

FABRICATING TIPS

ASHLAND.

DERAKANE"

FABRICATING TIPS

Table of Contents

Foreword
SECTION 1
Selecting reinforcements, initiators and cure-control additives
Reinforcement types
Glass storage
Initiators
Non-foaming MEKP substitutes
Promoters, accelerators, gel time retarder
Trade names and material suppliers
SECTION 2
Other additives
Surfactants, antifoam agents
Thixotropic additives
Mold release agents
UV protection materials
Wax topcoats
SECTION 3
Food contact and FDA/USDA compliance
Resins able to comply
Fabricating for FDA/USDA compliance
SECTION 4
Formulating guide
Preparing a masterbatch of resin
Conversion factors
Measuring small quantities of chemicals
SECTION 5
Gel time formulations
SECTION 6
Working life of resins
Factors affecting working life
Effect of antimony compounds
SECTION 7
Troubleshooting guide
Gels too slow
Resin does not get hard after gelling
Gels too fast
Resin gel time drifts
Too much exotherm
Resin foams

SECTION 8
Making a laminate and applying FRP linings
Making a test laminate
Guide to making a laminate
Guide to making a conductive lining
Postcuring
Secondary bonding
Applying a test patch
Making a laminate lining
Lining metal
Lining FRP
Lining concrete
Epoxy vinyl ester resin anchors for concrete
Aurasion
SECTION 9
Inspecting a laminate
Checking for complete cure
Recognizing FRP defects
SECTION 10
Other processing techniques
Spray-up applications
Filament winding
Pultrusion
Resin transfer molding
SECTION 11
Industrial health information
Safety Data Sheets 42
Base resins and styrene dilutions
Physical properties
Fire and explosion hazard data
In case of fire
Reactivity data
First aid
Personnel protection
Spills
Environmental and disposal information
Health and hazard data46
SECTION 12
Resin storage
Storage temperatures
Resin shelf life
Aerating the resin
Bulk storage
Storage of promoted resin
Product stewardship



FABRICATING TIPS



This bulletin is intended to provide practical information that will help fabricators choose the most effective combination of DERAKANE and DERAKANE MOMENTUM™ epoxy vinyl ester resins and reinforcement for many end-use applications.

Excellent chemical and corrosion resistance coupled with outstanding heat performance and toughness have made DERAKANE resins the material of choice for a wide range of fiber reinforced plastic (FRP) applications. All conventional fabricating techniques are used with DERAKANE resins to produce FRP structures having an advantageous combination of strength, toughness and corrosion resistance. DERAKANE resins provide finished structures that require little maintenance over a long service life.

Because the FRP industry has such a wide range of needs, our comments and suggestions are general.

IMPORTANT: Fabricators should determine suitability as to any product, additive, process, formula, ratio or end use through their own expertise or tests. No warranty, expressed or implied, is made with respect to the fabricating tips contained in this bulletin.

All this information is based on extensive experience and is offered in good faith, but Ashland cannot accept responsibility for processes or actions not under its direct control. It is the fabricator's responsibility to use safe operating and disposal practices that are in compliance with all applicable laws and regulations for their country.

More information about our family of DERAKANE and DERAKANE MOMENTUM resins is available from your Ashland sales representative or by accessing our web site at www.derakane.com. For more information, contact derakane@ashland.com.

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Note: Potential hazards exist in the handling, storage, and use of DERAKANE resins and the various materials used in preparing resin formulations. Before working with any material, be sure to ask the supplier for appropriate safety and storage recommendations, and observe all precautions and safe handling recommendations. General guidelines are offered in Sections 7, 8, 11 and 12 of this bulletin, and Safety Data Sheets will be provided by your Ashland sales representative.



Selecting Reinforcements, Initiators and Cure-control Additives

DERAKANE epoxy vinyl ester resins have proven to be the FRP industry's best choice for quality and reliability in the fabrication of corrosion-resistant structures and equipment. However, for consistently high-quality end products the other materials used in fabrication must be selected with care, precisely formulated and used by well-trained workers who are familiar with the special techniques of FRP.

This section is a basic guide to the selection of reinforcements, initiators, promoters, accelerators and gel-time retarders.

Note: The names and web sites of national suppliers of product types discussed below are provided at the end of this section. (Page 8)

TYPES OF REINFORCEMENT MOST FREQUENTLY USED IN FRP CONSTRUCTIONS

Seven types of reinforcement are frequently used with DERAKANE resins:

- surfacing veil
- chopped strand
- chopped strand mat
- woven roving
- biaxial mat
- continuous strand
- unidirectional mat

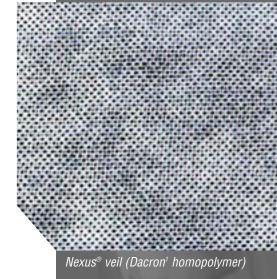
Fabrication of an FRP laminate usually begins with the interior layers (the surface to be in contact with corrosive materials). The first laminate layers are commonly known as the corrosion liner. The corrosion liner's main function is to provide corrosion protection. A surfacing veil saturated with catalyzed resin forms the base for successive layers. The corrosion liner generally consists of the surfacing veil and two layers of chopped strand mat, which are also saturated with catalyzed resin.

The chemical resistance of a fiberglass composite is provided by the resin; surfacing veils are used to help control the thickness of the resin-rich layer and give strength to the most critical part of the corrosion barrier.

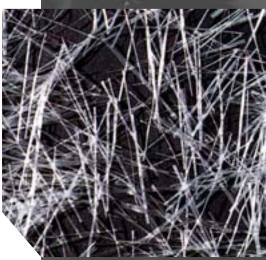
The interior surface, which is normally 10 to 20 mils thick, contains about 90% resin and 10% veil material.

Monofilament glass or polyester fibers are commonly used as surfacing veil materials. Several surfacing veil types are available. The ones most frequently used are made from "C" glass or from Dacron¹ 106 homopolymer. Veil materials made from "A" glass and other polyester fibers are less commonly used, but are gaining acceptance in the industry.

Chopped strand mat reinforcement consists of a felted matrix of chopped strand "E" or "ECR" glass fibers, 0.5" to 2.0" long and loosely held together by a styrene-soluble resin binder. "ECR" mat is made with more corrosion-resistant "E" type glass fibers. When these mats have been wetted with resin, they are easily shaped.







Chopped strand

SECTION



Two mats are generally used with the surfacing veil to form the corrosion liner, providing about 80 mils of additional corrosion protection. Chopped strand mats also are used in the structural layer between layers of woven roving or as the sole reinforcement for the structural wall.

The chopped mat layers in the finished laminate consist of about 70% resin and 30% glass. The short fiber bundles and high resin content help to produce laminates with good strength and excellent chemical resistance.

Saint-Gobain Vetrotex² M113 and OC² 723A and MPM³ mats have given consistently good results with DERAKANE resins.

Other chopped strand mats may work as well, but should be thoroughly evaluated for laminate quality.

Woven roving is a coarse, heavy fabric made of long, continuous glass fiber strands. It is commonly used in alternate layers with chopped strand mats in the structural layer of laminates, and forms the primary structure of many large hand lay-up vessels. In hand lay-up, the resulting laminates are generally about 40-50% glass. In hand lay-up with DERAKANE resins, Hybon³ and Saint-Gobain Vetrotex 324 roving have given the most consistent results. Other rovings may work as well, but should be thoroughly evaluated for laminate quality.

Continuous strand roving comes in various densities and sizing finishes for applications in filament winding and pultrusion. Gun roving is used as a substitute for chopped strand mat in the corrosion barrier and the structural wall. The resulting laminates are generally about 30-40% glass. Good quality roving with epoxy vinyl ester resin compatible sizing are available from Owens Corning, PPG, and Saint-Gobain Vetrotex.



Glass fiber products should be stored carefully so that they will remain clean and will not absorb water. Glass that is dirty or has absorbed water should not be used, because the presence of dirt or moisture could inhibit resin/fiberglass compatibility, resulting in poor laminate quality.

INITIATORS, PROMOTERS, ACCELERATORS, RETARDERS

Initiators are materials which initiate the chemical reaction that causes the resin to cure. Promoters and accelerators are used to speed up and enhance the cure. Retarders are used to extend gel time.

Two types of initiators are used with DERAKANE resins: one which requires cobalt naphthenate and one which does not. Methyl ethyl ketone peroxide (MEKP) and cumene hydroperoxide (CHP) are normally used with promoters, typically cobalt naphthenate (CoNap) and accelerators, typically dimethylaniline (DMA). CHP promoted with CoNap can be used with rapid-cure resins (such as the DERAKANE 470-series) to reduce the exotherm (heat buildup). Benzoyl peroxide (BPO) requires only DMA and is used in the production of FRP where heavy metals must be avoided.

