residual resist. Prior to makeup of the clamp strip, check that materials of construction are compatible. Compatible materials include: reinforced polyethylene, polypropylene, stainless steel and Teflon™ fluoropolymer.

Suggested Clamp-Strip make-up

0.6–0.9% (0.28–0.32N) NaOH at 50–60°C

Deionised water:	70 parts by volume
Cuposit™ Z:	3.4 parts by volume
Deionised water:	to 100 parts total

Analyze and adjust concentration

Heat to 50–60°C.

Residual Resist should be removed in 3–5 minutes.

Other grades and forms of sodium hydroxide can be used with appropriate adjustments in amounts.

Control limits

Normality:	0.28–0.32N

BATH REPLACEMENT

InterVia 3D-P Photoresist has a 6-month shelf life in the concentrate as made if stored between 40–60°F. Under normal circumstances, there should be no reason to replace the InterVia 3D-P Photoresist except in cases of contamination. Throughput should be sufficient to completely replenish the ED bath once every two months.

In the event of low throughput, sufficient volumes of the ED bath will regularly need to be removed and replaced with freshly made up resist solution. This will ensure that the ED resist performs at its optimum.

Bacterial Contamination.

Bacteria from the DI water can contaminate the resist bath and multiply. The bacteria take nourishment from the photoactive compound and as a result, the bath life will be shortened greatly. Decreased photospeed, increased development times and increased unexposed film loss can be caused by bacterial contamination.

PREVENTIVE MAINTENANCE

- Maintain solution volume with DI water.
- Do not allow solution drag out or spills to dry
- Check flow rates through the permeate system
- Every week check the pre-filters for clogging and change if necessary

LAB-BENCH SCALE COATING

The following is a suggested set-up for coating InterVia 3D-P Photoresist on a small scale at a lab bench

Power Supply

The power supply should have a voltage range of 0–150 Vdc and an amp range of 0–7

(A suggested power supply for lab use is the Sorensen DCS 150-7) The power supply should have zero ripple. The power supply is operated at constant voltage mode where the output voltage is regulated and the output current varied with the load requirement.

Coating Container

The size of the beaker used depends on the size of the part being coated.

Possible containers for the resist are glass 4-liter beakers, Aldrich chromatography beakers, or plastic TriPour beakers.

For bench-scale coating for feasibility studies, filtration or ultrafiltration is not needed. However, coating without filtration can lead to coatings with more dirt on them. A 5-micron filter and a small diaphragm pump are recommended if filtration is desired.

Sample and Counter-electrode Holder

A modified pH probe arm can be wired for electrodeposition. A 1/4" polypropylene block can be glued to the probe holder and copper alligator clamps screwed to the polypropylene as contacts for the lead from the power supply. Clamps are used to hold the cathode and anode. Stainless steel (either 316 screen or solid 316) is used as the counter-electrode. Anode to cathode distance is about 2 inches. The set-up should be placed in a well-ventilated area. A hood is suggested.

Double safety interlocks are recommended including a safety interlock that requires the hood sash to be down in order for the power supply to operate. Safety First.

Temperature Control

The easiest way to maintain temperature control is by placing the beaker containing the resist into a water bath at a temperature which will result in the desired resist thickness.

Resist Make Up

InterVia 3D-P Photoresist is diluted 1:1 with very clean DI water. Ensure the resist is thoroughly mixed and let come to the desired temperature.

Wafer cleaning:

The metallization on the part should be fresh and clean. Depending on the condition of the metallization on the part, the part may need to be rinsed with clean DI water before coating.

Operation

The part to be coated is submerged into the resist with manual agitation for about 40 seconds prior to coating. The part is connected to a clamp. **Warning!** Double safety interlocks should be used to ensure that there should be no way contact can be made with any part of the body.

Voltage:	100–200V
Coating Time:	10–20 seconds.
Rinse:	DI water
Blow dry:	Air gun (water-free and oil-free air)
Bake:	Convection oven (90–105°C for 8–10 minutes)

Make sure the clamps are cleaned after each use. The container should be covered as much as possible to limit TC loss. TC additions should be made drop-wise with good stirring. Allow 1–2 hours for incorporation.

Note: At the lab-bench scale, the bubbles generated can be a restriction to coating. A 10–15 minutes wait between coating each wafer to get rid of the bubbles from the solution may be necessary.

ANALYSIS

- Solids
- Conductivity
- Thickness
- Unexposed loss

SOLIDS ANALYSIS

DETERMINATION OF SOLIDS CONTENT

I. Principle

The solids content of the InterVia 3D-P Photoresist is determined gravimetrically by drying a known mass of solution and determining the mass lost.

II. Equipment needed

a) Forced air convection oven

b) Analytical balance

c) Aluminum weighing dishes

III. Procedure

a) Weigh one dry empty aluminum weighing-dish to obtain a tare weight. (W_1)

b) Weigh approximately 2 grams of InterVia 3D-P Photoresist from the tank into dish (W_2).

c) Place sample in a convection oven at 105°C. Bake samples for 2 hours at 105°C.

d) Remove the dish and allow to cool in a desiccator.

e) Reweigh the cooled dish containing the residues (W_3).

