



## “VenPure® Decolorization of Epoxy Resins and Curing Amines”

The name VenPure® Polisher has become synonymous with chemical purification and color reduction in a variety of basic and specialty chemical applications. Through continued commitment to research, development and product improvement, Rohm and Haas Company is now able to offer its quality enhancing technology to the manufacturers of modified and unmodified epoxy resins. For only small additional treatment costs, this technology will allow producers of such products to increase the competitiveness of their resins by meeting their customers' increasing demands for low color/high performance materials.

Epoxy resins are used in applications such as coatings, laminates, adhesives, electrical coatings, tooling, casting, and molding resins. The advantages of epoxy resins include adhesion, corrosion resistance, durability, abrasion resistance, flexibility, low shrinkage, dimensional stability, and superior electrical properties.

Darker than desired color and color degradation over time are common problems observed with epoxy resins and may interfere with the use of these resins in color sensitive application areas such as thin film and clear coatings and adhesives. Efficient decolorization has the potential of opening new marketplaces for epoxies as well as making the resins more desirable in the current applications.

VenPure Polisher has been utilized effectively on laboratory, pilot, and commercial scales to prevent color formation and extend the low color life of epoxy resins. It aids in maintaining a low resin color throughout the manufacturing process.

VenPure Polisher acts by scavenging certain oxidized impurities present in epoxy resins. Oxidized impurities are known to be the cause of darkening color, partly due to an aldol condensation reaction that takes place during the manufacturing process.

**Any epoxies manufactured under base catalyzed conditions, such as glycidyl ethers and novolac resins are candidates for VenPure treatment.**

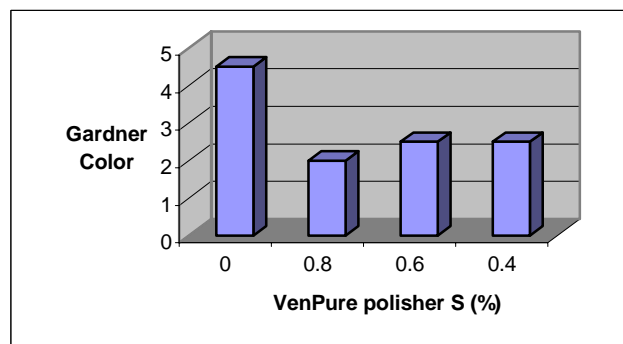
Rohm and Haas Company has done in-house laboratory testing on the ability of the VenPure

technology to reduce color in various epoxy resins. The results of the lab trials should be regarded as guidelines, which are amenable to optimization. Actually, due to the specificity of large-scale production of resins, we expect the results on production-scale to outperform the lab results displayed in this document.

### Bisphenol A / Epichlorohydrin

Bisphenol A / epichlorohydrin resins are the most widely used epoxy resins today. These resins are typically sold as a light amber color. Color reduction would be beneficial in order to extend work with these types of epoxies into underdeveloped markets such as coatings and embedments.

Rohm and Haas Company has manufactured this type of resin on a laboratory scale utilizing VenPure Polisher S treatment *during glycidylation*, at concentrations of 0.4 – 0.8% (based on epichlorohydrin weight) effectively reduced the final resin color from Gardner 4-5 to Gardner 1-2.



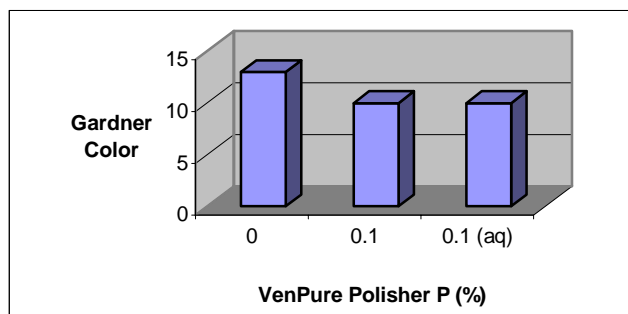
Another possible treatment is *prior to glycidylation*. In the production of bisphenol A glycidyl ether, 0.08% VenPure Polisher 2 has been pre-added to Bisphenol A. As compared to untreated Bisphenol A, the resulting BA glycidyl ether's color was reduced from 3 to 1 Gardner.