A broad range of working times can be achieved by varying the amounts of initiators, promoters and accelerators within recommended limits. To assure complete resin cure, it is essential to use the recommended amounts of these components. Using too little could result in incomplete resin cure; using too much could result in reduced laminate properties.







Smaller amounts of initiators and accelerators are required for curing DERAKANE resins than for curing polyesters because of the efficient molecular structure of DERAKANE resins.

As already stated, it is essential to use the correct ratios of the activators. Also, thorough mixing is necessary for optimum results, and it is important to mix the activators into the resin in the proper sequence.

NOTE: The promoter should never be mixed directly with a peroxide catalyst (such as MEKP). Mixing would cause a violent reaction, and a fire or explosion could result.

For example, standard procedure is to mix CoNap and DMA thoroughly into the resin before MEKP is added.

INITIATORS

MEKP - Methyl Ethyl Ketone Peroxide

MEKP is sold as a 9% active-oxygen solution of MEKP and a plasticizer. Different brands behave very differently when used with DERAKANE resins. This varying reactivity results from subtle differences in the monomer and dimer content of the various peroxides. The following brands have shown consistent reactivity and have provided rapid resin cure development when used with DERAKANE resins:

- Hi Point⁴ 90
- Luperox⁵ DHD 9
- Norox⁶ MEKP 925 H

Other brands may work as well, but should be thoroughly evaluated before actual use.

MEKP should be stored in a well-sealed container to prevent water contamination. Water in the catalyst will adversely affect resin cure. MEKP can be checked for excessive water content by mixing small amounts with equal parts of styrene. A haze in the mixture indicates excessive water.

For optimum results, it is important to maintain the recommended ratio of MEKP to CoNap in the cure system. A minimum ratio of 3:1 (MEKP: CoNap) is required. A maximum ratio of 10:1 has also proven satisfactory.

Note: A minimum of 0.2% CoNap (6% cobalt) is suggested for all standard DERAKANE resins, except 470. DERAKANE MOMENTUM™ resins may work with lower cobalt levels.

Using proportions outside of a 3:1 to 10:1 range may produce a poorly cured laminate with low Barcol hardness and inadequate corrosion protection. The ratios can be made less critical by use of dimethylaniline or dimethylaceto acetamide as an accelerator.

BPO – Benzoyl Peroxide

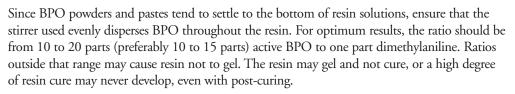
The catalyst BPO is available in powder, emulsion and paste forms for curing all DERAKANE resins. The pastes and emulsions are easier to use than the powders. Cadox⁷ 40E has proven satisfactory. This product contains 40% active BPO. Other brands of BPO at different concentrations are available and will produce satisfactory results after appropriate adjustments in the amount in the formulation.

BPO solutions tend to settle on standing and should be thoroughly mixed immediately before use to ensure uniformity.

For room temperature applications, BPO is accelerated with dimethylaniline. Standard procedure is to mix the dimethylaniline thoroughly into the resin before adding the BPO solution.

⁴ Crompton Corporation ⁵ Elf Atochem America

rporation ⁶ Norac Company, Inc. America ⁷ Akzo Nobel Chemicals B.V.



Diethylaniline is a less active accelerator than dimethylaniline and can be used when longer gel times are required. Optimum ratios are from 4 to 12 parts active BPO to one part diethylaniline.

CHP - Cumene Hydroperoxide

The use of CHP with DERAKANE 470-type resins results in lower exotherms, less shrinkage and less warpage. In cool weather, in fact, a small amount of dimethylaniline may be used to accelerate cure. CHP is 80-90% active peroxide and 20% cumene, and material of this ratio has been used in formulation work.

Mixing procedures are similar to those used with systems of MEKP and CoNap.

The following brands of CHP have shown consistent reactivity when used to catalyze DERAKANE 470 resin: Luperox CU90, Norox CHP, Superox⁸ 727, and Trigonox⁹ K-90. Other brands may work as well, but should be thoroughly evaluated before use.

NON-FOAMING MEKP SUBSTITUTES

Trigonox 239A (239A), CHP-5, and Norox CHM-50 are available for use with DERAKANE epoxy vinyl ester resins. These systems can eliminate the foaming experienced with epoxy vinyl ester resins catalyzed with MEKP/cobalt naphthenate catalyzed systems. Another advantage of these systems is that when used to catalyze DERAKANE 411-type resins, peak exotherms are lowered 40° to 50°F (22° to 27°C), based on 50-g mass, as compared to equivalent MEKP/cobalt naphthenate catalyst systems.

NOTE: The peak exotherms for DERAKANE 470 and other resins are lowered by only about 20°F (11°C).

Trigonox 239A, CHP-5 and Norox CHM-50 provide as complete and as rapid a cure as MEKP with DERAKANE resins at ambient temperatures and function with normal accelerator/ promoter systems. The typical gel time formulations for 239A, CHP-5 and CHM-50 are similar to those shown for MEKP with the different DERAKANE resins and should be used as guidelines for formulation development. When 239A, CHP-5 or CHM-50 are used, the addition of 0.1 wt% CoNap (6% cobalt) above the CoNap levels recommended in the formulation tables should provide gel times comparable to those for MEKP.

PROMOTER

CoNap - Cobalt Naphthenate

CoNap promoter is a dark, purplish liquid sold in solutions of 6%, 12% and 21% active cobalt in a solvent.

The 6% solution has been used in formulation work with DERAKANE epoxy vinyl ester resins. Different brands have shown little difference in reactivity with DERAKANE resins.

Akzo Nobel Chemicals B.V.



ACCELERATORS

N,N-Dimethylaniline (DMA),

N,N-Diethylaniline (DEA), and

N,N-Dimethylacetoacetamide (DMAA)

N,N-Dimethylaniline and N,N-Diethylaniline are amines used in small quantities to accelerate MEKP, BPO, 239A, CHP and CHP-5 type cures. N,N-Dimethylacetoacetamide also can be used to accelerate MEKP, CHP, 239A and CHP-5 cures.

NOTE: DMAA does not work with BPO. Resins formulated with DEA or DMAA have less gel time drift than with DMA.

Formulation work has been done with 100% active DMA and DEA, and 80% active DMAA.

GEL TIME RETARDER

2,4-Pentanedione (Acetylacetone)

The retarder 2,4-Pentanedione (2,4-P) can be incorporated into systems of MEKP, CHP, 239A, or CHP-5 and CoNap at a level of 0.05 to 0.30 to retard the gelling of DERAKANE resins. The gel

Table 1.1 - Delayed gel times for DERAKANE 411-45 resin

MEKP WT%	CoNap (6%) WT%	DMA WT%	2,4-P WT%	TYPICAL GEL TIME @77°F (25°C) (MINUTES)	TYPICAL PEAK TIME (MINUTES)
1.00	0.25	0.00	0.00	21	37
1.00	0.25	0.00	0.05	23	39
1.00	0.25	0.00	0.10	60	74
1.00	0.25	0.00	0.20	180	191
1.00	0.25	0.00	0.30	265	280
1.00	0.20	0.10	0.00	15	20
1.00	0.20	0.10	0.05	28	43
1.00	0.20	0.10	0.10	72	103
1.00	0.20	0.10	0.20	171	233
1.00	0.20	0.10	0.30	225	317
2.00	0.20	0.10	0.00	13	17
2.00	0.20	0.10	0.10	65	76
1.00	0.30	0.10	0.00	13	17
1.00	0.30	0.10	0.10	29	38
1.00	0.20	0.20	0.00	10	12
1.00	0.20	0.20	0.10	29	69

^{*} Typical results; not to be construed as warranties or specifications.



SECTION 1

time can be extended as much as tenfold without adverse effects on the final cure or the corrosion resistance of the laminate. This material can be formulated into high-build coatings to give an extended pot life or can be used for fabrication when very long gel times are required.

Table 1.1 and Table 1.2 show how gel time can be extended by adding 2,4-P. Similar effects are observed with all DERAKANE resins. Table 1.1 shows that the amount of time is very dependent on temperature and the levels of peroxide/promoter used.