IV. Calculation

$$\text{Solids }(\%) = \frac{(W_3 - W_1) \times 100\%}{(W_2 - W_1)}$$

V. Replenishment

Solids Replenishment (100 liter volume)

Solids Content %	InterVia 3D-P Addition
11.0	—
10.5	—
10.0	—
9.5	2.5 liters
9.0	5.0 liters
8.5	7.5 liters

DETERMINATION OF CONDUCTIVITY

I. Principle

The conductivity of a solution is determined using a commercially available conductivity meter.

II. Material/Equipment

Conductivity meter calibrated for use in the range of 0–800 µS/cm^2

III. Procedure

a) Place approximately 100 ml of working bath into a 250 ml beaker and allow the sample to equilibrate to 25°C.

b) Calibrate the conductivity probe in accordance with the manufacturer's instructions.

c) Rinse the conductivity probe thoroughly with alcohol and deionised water.

d) Immerse the conductivity cell into the solution and measure the conductivity.

e) Rinse probe and store in accordance with manufacturer's instructions.

IV. Thickness

Thickness can be measured using a profilometer or by interferometry. Special programs may need to be installed in order to measure thick resists.

Cauchy Coefficients may be needed for thickness measurement on some equipment.

Cauchy Coefficients

Resist	n_1	n_2	n_3
InterVia 3D-P Photoresist	1.513	6.4E+5	-3E+12

GC PROCEDURE FOR ANALYSIS OF INTERVIA 3D-P PHOTORESIST TC AND NMP

I. Principle

InterVia 3D-P Photoresist TC and NMP contents should be analyzed by gas chromatography.

II. Reagents and Apparatus

a) InterVia 3D-P Photoresist TC standard sample

b) N-methyl-2 pyrrolidone

c) Ethylene glycol mono butyl ether

d) Acetone—HPLC-grade or equivalent

e) Analytical balance

f) 10 µliter micro-syringe

g) 100 ml volumetric flask

h) Gas chromatograph—Varian 3600, 6000 or equivalent

i) DB-5 capillary column-1 µm thick phase, 30 meter x 32 mm I.D

j) GC autosampler—Varian Vista 8034 or equivalent

k) Integrator—SpectraPhysics Data Jet CH1 or equivalent

III. GC Parameters

Column type:	10 feet, 10% FFAP CW80-100AW
Column temperature:	150°C
Injection temperature:	270°C
Detector temperature:	300°C
Detector type:	FID
Carrier gas flow:	N_2 30 ml/min.

IV. Procedure

a) Accurately weigh about 0.2g of 2-octanone, 0.1g N-Methyl-2 Pyrrolidone and 0.15g of ethylene glycol mono butyl ether into a tared 100 ml volumetric flask, dilute to mark with acetone and mix. Label flask as standard.

b) Pipette 10 ml of sample into a 100 ml volumetric flask using a TC pipette. Rinse pipette into flask with acetone. Accurately weigh about 0.15g of ethylene glycol mono butyl ether into the flask. Dilute to mark with acetone and mix. Label as sample.

c) Set GC temperature program to 160°C isothermal, with a splitter flow rate of 60 ml/minute (Varian 6000) or 20 ml/min. (Varian 3600).

d) Set integrator peak threshold to 1,000 and peak width to 3. Attenuation should be set according to detector response (~32).

Note: Integrator conditions and splitter flow may vary based on instrument types and column age.

e) Using autosampler inject 1 µl of the standard into the column (perform in duplicate). Record areas of the ethylene glycol mono butyl ether (~2.87 min.), the InterVia 3D-P Photoresist TC (~3.32 min.), and the n-methyl-2 pyrrolidone (~3.95 min.).

f) Inject 1 µl of sample onto the column (perform in duplicate). Record the areas of the three peaks.

V. Calculation

RF (InterVia 3D-P TC) =

$$\frac{\text{area (Ethylene Glycol Mono Butyl Ether) x std x wt (InterVia 3D-P TC) in std}}{\text{wt (Ethylene Glycol Mono Butyl Ether) in std X area (InterVia 3D-P TC) in std}}$$

RF n-methyl-2 pyrrolidone =

$$\frac{\text{area (Ethylene Glycol Mono Butyl Ether) x (std) x wt (n-methyl-2 pyrrolidone) in std}}{\text{wt (Ethylene Glycol Mono Butyl Ether) in std X area (n-methyl-2 pyrrolidone) in std}}$$

InterVia 3D-P TC% =

$$\frac{\text{area (InterVia 3DP TC) in sample x RF (InterVia 3DP TC) x wt (Ethylene Glycol Mono Butyl Ether) in sample x 10}}{\text{area (Ethylene Glycol Mono Butyl Ether) in sample}}$$

n-methyl-2 pyrrolidone % =

$$\frac{\text{area n-methyl-2 pyrrolidone in sample x rf n-methyl-2 pyrrolidone x wt. ethylene glycol mono butyl ether in sample x 10*}}{\text{area ethylene glycol mono butyl ether in sample}}$$

*10 = dilution factor

It is suggested that NMP level be 1.0–1.5 %

DETERMINATION OF TEA LEVEL INTERVIA 3D-P PHOTORESIST ANALYSIS

I. Item

Free amine value

II. Principle

A sample is precipitated with alcohol and titrated to a pH 4.8 with hydrochloric acid.