### Cresol/ Novolac

Cresol novolac resins are noted for their thermal stability and excellent chemical resistance. These resins are commonly used in such application areas as high performance aerospace composites, high temperature adhesives, and powder coatings. They are generally characterized as being extremely dark in color. As a result, any color relief would be

welcomed by a number of different marketplaces. Treatments both prior to and during glycidylation were performed on this resin with both being equally successful at reducing color.

Pretreatment of the raw materials with 0.1% VenPure Polisher P (based on epichlorohydrin weight) succeeded in reducing Gardner color from 13 to 10.

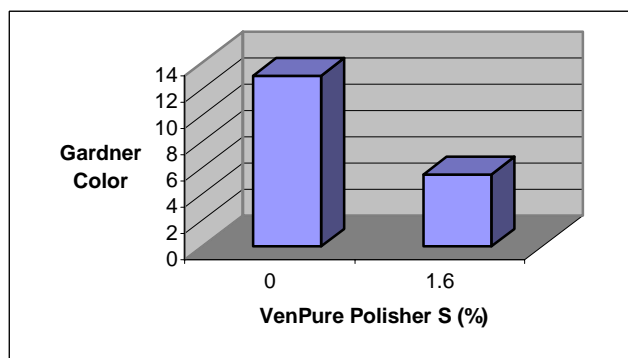


When 0.8% VenPure Polisher S (based on EPI weight) was added together with the aqueous NaOH glycidation catalyst, a Gardner color reduction from 13 to 10 was obtained.

A customer optimized our initial lab results into a treatment using 1.6% VenPure Polisher S to achieve a color specification of 100 APHA. VenPure is premixed into the 20% aq. NaOH catalyst solution.

### Resorcinol/Epichlorohydrin

These resins are noted for their rapid curing, low shrinkage upon cure, and good resistance to moisture and chemicals. They are used as casting resins and adhesives as well as to reduce the viscosity of some “epi-bis” resins for coating applications. Rohm and Haas Company manufactured this resin on a laboratory scale treating with 1.6% VenPure Polisher S during glycidylation (based on epichlorohydrin weight). The color was effectively reduced from Gardner 13 to Gardner 5-6.



### Curing agents

In order for epoxy resins to be useful in any of the applications previously described and to develop the inherent benefits associated with the resins it is necessary for the resins to be cured with cross-linking agents or hardeners. The reaction sites are the epoxy groups for liquid resins and the epoxy and hydroxyl groups for solid resins. Such curing agents or hardeners are classified as either catalytic, employing homo-polymerization as a consequence of a Lewis acid or base in the reaction, or co-reactive, employing poly-functional reagents with active hydrogen atoms, (i.e. polyamines).

Amines exposed to heat, air or light may degrade resulting in discoloration. The yellow, brown, and red colored species typically seen in amines can be removed by simple distillation but time and re-exposure to heat, air or light will cause discoloration reoccurrence. Significant discoloration can occur in as little as six days making extended shipment or storage impossible if low color, high quality amines are desired.

VenPure treatment has been found in both laboratory and commercial work to have dramatically impacted both the initial color and the retarded color degradation to extend low-color life of both aromatic and aliphatic amines. The dosage is typically less than 100 ppm of VenPure Polisher G. VenPure treatment of amines both before and after distillation have been tested and both show great benefit over no treatment.

### Aromatic Amines

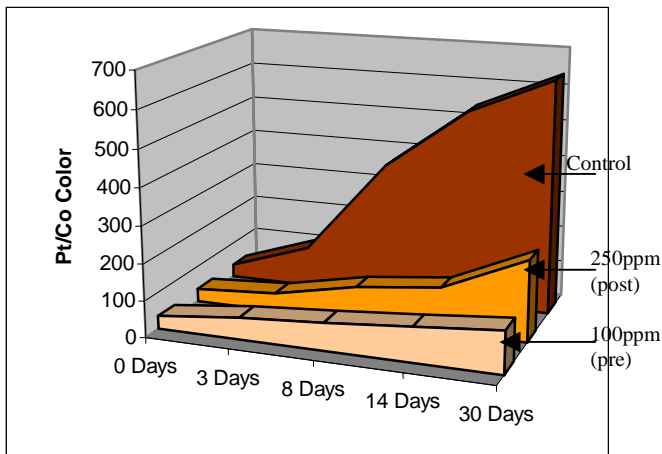
Most aromatic curing amines oxidize slowly in air or upon exposure to UV resulting in darker color amines and thereby darker cured epoxy resins. Many aromatic amines such as methylene dianiline are solids and therefore would be difficult to post-purify. These types of amines may be treated prior to distillation in the molten state, but purification of the raw materials is often a more beneficial option.