Caution: Gel time will not be delayed by addition of 2,4-P when a vinyl ester cure is catalyzed with BPO using DMA as an accelerator. Also, in many polyester systems, 2,4-Pentanedione may act as an accelerator. Tertiary butyl catechol (TBC) in the range of 0.01 to 0.03% can be used to retard the gel time of BPO/DMA systems.

Safety Note

Ashland Composite Polymers does not manufacture or market the peroxides, promoters, accelerators and retarders used with DERAKANE resins. Technical data and safe handling and storage information should be obtained from the manufacturers of MEKP, BPO, CHP, 239A, CHP-5, CoNap, DMA, DEA, DMAA and 2,4-P.

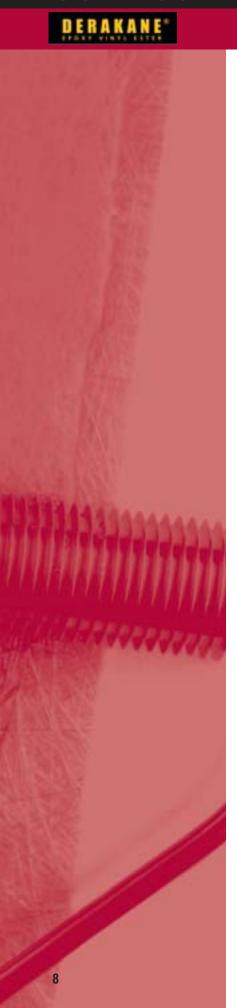
The peroxide MEKP is especially dangerous to the eyes. Workers should wear goggles at all times when handling, measuring and mixing the catalyst. MEKP may cause skin burns. Proper protective clothing should be used to minimize contact. To avoid a violent reaction, promoters should never be mixed directly with a peroxide such as MEKP.

The accelerator DMA is a contact poison. To prevent absorption through the skin, workers should wear protective clothing as well as eye protection when handling, measuring or mixing this material.

Table 1.2 – Delayed gel times for DERAKANE 470-36 resin

CHP WT%	CoNap (6%) WT%	DMA WT%	2,4-P WT%	TYPICAL GEL TIME @77°F (25°C) (MINUTES)	TYPICAL PEAK TIME (MINUTES)
1.50	0.20	0.10	0.00	29	40
1.50	0.20	0.10	0.05	43	59
1.50	0.20	0.10	0.10	63	90
1.50	0.20	0.10	0.15	100	131
2.00	0.20	0.10	0.00	30	44
	0.20	0.10	0.10	56	102
1.50	0.20	0.20	0.00	32	42
1.50	0.20	0.20	0.10	56	76
1.50	0.30	0.10	0.00	24	34
1.50	0.30	0.10	0.10	48	67

Typical results; not to be construed as warranties or specifications.





Trade Names and Material Suppliers

Trade name	Туре	Manufacturer	Web site
SURFACING VEILS Nexus® Avelle® APC M524-ECR25A Freudenberg T-1777	Polyester Polyester "C" Glass "C" Glass	PFG XAMAX Industries, Inc. Owens Corning Freudenberg	precisionfabric.com xamax.com owenscorning.com nonwovens.group.com
MATS M113 Advantex® M723A MPM-5	"E" Glass "ECR" Glass "E" Glass	Saint-Gobain Vetrotex Owens Corning PPG Industries	sgva.com/index.html owenscorning.com ppg.com
WOVEN ROVING 318, 322, 324, 326 HYBON® Woven Roving Knytex® Woven Roving		Saint-Gobain Vetrotex PPG Industries Owens Corning	sgva.com/index.html ppg.com owenscorning.com
CONTINUOUS ROVING HYBON® 2000 Series Advantex® Type 30 R099® 625 & 673		PPG Industries Owens Corning Saint-Gobain Vetrotex	ppg.com owenscorning.com sgva.com/index.html
GUN ROVING HYBON® 6700 OC® Multi-End Rovings 255, 292, 298, & 299		PPG Industries Owens Corning Saint-Gobain Vetrotex	ppg.com owenscorning.com sgva.com/index.html
INITIATORS HiPoint® 90 Luperox® DHD 9 Norox® MEKP 925H Cadox® L-50a Cadox® 40E Cadox® 40ES Cadox® BTW 55 Luperox® AFR 400 Benox® L-40LV CHP Luperox® CU90 Norox® CHP Trigonox® K-90	MEKP MEKP MEKP MEKP BPO BPO BPO BPO CHP CHP	Crompton Corporation ATOFINA NORAC, Inc. Akzo Nobel Akzo Nobel Akzo Nobel Akzo Nobel AKZO Nobel ATOFINA NORAC, Inc. Crompton Corporation ATOFINA NORAC, Inc. AKZO Nobel	cromptoncorp.com atofina.com norac.com akzonobel.com akzonobel.com akzonobel.com akzonobel.com atofina.com norac.com cromptoncorp.com atofina.com norac.com
Initiators — Non-Foaming MEKP Substitutes Trigonox® 239A CHP-5 Norox® CHM-50		Akzo Nobel Crompton Corporation NORAC, Inc.	akzonobel.com cromptoncorp.com norac.com
PROMOTERS 6% Cobalt Naphthenate 6% Cobalt Octoate		Servo Coating Additives OM Group Servo Coating Additives OM Group	servousa.com omgi.com servousa.com omgi.com
ACCELERATORS N, N-Dimethylaniline N, N-Diethylaniline N, N-Dimethylacetoacetamide	DMA DEA DMAA	Eastman Chemicals Sigma-Aldrich Sigma-Aldrich Sigma-Aldrich	eastman.com sigmaaldrich.com sigmaaldrich.com sigmaaldrich.com
GEL TIME RETARDER 2, 4-Pentanedione	2,4-P	Ashland Sigma-Aldrich	ashland.com sigmaaldrich.com

Other Additives

SURFACTANTS AND ANTIFOAM AGENTS

Air bubbles in a laminate reduce its strength and may seriously affect its corrosion resistance properties. Air bubbles in the corrosion liner¹ can be more damaging than those in the structural layer. The following practices should always be followed to minimize air bubbles:

- 1. Avoid violent mixing which mixes air into resin. However, be sure the catalyst is mixed thoroughly into the resin.
- 2. Apply resin to the mandrels first, then apply glass, and roll it into the resin. Air bubble problems are inevitable when resin is applied to "dry" glass.
- 3. Roll the laminate from the center to the edges. Roll firmly, but not too hard. Excessive pressure might fracture existing bubbles and make them more difficult to remove.
- 4. Eliminate all the bubbles from one ply before starting on the next.
- 5. Thoroughly clean rollers between uses.

The addition of certain surfactants and antifoam agents can improve the glass wet-out and air bubble release of DERAKANE resins. One of the most effective surfactants evaluated is BYK² A515. When added to a DERAKANE resin at a level of 0.15%, this product significantly reduces the resin's surface tension, thus improving glass wet-out and air bubble roll-out.

BYK A555 antifoam agent at a level of 0.15% causes entrapped air bubbles to readily burst by themselves. The antifoam agent and surfactant at 0.15% do not reduce the corrosion resistance of properly cured laminates.

Another widely used antifoam agent is SAG 47³ silicone-based antifoam compound. The suggested maximum amount of SAG 47 for DERAKANE resins is 0.05 wt%.

Foamkill⁴ 8R and 8G, used in the 0.05 to 0.06 wt.% range, are other effective air release agents.

Note: Foamkill 8G meets FDA requirements for use in FRP applications with a use level of 0.05 to 0.06 wt.%. BYK A515, BYK A555, SAG 47 and Foamkill 8R are not on the list of additives complying for FDA requirements.

The compatibility between DERAKANE resins and glass fibers can be improved by adding 2, 4-Pentanedione to the resin at levels up to 0.05%. At such low levels, 2,4-P is unlikely to significantly affect the gel time of resin systems with a minimum of 0.3% CoNap (6% cobalt).

THIXOTROPIC ADDITIVES

In fabricating certain structures with DERAKANE resins, it may be desirable to control resin flow or runoff by adding a thixotropic material – usually a thickener-filler. The most efficient and effective of these additives for use with DERAKANE resins are Cab-O-Sil⁵-TS-720 and Aerosil⁶ 202. (See Table 2.1.)

¹ Corrosion liner–Veil-Mat-Mat = 100 mils

² BYK-Chemie USA

³ Sigma-Aldrich

⁴ Crucible Chemical

Cabot Corporation
 Degussa Corporation

This bulletin contains tips and suggestions, but no warranty, expressed or implied, is given; fabricators are advised to determine suitability as to any product, additive, process, formula, ratio or end-use through their own expertise or tests.