III. Procedure

a) Weigh into a 300 ml beaker approximately 5g

b) Add approximately 100 ml of isopropyl alcohol

c) Titrate with 0.1N HCI to pH 4.8.

IV. Calculation:

Amine value (mEq/g)=HCl (ml) x Normality(N)/Sample weight W_1 (g)

Control range: 0.018~0.024 mEq/g

Replenishment: ED Tank (For 800 liter bath)

TEA (g) =(0.021 Result) x 800 x 149.2

Replenishment: Conservation Rinse Tank

TEA (g) =((0.021 Result) ÷ 3.5) x 800 x 149.2

V. Replenishment

Additions of InterVia 3D-P Photoresist TC should be made according to thickness volume achieved once all other bath parameters have been optimized.

GC analysis results should only serve to monitor the level of TC in the bath.

InterVia 3D-P Photoresist TC Replenishment Schedule for 100-liter volume

Thickness is also dependant on TEA content and temperature. Ensure both are at optimum conditions.

FACILITIES

Rohm and Haas Electronic Materials Recommends coating of substrates with InterVia 3D-P Photoresist and exposure of parts take place in at least a class 10K clean room under yellow light. The class of clean room needed depends on the feature size being resolved. Temperature control is not critical, and may be adjusted to minimize impact on the phototools. Humidity must be maintained in the exposure area between 40 and 60%.

Developing, etching/plating, and stripping can be done under normal clean conditions. The developing area should be equipped with yellow lights.

EQUIPMENT

The coating process should be carried out in a piece of coating equipment designed for use with ED resists and certified by a Rohm and Haas Electronic Materials representative. Contact your Rohm and Haas Electronic Materials representative for suggested manufacturers.

Ensure tank is clean and contains only compatible material.

Materials of construction: Compatible materials are polyethylene, polypropylene, stainless steel, Teflon fluoropolymer, EPDM, and Kalrez™ perfluoroelastomer.

Incompatible Materials: PVC, CPVC, TYGON, and Polyurethane.

Heaters:	Heaters should be of low watt density with good bath circulation to avoid overheating; use of heat exchangers is preferable
Anode Material:	316 Stainless Steel is recommended
Anode to Cathode Ratio:	1:1
Rectification:	A 0–250V power supply is recommended; amperage is not limited and is approx. 3–4 amperes per square foot; **WARNING!** Refer to Handling Precautions prior to making up bath **WARNING!** At least two safety interlocks should be included in the rectification system

New equipment should be leached with a solution of 3% n-methyl-2-pyrrolidone (NMP). This solution should be allowed to circulate through the entire resist system (resist tank, permeate tank, and ultrafilter) at least overnight. The system should then be drained and thoroughly rinsed with deionized water. See note on DI water quality below.

DI WATER QUALITY

Only very high quality DI water should be used for bath makeup, evaporation replenishment and rinsing. Pinholes and other coating defects are known to be caused by poor water quality. Conductivity should be <10 microS/cm. The DI water must be kept free of bacteria. This can be accomplished by running the water first through UV sterilization unit then through consecutive 0.2 and 0.1 micron membrane filters. Failure to use bacteria free DI water can shorten the life of the InterVia 3D-P Photoresist bath and cause failures in the resist.

PRODUCT DATA

For the specific Product Data values, please refer to the Certificate of Analysis provided with the shipment of the product(s).

HANDLING PRECAUTIONS

Before using this product, consult the Material Safety Data Sheet (MSDS)/Safety Data Sheet (SDS) for details on product hazards, recommended handling precautions and product storage.

CAUTION! Keep combustible and/or flammable products and their vapors away from heat, sparks, flames and other sources of ignition including static discharge. Processing or operating at temperatures near or above product flashpoint may pose a fire hazard. Use appropriate grounding and bonding techniques to manage static discharge hazards.

CAUTION! Failure to maintain proper volume level when using immersion heaters can expose tank and solution to excessive heat resulting in a possible combustion hazard, particularly when plastic tanks are used.

STORAGE

Store products in tightly closed original containers at temperatures recommended on the product label.

DISPOSAL CONSIDERATIONS

Dispose in accordance with all local, state (provincial) and federal regulations. Empty containers may contain hazardous residues. This material and its container must be disposed in a safe and legal manner.

It is the user's responsibility to verify that treatment and disposal procedures comply with local, state (provincial) and federal regulations. Contact your Rohm and Haas Electronic Materials Technical Representative for more information.

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