Methylene dianiline is produced by reacting formaldehyde with aniline under acid catalyzed conditions. If the aniline raw material is pre-treated with VenPure, then the discoloration of the resultant methylene dianiline will be inhibited.

In order to assess the effect of VenPure treatment on improving the color stability of aniline, laboratory samples with various treatment levels were stored

under air with exposure to UV and compared to an untreated control. Untreated samples reached 300 Pt/Co color levels within 8 days of initial distillation whereas samples treated with as little as 100 ppm VenPure Polisher P didn't reach this level even after 30 days.

Whereas VenPure treatment prior to distillation is the most efficient means of maintaining color stability in this amine, post-addition also significantly retards the color formation.

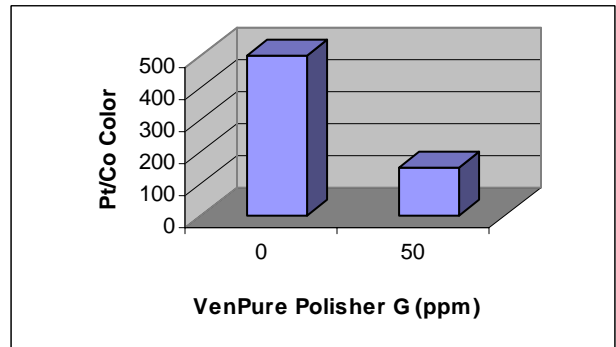


**Aliphatic Amines**

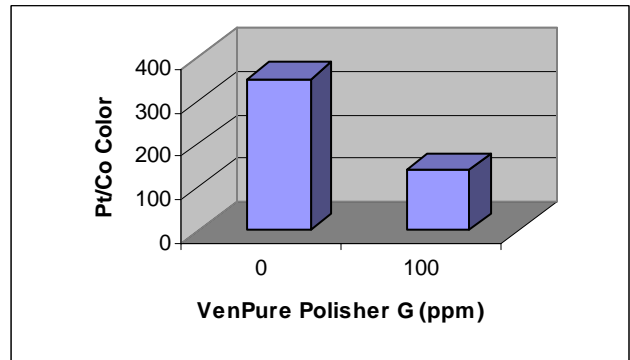
Aliphatic amines are widely used as epoxy curing agents as they allow curing of the epoxy at room temperature. Aliphatic amines such as ethyleneamines exhibit rapid and dramatic color degradation prohibiting long shipment or storage periods when low color products are desired.

Both diethylenetriamine (DETA) and triethylenetetramine (TETA) which are typically used as epoxy curing agents were directly treated with VenPure Polisher. Significant color improvements were registered versus their non-treated controls. In each case both the control and treated samples were stored for 48 hours at 90°C in an air atmosphere and color was then measured using Pt/Co color scale.

DETA samples treated prior to distillation with 0.005% VenPure Polisher G showed a color degradation of only 140 Pt/Co versus untreated samples of color greater than 500.



Preliminary studies of TETA purification show that VenPure™ post-treatment is the most effective method of color reduction. At levels of 0.01% VenPure Polisher G, TETA stored at 90°C under air atmosphere for 48 hours degraded to a color of only 140 Pt/Co versus an untreated control which degraded to a Pt/Co color of 350.



Rohm and Haas Company's investigation of systems encompassing both epoxy resins and amine curing agents has shown that tremendous color benefits can be derived from treatment of these products with low levels of VenPure products. These color reductions may prove beneficial in extending the uses of epoxy resin systems into unexplored or underdeveloped marketplaces which have previously been reluctant to examine epoxy system components due to poor color stability. This technology can also be employed by epoxy system suppliers committed to "total quality management" or "customers first" programs by improving their ability to meet and exceed customers' increasing expectations of low color, high performance products.

Rohm and Haas Company is a full service supplier with research, analytical and technical services capabilities to meet all of your developmental needs.

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