In structural applications, the level of thixotropic additives should be as low as possible to maximize the strength of the laminate. Also, because thixotropic additives reduce corrosion resistance to some chemicals, they should never be used in corrosion-resistant liners. When thixotropic materials are used, uniform wetting is necessary for optimum results, and a high speed, high shear mixer, such as a Cowles Dissolver⁷ or its equivalent, should be used for blending.

FRP fabricators should contact additive manufacturers for recommendations on safe handling and storage of thixotropic additives.

Table 2.1 – Effect of Cabot Cab-O-Sil TS-720 thixotropic additive

(Comparable results are obtained with Aerosil 202 from Degussa.)

Product	Percent Additive	VISCOSITY, mPa.s (cP) 3 RPM	Thixotropic 30 RPM	Index
DERAKANE 411-45	0	560	660	0.85
	1	2400	1400	1.71
	2	5600	2320	2.41
	3	11,200	3760	2.98
DERAKANE MOMENTUM™ 411-350	0	350	380	0.92
	1	2000	1400	1.82
	2	12,000	3500	3.43
	3	19,000	4500	4.22
DERAKANE 441-400	0	400	400	0.91
	1	2700	1000	2.71
	2	6000	1700	3.53
	3	18,500	4400	4.21
DERAKANE 470-36	0	160	200	0.80
	1	2000	880	2.27
	2	5500	1450	3.79
	3	16,000	3400	4.71
DERAKANE MOMENTUM 470-300	0	300	320	0.94
	1	2500	1100	2.27
	2	5800	1650	3.51
	3	18,000	3990	4.51
DERAKANE 510A-40	0	280	300	0.93
	1	4000	1550	2.58
	2	11,500	3500	3.29
	3	32,500	7700	4.22
DERAKANE MOMENTUM 510C-350	0	320	350	0.91
	1	4150	1750	2.37
	2	10,000	3050	3.28
	3	24,500	5500	4.46
DERAKANE 8084	0	400	450	0.89
	1	1820	1040	1.75
	2	5410	1970	2.75
	3	13,200	3610	3.66

 $^{^{\}star}$ Typical results; not to be construed as warranties or specifications.



⁷ Cowles Division, Morehouse Industries, Inc.

OMPOSITE POLYM

SECTION



Wax, or films of PVA, Mylar¹, or cellophane may be used as mold release agents to facilitate the removal of fabricated parts from metal, wooden, cardboard or plastic molds and mandrels. It is important to check the release characteristics of any mold release agent before using it in production.

Wax

Hard-based paraffin waxes and carnauba waxes are used successfully as mold release agents in part fabrication with DERAKANE resins. Several brands provide excellent results, including:

- Meguiars² Mirror Glaze
- Trewax³
- Johnson's Traffic Wax
- TR5 Mold Release

These products are generally available from local fiberglass supply distributors. Waxes based on acrylics have not been accepted for use as mold release agents because they tend to inhibit cure. The use of some silicone-based mold release agents has been shown to inhibit laminate cures of DERAKANE resin.

Optimum results are obtained by applying the wax to the mold or mandrel with a clean cloth, then buffing the surface vigorously to a hard, glossy, ultrathin film. Several coats of wax should be applied before a mold is used, then a single coating of wax after each release.

Excess wax left on the mold surface may inhibit resin cure, resulting in a hazy corrosion liner. Even though the surface beneath the wax may be fully cured, the hazy surface may give a low Barcol reading. (See Section 9, "Inspecting a Laminate.") If this occurs, remove approximately 2 to 3 mils of the wax coating by sanding, then check the surface again using the Barcol hardness check described on page 32. If a high-gloss surface is desired, overcoat the buffed wax surface of the mold with a PVA film.

Films of PVA (Polyvinyl Alcohol)

Films of polyvinyl alcohol, when properly applied, can provide excellent mold release for parts made with DERAKANE resins. PVA films may be sprayed or painted over waxed or polished metal molds. After application, the film must be dried thoroughly, because any residual moisture could inhibit the cure of DERAKANE resins cured with MEKP or CHP catalyst systems. Because PVA films are hydrophilic, it may be necessary (in high humidity atmospheres) to force-dry them with heat lamps.

Films of Mylar and Cellophane

Tapes of Mylar and cellophane 1-6" wide and 1-2 mils thick have been used successfully over mandrels as a mold release. Films of Mylar 5-10 mils thick have excellent release characteristics and are used on benchtops for the production of parts and flat sheets.

5 T. R. Industries



E. I. DuPont de Nemours & Co., Inc.

² Mirror Bright Polish Company ³ Trewax Company

S. C. Johnson and Sons, Inc.

ULTRAVIOLET PROTECTION

FRP parts fabricated with either polyester or vinyl ester resins may display surface chalking and discoloration after long exposure to ultraviolet radiation. These effects may be reduced or eliminated by:

- incorporating UV absorbers into the resin
- pigmenting outer resin layers
- painting the surface with epoxy-polyamide paints
- incorporating fillers

UV absorbers do not always provide effective protection for long-term service and they also inhibit resin cure. Two that can be used without inhibiting the resin are:

- Cyasorb⁶ UV-24
- Tinuvin⁷ 326

These products are incorporated into the external coat at a level of 0.1-0.3%.

Manufacturers who wish to formulate a pigmented gel coat should contact a pigment manufacturer.

REMINDER: Many inspectors require that the equipment be inspected visually before pigmented topcoats are applied. Don't paint or pigment unless the specifier has requested it.

If equipment is finished several days or weeks before the inspection, grinding the exterior before top-coating can be avoided by using a polyamide epoxy coating. The only surface preparation required is the removal of dirt, dust, oil, grease and wax with a degreaser-type solvent and clean rags.

The polyamide epoxy paint is mixed and applied according to the manufacturer's instructions, making sure that the air and surface temperatures are above 50°F (10°C) throughout application and cure. The coating should not be applied when the weather is humid or when rain is expected.

The polyamide epoxy paints listed in Table 2.2 have been used successfully on equipment fabricated with DERAKANE resins.

For non-pigmented equipment, a surfacing veil made with polyester fibers has been shown to prevent fiber bloom. Nexus⁸ veil, type 100-10, has provided consistent results.

AIR INHIBITION AND WAX TOPCOATS

Oxygen in the air may inhibit the complete cure of an exposed FRP surface. If surface inhibition occurs, it may result in reduced chemical resistance, and/or premature failure. Several techniques are used to overcome this problem, all of which involve preventing or reducing air contact at the curing surface.

A wax/resin topcoat acts as a film to protect the surface from air contact. A layer (2-3 mils) of resin containing a small amount (0.4%) of paraffin wax with a melting point of 120°F (49°C) is applied. During polymerization, the wax rises to the surface and forms a thin wax film.

Wax topcoats should be used as the final coating of all secondary lay-ups and on all repairs of areas exposed to a corrosive environment.

Caution: Wax topcoats should not be used between laminate layers because they may interfere with adhesion of one layer to the next.



American Cyanamid

Geigy Chemical

⁸ Precision Fabrics

SECTION 2

The small amount of paraffin is readily incorporated into the system by preparing a masterbatch of 10% wax in hot (140°F [60°C]) styrene. Refer to Table 2.3. Wax-styrene mixtures also can be purchased from FRP distributors. Add sufficient wax-styrene mixture to the resin to provide a wax-topcoat system containing about 0.4% paraffin (between 3-5% of wax-styrene mixture). The resin should then be catalyzed to give a 5- to 20-minute working life, as needed. A fast gelling system is suggested.

Since DERAKANE 470 type epoxy vinyl ester resins produce such hot exotherms when curing, a wax topcoat may not be necessary to prevent air inhibition of resin cure. To determine if the laminate is sufficiently cured at the surface, an "acetone wipe test" should be performed. If the cured resin surface passes the "acetone wipe test," a wax topcoat is not necessary.

Note: The uses cited above are subject to good manufacturing practices and any limitations that may be imposed by governmental regulations. All applicable regulations should be consulted for complete details.

Table 2.2 - Polyamide epoxy paints typically used in topcoating

Manufacturer	Designation	Color	Wet Film Thickness
Ameron	Amerlock® 400	Light Gray	8 mils (200 microns)
Carboline	Carboguard® 890EF	Light Gray	9 mils (225 microns)
PPG	95-245 Rapid Recoat Pitt-Guard	Light Gray	9 mils (225 microns)
DuPont	DuPont® 25P	Light Gray	9 mils (225 microns)

Products listed above are registered trademarks of corresponding companies.

Table 2.3 – Typical wax topcoat*

Formulation	5 Gallons	1 Gallon			
Part A					
Melted Paraffin**	80 g (88 cc)	16 g (18 cc)			
Styrene	720 g (800 cc)	144 g (160 cc)			
Styrene heated to 140°F (60°C) is added to melted paraffin with a temperature of at least 140°F (60°C) in a clean container with vigorous agitation to form a suspension. Before working with this material, operators should be sure to understand the possible hazards associated with handling styrene, and to take appropriate precautions before working with this material.					
Part B DERAKANE Resin	5 Gallons	1 Gallon			
Add Part A to Part B with vigorous agitation and catalyze with appropriate catalyst, promoter and accelerator for 5- to 20-minute pot life.					

* Starting point formulation

^{**} Paraffin (melting point approximately 120°F [49°C]), Exxon or equivalent

Food contact and FDA/USDA compliance

FOOD CONTACT

DERAKANE 411, 411C and 441-400 epoxy vinyl ester resins, when properly formulated and cured, will comply with the U.S. Food, Drug and Cosmetic Act, as amended, and applicable FDA regulations (21 CFR 177.2420). These resins may be used as articles or components of articles intended for repeated use in contact with food, subject to certain limitations described in that regulation.

NOTE: It is the responsibility of the customer/user to test the finished laminate or coating to determine compliance with 21 CFR 177.2420 and all other appropriate regulations.

DERAKANE 411, 411C and 441-400 epoxy vinyl ester resins are chemically acceptable in processing or storage areas for contact with meat or poultry food products prepared under federal inspection and used at temperatures below 250°F (121°C). This acceptance has been given by the United States Department of Agriculture. Catalyst levels should not exceed levels set out in the FDA regulations in 21 CFR 177.2420.

Note: DERAKANE 411 resin is available with different styrene levels. Achievement of appropriate complete cure (using recommended catalyst levels, etc.), together with careful attention to the special five-step, post-fabrication techniques listed above, will enhance the fabricators' ability to comply with regulations.

FABRICATING FOR FDA/USDA COMPLIANCE

Attention to the following procedure can help you achieve FDA compliance:

- 1. Use a catalyst formulation that will yield a resin laminate with low residual styrene after a room temperature cure. FDA regulation 21CFR 177.2420 lists the following approved initiators, promoters and accelerators: MEKP, CHP, BPO, CoNap, DMA and DEA.
- 2. Thoroughly clean the part to remove any dust or dirt prior to postcuring.
- 3. Postcure with dry heat for 2 hours at 200°F (93°C) or 4 hours at 180°F (82°C). The purpose is to reduce residual styrene to the range of 0.01-0.2%.
- 4. After postcuring, steam-treat the part or steep it in hot water for 8-16 hours at 160°F (71°C) or higher. It is recommended that only hot water be used in FRP-lined metal vessels. This should remove all residual styrene from the laminate surface.
- 5. Wash the part thoroughly with detergent and rinse it thoroughly before placing it in service.



SECTION 4

Formulating Guide

PREPARING A MASTERBATCH OF RESIN

A successful technique for obtaining consistent curing characteristics is to prepare a masterbatch containing the resin CoNap, and dimethylaniline (if required) in a quantity appropriate for your operation. Masterbatches of 5 to 55 gallons are common. The masterbatch contains promoters and accelerators. It does not contain initiators. A properly prepared masterbatch will have a shelf life of several weeks.

Masterbatches should be carefully prepared by a designated, reliable technician and should be checked frequently for curing characteristics and uniformity. The masterbatch approach assures that the proper ratio of promoters and accelerators will be used each time. Masterbatches also give operators latitude in determining the amount of catalyst needed and the time required for a specific job. It helps them compensate for changes in temperature and other ambient conditions.

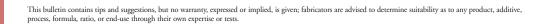
A masterbatch is prepared by thoroughly mixing CoNap and the dimethylaniline into the DERAKANE resin. High speed air-driven mixers, such as Lightnin¹, give satisfactory results. Bunghole mixers with small propellers may not give adequate mixing.

Use Table 4.1 to determine how much promoter and accelerator should be added to prepare a masterbatch.

Table 4.1 - Conversion factors²

WT%	CC/LB	CC/GAL	FLUID OZ/ 5 Gal	FLUID OZ/DRUM (205 KG/450 LB)	CC/DRUM (450 LB)
.10	0.4	3.6	0.6	6.3	186
.20	0.8	7.2	1.2	12.6	371
.25	1.0	9.0	1.5	15.7	464
.30	1.2	10.7	1.8	18.8	557
.40	1.7	14.3	2.4	25.1	743
.50	2.1	17.9	3.0	31.4	929
.60	2.5	21.5	3.6	37.7	1114
1.00	4.1	35.8	6.1	62.8	1857
1.25	5.2	44.7	7.6	78.5	2322
1.50	6.2	53.7	9.1	94.1	2766
2.00	8.3	71.6	12.1	125.6	3715

⁽Chart based on Resin Density = 1.04; Additive Density = 1.10)



¹ Mixing Equipment Company

² Fabricators should confirm by their own tests.

SAFETY NOTE

Proper safety precautions must be used in all these operations. Suitable eye protection and protective clothing should always be used when handling resins, promoters, accelerators and initiators. Good housekeeping practices should be observed to minimize contamination of the working area. Proper ventilation should be maintained to meet government standards. Disposable containers should be used whenever possible for mixing. Used containers and resin waste should be disposed of properly in accordance with governmental regulations. See Section 12, "Resin Storage."

For each batch, the operator may vary the amount of MEKP (within prescribed limits) to get the gel time desired. (A typical gel time is 25 to 30 minutes.) For small jobs, a typical batch size is about one quart of resin. The correct amount of MEKP should be measured carefully and mixed thoroughly into the resin. A minimum mixing time is 30 seconds, including time for scraping the sides and bottom of the mixing container. The operator should take care to minimize air entrainment during mixing.

MEASURING SMALL QUANTITIES OF CHEMICALS

The quantities of initiators, promoters and accelerators to be used in curing DERAKANE resins should be accurately measured. Measurements are made with such devices as throw-away syringes, graduated cylinders or plastic squeeze bottles with graduated pour spouts. Actual gel times may vary as a result of subtle variations in curing agents and resins; slight variations in measuring small amounts of reactants; the size of the resin batch; and environmental parameters, such as temperature, humidity, thickness of the laminate and mold type.



Gel time formulations

GEL TIMES AND TEMPERATURES

DERAKANE epoxy vinyl ester resins are polymerized by the use of free-radical initiating initiators. Since many variables affect the cure of the resins, the catalyst system must be adjusted for each individual application.

The selection criteria for the catalyst system are:

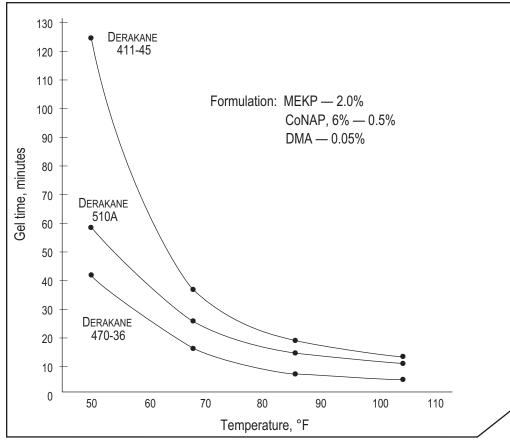
- gel time desired
- working temperature
- effect of the mold as a heat sink
- effect of post cure

Figure 5.1 shows the effect of different temperatures on the typical gel times of DERAKANE epoxy vinyl ester resins.

TYPICAL GEL TIME FORMULATIONS FOR DERAKANE EPOXY VINYL ESTER RESINS

Reactivity and material properties for the family of DERAKANE and DERAKANE MOMENTUM™ resins can be found at www.derakane.com.

Figure 5.1
Typical gel times vs. temperature for DERAKANE resins*



^{*} Typical results; not to be construed as warranties or specifications.

This bulletin contains tips and suggestions, but no warranty, expressed or implied, is given; fabricators are advised to determine suitability as to any product, additive, process, formula, ratio or end-use through their own expertise or tests.

Working life of resins

FACTORS AFFECTING THE WORKING LIFE OF A RESIN

For successful bonding to glass fibers, a resin should be used as soon as possible after mixing with the catalyst and other additives. The manufacturer's suggested pot life refers to the amount of time after the resin is properly and thoroughly mixed with initiators, promoters and accelerators before it sets up in the container. The pot life times stated by resin manufacturers are determined by laboratory testing with small batches of about 100 grams under controlled temperature and humidity conditions, and conditions in the field or commercial shop rarely match laboratory conditions. Changes in ambient temperature and humidity, for example, can affect pot life. However, the pot life of a resin can be adjusted by varying the amounts of other materials in the mix to make allowance for actual shop conditions. Such adjustments help determine the working time – the period after catalysis and before gelation when a resin can be applied successfully to glass fibers. In practice, the working time of a resin is very significant to the fabricator. Shop or field conditions and practices can extend or decrease working time.

Conditions that extend working time:

- cooler temperatures
- cool resin
- large heat sinks (metal molds)
- high humidity
- brisk winds
- · addition of fillers

Conditions that decrease working time:

- high temperatures
- warm resin
- insulated heat sinks (wooden molds)
- thick laminates
- direct sunlight

EFFECT OF ANTIMONY COMPOUNDS ON GEL TIMES OF FIRE RETARDANT RESINS

Antimony compounds can be added directly to the resin and dispersed using normal mixing equipment. Resin mixed with antimony should be stirred frequently to keep the materials well mixed. To minimize the effect of Nyacol¹ APE-15401 on the gel time of DERAKANE resins, add the antimony compound immediately prior to use. If left overnight, resin with a cobalt promoter and APE-1540 added may experience significant gel time drift, and the resin may not cure properly. APE-3040 has been shown to have less impact on gel time and gel time drift in resin formulations. Antimony trioxide added to resins containing a cobalt promoter may affect resin gel times as well, but any noticeable effect will occur at a much slower rate than with APE-1540.



¹ Nyacol Nano Technologies, Inc.

DERAKANE"

SECTION 7

Troubleshooting guide

Table 7.1 – Problem Solving for Ambient Temperature Cure Resins

Problem	Po	eroxide
Fioniciii	MEKP or CHP	вро
Resin gels too slowly or will not gel	Review concentrations. No less than 1.0% MEKP or CHP required. No less than 0.15% CoNap (6%) required unless ambient temperature is over 85°F (29°C). Review other additives. Some pigments and flame retardant additives retard gelling and need to be added just before use. Review addition sequence. CoNap is difficult to mix and requires good agitation. Resin is purple to rose color after CoNap is added and turns dark when peroxide is added. DMA does not affect color. 2,4-P almost eliminates color change.	Review additives. DMA required (DMAA not a substitute). BPO required. BPO is not 100% active. Adjust formulation to give required amount of active peroxide. Check temperatures of resin, shop and mold. Adjust formulation to temperature. BPO requires good agitation. Review other additives. Some pigments and flame-retardants may slow gelling. Add them just before use, or increase catalyst/promoter concentration.
Resin does not get hard after gelling	Review levels of peroxide and CoNap. If mold or resin is cold, DMA is required. To achieve similar gel time with DMA, reduce peroxide level or add 2,4-P. If weather is hot, formulators often reduce peroxide levels too far to get a good cure. Use 1.0% minimum. Check to see if problem is at the surface. Use a wax topcoat. Check fittings. Bronze, zinc and copper inhibit cure.	Ratio of active BPO to DMA or DEA is critical. Preferably, the BPO level must be between 10 to 20 times the DMA level (preferably 10 to 15 times) or between 4 to 12 times the DEA level. The absolute minimum peroxide level is 0.75% but should be 1% if at all possible. Check to see if problem is only at the surface. If so, a wax topcoat is recommended. Review other additives. Some pigments and flame-retardants may slow gelling. Add them just before use or increase catalyst/promoter concentration.
Resin gels too fast	Review promotion schedule. Check resin temperature. Reduce or eliminate DMA. Add 2,4-P. Reduce level of CoNap to 0.1% minimum.	Reduce level of BPO and/or DMA. Remember to watch BPO/DMA ratio.

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Problem Solving for Ambient Temperature Cure Resins

Problem	Pe	eroxide
i ioniciii	MEKP or CHP	вро
Resin gel time drifts after sitting overnight	If the resin container is left open, the resin can absorb high amounts of water even in an air conditioned building. High water levels will greatly extend gel times and may prevent the resin from curing properly. This scenario is especially prevalent in the hot, humid summer months. Increasing the amount of promoter, accelerator and catalyst may help overcome the effects of water. If Nyacol APE-1540 was added to a fire-retardant resin, the Nyacol material has absorbed the CoNap to such an extent that there is not enough free CoNap to promote the gelling reaction. Add additional CoNap and, if needed, accelerator to restore the resin to its initial gel time.	Add additional DMA and possibly BPO. Remember to keep the final ratio of BPO to DMA correct. See cause under MEKP or CHP of this section.
Resin generates too much exotherm in curing	Reduce or eliminate DMA. Adjust peroxide to 1.0% minimum. Make thinner lay-ups and allow to exotherm before continuing. Use CHP with DERAKANE 470 resin. Use 239A and CHP-5 with DERAKANE 411s resin.	BPO/DMA is a hot system. Apply fewer layers and allow them to exotherm before continuing. Add up to 0.03°L TBC.
Resin foams, resulting in entrained air in the laminate	Use 239A and CHP-5 for all DERAKANE resins. For greater control with DERAKANE 470 resin, use CHP.	

Making a laminate and applying FRP linings

MAKING A TEST LAMINATE

This short photographic essay illustrates the major steps often used in FRP laminate manufacture. A more detailed step-by-step guide for making an FRP laminate is given on pages 24-26.

Three important factors could not be shown in a photo essay: 1) precise, accurate formulation of materials; 2) the variables involved in making a laminate to satisfy particular specification requirements; and 3) the expertise, skill and artistry of the worker making the laminate. Detailed information is provided elsewhere in this bulletin to help fabricators manufacture quality FRP laminates with DERAKANE resins. We believe these instructions can help FRP workers formulate materials properly and control the variables involved in meeting specification requirements. Only the workers' experience and attention to detail, however, can provide the craftsmanship.

Put yourself, your experience, and your hands into these photos; then remember what's shown is only part of the complete picture.

Step 1: Cover bench with release sheet



After you have formulated a batch of resin and organized the work area and tools, lay a Mylar¹ polyester release sheet over the mold surface.

Step 3: Apply surfacing veil



Position the glass surfacing veil carefully over the wet resin, and roll it into the resin with a serrated roller.

Step 2: Spread resin base



Pour catalyzed DERAKANE resin onto the release sheet, and spread it evenly with a roller.

Step 4: Add more resin



Apply additional catalyzed resin, and roll again.

This bulletin contains tips and suggestions, but no warranty, expressed or implied, is given; fabricators are advised to determine suitability as to any product, additive, process, formula, ratio or end-use through their own expertise or tests.

Step 5: Apply layer of chopped strand mat



Now place and roll the first layer of chopped strand mat.

Step 6: Add resin and apply second mat



Spread catalyzed resin over the first chopped strand mat; then apply a second chopped strand mat, and roll it into the resin.

Step 7: Apply woven roving



Spread a little more resin over the second chopped strand mat, and apply a layer of woven roving.

Step 8: Roll roving into resin



Roll the woven roving carefully into the resin with a serrated roller.

Step 9: Add final chopped strand mat



Now apply a final chopped strand mat, add catalyzed resin, and roll again.

Step 10: Finish with wax topcoat



Finally, coat the surface with a protective wax topcoat. Allow the resin to gel and cure. The laminate is now ready for use. (For a more detailed explanation, refer to the fabrication guide that follows.)



SECTION

GUIDE TO MAKING A LAMINATE

The following outline is a guide to the use of DERAKANE epoxy vinyl ester resins in producing typical FRP laminates by hand lay-up.

A. Safety considerations

- 1. Be knowledgeable about safety, handling precautions and procedures, and proper disposal of resins, glass, initiators, promoters and accelerators.
- 2. Wear clean, body-covering, proper protective clothing, gloves and chemical workers' goggles.
- 3. Care must be taken to avoid unnecessary contact with the resin, initiators and other chemicals which are used in conjunction with normal use of DERAKANE resins.
- 4. Good, general ventilation should be satisfactory for most conditions. However, local exhaust may be required where there is little air circulation, such as in confined areas. Respiratory equipment—either air purifying or self-contained breathing apparatus—may be required when the possibility of exposure to levels over the peak concentration may be encountered.
- 5. Keep yourself, and the work area, as clean as possible.
- 6. During lay-up, keep laminates in an area away from open flames or sparks.
- 7. Wear a respirator when sanding.
- 8. Be aware that thick laminates may undergo an exothermic reaction (get hot and emit noxious odors).
- 9. Dispose of wastes properly.

B. Plan what you need for the job

- 1. Assemble proper tools—mixers, brushes, rollers, solvents and cleaning tools.
- 2. Assemble proper fabricating materials—resin, glass, catalyst, etc.
- 3. Be sure proper safety equipment is available and ready for use—safety glasses, clean gloves, fire extinguishers, etc.

C. Be sure the work area is suitable for safe operation

- 1. Have a clean, well-lighted work area.
- 2. Have adequate ventilation.

D. Prepare the mold surface

- 1. Prepare the mold surface for release of laminate by:
 - covering it with a sheet of Mylar (3-5 mils) and taping down edges, or
 - waxing the mold surface and buffing it thoroughly.
- 2. The sheet of Mylar should be wrinkle-free and should extend well beyond the edges of the laminate. Also, if wax is used, the waxed area should extend well beyond the edges of the laminate.

E. Cut the glass fiber

- 1. Cut glass fabrics larger than the desired laminate size to permit trimming. For example, to prepare a 12"x12" finished laminate, you should cut a 14"x14" mat.
- 2. The sequence for applying layers of glass, from base to surface, is:

b. two layers of chopped mat } corrosion liner

c. one layer of woven roving

structural layer

d. one layer of chopped mat

(Repeat c and d as required for desired thickness and always finish with chopped mat.)

Note: Some specifications may require an additional surfacing veil on the exterior (atmospheric) side of the laminate.



F. Making a 1/4" laminate

The following symbols are used here to indicate the application sequence:

V = standard 10-mil corrosion surfacing veil

M = chopped strand mat of 1.5 oz/sq ft

R = woven roving glass

The sequence VMMRMRM will produce a laminate approximately 1/4" thick with a corrosion liner on one side. The side next to the Mylar will have a hard, glossy surface and should have a high Barcol value when cured. This 1/4" laminate should contain about 40% glass by weight and have the following physical properties:

- flexural strength 28,000 psi (ASTM D790)
- tensile strength 20,000 psi (ASTM D639)

G. Measuring and mixing the resin

- 1. The amount of resin to use will depend on the size of the part. A test laminate of 12"x12" will require about three 200-gram batches of resin or one 600-gram batch.
- 2. The proportion of catalyst and accelerators to use with a DERAKANE epoxy vinyl ester resin is determined by shop conditions such as temperature and humidity and by the time needed to make the lay-up.
- 3. The following formulation will give a 15- to 20-minute pot life at 70°F (21°C). The three batches of resin may be accelerated with CoNap promoter and DMA at the same time, but the catalyst MEKP should be added only immediately prior to making the laminate. Wear safety goggles, protective clothing and gloves when measuring and mixing resins, promoters and the catalyst.
 - a. measure DERAKANE resin 200 g
 - b. measure and add CoNap promoter (6%) . .0.6 cc
 - c. measure and add DMA (100%) 0.2 cc
 - d. mix thoroughly
 - e. measure and add MEKP (60%) 2.0 cc
 - f. mix thoroughly and scrape sides and bottom of mixing vessel while mixing (This final mixing should take approximately 60 seconds.)

H. De-air resin

Allow resin to stand for several minutes to de-air.

I. Apply resin

Apply a thin layer of resin with a short-bristled brush or serrated roller over a 14"-square area of Mylar. It is important to apply the resin first; then roll the glass into the resin. This will decrease the possibility of entrapping air bubbles and will assure that the glass will be saturated from the bottom up.

J. Apply the first layer of surfacing veil

- As you apply the surfacing veil, resin will bleed up through the glass. You may have to apply additional resin to wet the surfacing veil completely. This additional resin will help in wetting the next layer. When the surfacing veil loses its fibrous appearance, the glass is well-wetted.
- 2. Use a serrated lay-up roller to work the resin through the glass and work out air bubbles. Rolling also compresses the glass and removes excess resin. Roll from the center of the laminate toward the edges. Roll firmly, but not too hard.

NOTE: The proper technique is an art. You will need to develop skill in rolling to produce FRP parts that will provide optimal performance.



SECTION 8

K. Apply additional layers

The following points should be noted:

- 1. A resin-saturated surfacing veil holds about 90% resin by weight.
- 2. A chopped strand mat is harder to saturate with resin than a surfacing veil and will require much more resin. Chopped strand mats hold about 75% resin by weight.
- 3. Woven roving typically holds 40-60% resin by weight. When saturated with resin, it becomes harder to hold in place.
- 4. In all layers, the glass reinforcement should always be rolled into the resin; the resin should not be rolled into the glass.
- 5. As each layer is finished, catalyze a fresh batch of accelerated resin. Be sure to keep your roller in the solvent, clean it frequently and dry it before each use.

L. Apply a final coat of resin

When all the layers have been applied and rolled, apply a final coat of resin to cover the glass completely and seal the laminate. This final coat should contain a small amount of wax to prevent air contact, which might inhibit cure.

M. Clean up thoroughly

Clean the tools, the work area and yourself carefully and safely. Dispose of wastes properly.

N. Give laminate time to gel and cool

Let the laminate gel, exotherm and cool before removing it from the bench.

NOTE: For a high degree of cure, post-cure the laminate for 2 hours in an oven at 200°F (93°C). Leave the Mylar release sheet attached. Removing the release sheet before post-curing can produce a glossy surface, but the finish may have a slight orange-peel appearance.

GUIDE TO MAKING A CONDUCTIVE LINING

The purpose of a conductive lining system in a FRP vessel or pipe is to provide a pathway through which the possible buildup of a potentially hazardous static charge can be grounded. This process of grounding will prevent the possible arcing that can occur when a charge buildup is present.

- 1. A resin formulation containing 25 wt% graphite is applied in conjunction with a 0.5 oz/sq yd carbon veil. The graphite powder #635¹ made by Dixon Ticonderoga Co. has been found to offer consistent results with DERAKANE resins. If a different graphite is used, it is recommended that tests be conducted to ensure the resin cure will not be affected.
- 2. The resin formulation containing graphite should only be used in the carbon veil layer; then a Nexus² or C-glass veil and usual corrosion-resistant lining is applied with resin containing no graphite.
- 3. The use of graphite is not recommended in the normal corrosion-resistant lining and structural portion, because the graphite has a slight detrimental effect on secondary bonding strength. If graphite must be used throughout the FRP equipment, the overlay areas must be increased. The high loading of graphite in the resin formulation will have some detrimental effect on the corrosion-resistant properties of the cured resin laminate.
- 4. It is recommended that metal strips, such as Monel³, be placed inside the resin layer containing graphite in areas next to flanges and that the Monel metal strips be tied to the flanges. The flanges should be then properly connected to ground. Copper, brass, zinc, and galvanized metal strips are not recommended, because they have been found to prevent DERAKANE resins from curing properly.

³ INCO Alloys



Dixon Ticonderoga Co.

² Precision Fabrics

POSTCURE

For a service temperature below 100°C/210°F:

A postcure may extend the service life if the operating temperature is within 20°C/40°F of the present CR guide maximum temperature for the service. This means that a postcure can be beneficial for solvent applications with a temperature limit of 25-40°C/80-100°F.

For a service temperature above 100°C/210°F:

Postcure in service may be sufficient, provided the resin specific minimum Barcol hardness values are reached before start up.

For service in pure and neutral salt solutions:

Postcure may in general not be required, provided the resin specific minimum Barcol hardness values are reached and no acetone sensibility is shown before start up.

When using a BPO/Amine cure system, postcure is strongly recommended and should be done within two weeks of construction.

The postcure conditions as detailed in DIN 18820 may be used:

- For 411, 441, 510A/C and 8084 resins: 80°C/180°F
- For 470 and 510N resins: 100°C/210°F
- This norm recommends 1 hour per mm thickness of the laminate (between 5 and 15 hours).

SECONDARY BONDING

Secondary bonding can be a concern in many FRP applications. This is particularly true with DERAKANE 470 epoxy vinyl ester resin because of its high degree of reactivity.

To achieve a good secondary bond with DERAKANE 470 resin, it is necessary to prepare the surface properly. The entire surface must be sanded uniformly, giving at least a 2- to 3-mil anchor pattern. This can be achieved by grinding the surface with a #16 or #24 sanding disc. When using the sanding disc, it is necessary to check the pad periodically to make sure there is no build up of glass fiber and resin. If build up occurs, proper sanding and the consequent formation of a good secondary bond will be impaired.

After the surface has been properly prepared, it must be kept clean and dry until laminating can be started. Dust, moisture or traces of oil that come in contact with the surface may act as a mold release or act to inhibit the cure and prevent a good secondary bond. Laminating should be done within a couple of hours of the surface preparation.

The addition of the surfactant BYK⁴ A515, at 0.1 wt%, to DERAKANE resins can improve the adhesion of the resins to primary laminates. The surfactant properties of the BYK A515 reduce the surface tension of epoxy vinyl ester resins, thus allowing the resins to flow more easily into laminate surface cavities. For structural walls on spas and hot tubs, BYK A515, at a level of 0.2 wt%, in DERAKANE resin laminates has been shown to improve the resins' adhesion to the smooth backing of polyacrylic sheets.

NOTE: Solvent wiping of a primary surface before secondary laminating starts is not necessary. In fact, it can cause poor secondary bond formation.

A solvent wipe is usually done to remove the dust accumulated during sanding. If a rag wetted with solvent is used to wipe off the primary surface, it is likely to have picked up contaminants that might act as a mold release, and this would prevent a good secondary bond. Thorough dusting with a dry cloth is sufficient.



SECTION

APPLYING A TEST PATCH

It is good practice to apply a test patch to check the soundness of the prepared surface before the laminate is applied. To do this, first prime a 12" x 12" area with 3-5 mils of catalyzed DERAKANE 8084 resin, and allow it to cure until dry to the touch. Then apply four layers of 3" x 8" 1-1/2-oz glass fiber mat, placing a piece of Mylar film under one end to prevent bonding, and allow to cure. After the test patch is cured, pry it off by placing a screwdriver under the Mylar film.

If the test patch is good (pull glass from the primary laminate), prime the remaining surface, allow it to cure and begin lamination. If the test patch separates cleanly and easily from the mat, wash the surface with hot water to remove contaminants; then sandblast it with #3 sand and allow it to dry. When the surface is completely dry, prime a 12"x12" area of the mat with D.E.R. 331 epoxy resin and D.E.H. 58 hardener. When the primed area is cured, apply another test patch as described above. If the test patch is good, prime the remaining surface as described above and begin lamination after sandblasting. If the test patch separates cleanly and easily again, it is likely that the surface is too contaminated to accept a secondary bond.

MAKING A LAMINATE LINING

Reinforced DERAKANE resins can be used for lining steel, FRP and concrete structures and equipment, such as tanks, ductwork, sumps, pits, floors and drains, to provide optimum chemical resistance. Linings of DERAKANE epoxy vinyl ester resins are less permeable to moisture than polyester linings. Proper surface preparation is essential for laminate lining any surface. Using the procedures illustrated in the following photo essay will provide a suitable surface for most applications. After preparing the surface, you should apply a test patch to verify that it is ready for lining.

LINING A METAL SURFACE

Step 1: Clean and sandblast



Steam-clean or wash the metal with 1% sodium triphosphate to remove grease, oil or other contaminants. When the surface is dry, grit blast it to a bright metal finish. Don't wipe the surface with solvent; brush or (preferably) vacuum off the abrasive dust. The grit-blasted surface should have a 2- to 3-mil anchor pattern. Fill pits, cracks, holes and other irregularities with a catalyzed resin grout. Outside corners should have a 1/8" minimum radius, and inside corners should be filled to a 1" radius. Feather FRP edges by grinding. Allow resin grout to cure; then sand smooth and remove dust.

Step 2: Prime the surface



Brush-coat the clean metal with 2 to 3 mils of catalyzed DERAKANE 8084 resin to provide an anchor for the lining. When this prime coat is dry to the touch (usually after 6-8 hours at 70°F [20°C]), check the surface with a test patch as explained in "Secondary Bonding," page 27. If liner application is delayed, cover the primed surface to protect it from contamination. If the delay is longer than three days, the surface may have to be blasted again very lightly.



Step 3: Apply chopped strand mat



Cut two layers of 1.5-ounce chopped strand mat and two layers of surfacing veil so that the full liner thickness can be applied in one lay-up, making allowance for staggered joints with at least a 2" overlap. Apply catalyzed DERAKANE resin to the primed surface with a brush or roller; then lay a chopped strand mat over the resin, and roll with a serrated lay-up roller until all air bubbles have been removed. Repeat until two bubble-free layers of chopped strand mat have been applied.

Step 5: Inspect and repair the lining



Before topcoating, inspect the lining for defects such as voids, dry glass or exposed fibers. The 20,000-volt spark tester shown above is an effective tool for this inspection. Defects should be removed to the bare substrate by grinding. The low area should then be covered with two layers of chopped strand mat and two layers of synthetic veil applied with DERAKANE resin. The glass should overlap the surrounding area at least two inches and build up the laminate to its original thickness.

Step 4: Apply surfacing veil



Using the same sequence of operations, apply two layers of surfacing veil with DERAKANE resin. The lining should now be at least 1/8" thick.

NOTE: If any layer of mat or surfacing veil is allowed to cure overnight, it must be wiped with clean, dry rags to remove any contaminants before the next layer is applied. If a layer or an overlap is allowed to cure for more than 72 hours at 70°F (20°C), it must be etched by grinding or sandblasting before the next layer can be applied.

Step 6: Apply topcoat



Once the laminate has passed inspection, apply a resin/wax topcoat to prevent air inhibition. (See "Air Inhibition and Wax Topcoats" on page 13.)





SECTION 8

LINING AN FRP SURFACE

Step 1: Sandblast or grit blast the surface

Blast the surface until sound FRP is exposed. The FRP surface must be free of dirt, grease and other foreign matter. Don't wipe the surface with solvent; brush or (preferably) vacuum off the abrasive dust. The exposed surface should have a 2- or 3-mil anchor pattern. Any portion of the structure removed during surface preparation should be restored before a liner is applied.

Step 2: Prime the surface

Immediately after the dust has been removed, apply 2 to 3 mils of catalyzed DERAKANE 8084 resin with a brush or roller to provide an anchor for the lining.

Steps 3, 4: Apply the laminate

Apply the laminate in two phases:

- A. Apply two layers of 1.5-ounce chopped strand mat and catalyzed resin. It should be allowed to cure until dry to the touch before applying the second phase.
- B. Apply one layer of 1.5-ounce chopped strand mat and catalyzed resin and two layers of a surfacing veil. Joints on the second phase should be positioned so that they are not over joints of the first phase.

Steps 5, 6: Inspect, repair and topcoat

Follow the procedures given for laminate lining metals.

LINING A CONCRETE SURFACE

Step 1: Sandblast or grit blast the surface

The concrete must be free of dirt, grease and other foreign matter. Sandblast until clean aggregate is exposed.

Step 2: Prime the surface

Apply 2 to 3 mils of catalyzed DERAKANE 8084 resin with a brush or roller to provide an anchor for the lining.

Steps 3, 4: Apply the laminate

Apply three layers of chopped strand mat and two layers of surfacing veil, using the procedure given for laminate lining metals.

Steps 5, 6: Inspect, repair and topcoat

Follow the procedures given for laminate lining metals.

SAFETY, PLANNING AND MAINTAINING WORK AREA

The comments on safety, planning and maintaining the work area given in "Guide to Making a Laminate" on pages 24-26 also apply to laminate lining.



EPOXY VINYL ESTER RESIN ANCHORS FOR CONCRETE

For service temperatures of up to 180°F (82°C), DERAKANE 8084 epoxy vinyl ester resin can be used in a putty mix to produce anchors in concrete tank walls. To install the anchors, two-inch-diameter holes are drilled two inches deep in the concrete on two-foot centers. These holes are filled with freshly catalyzed DERAKANE 8084 resin putty, leaving the surface of the anchor flush with the surface of the concrete wall.

ABRASION

Fast flowing process streams containing solid particles can be very abrasive to the surface of FRP equipment, such as tanks, piping and ducting. Particles that are less than 100 mesh (150 microns) in size are not a major abrasion threat, while liquid velocities of less than 6 ft/sec are no problem for standard FRP systems. However, when flows and particle sizes get too large, steps must be taken to resist the abrasive effect. One practice is to make the corrosion liner twice the normal thickness in order for the liner to last longer. Another, possibly more cost- and service-effective alternative, is to incorporate hard, generally corrosion-resistant material in the veil portion of a corrosion liner or throughout a normal corrosion liner. Some materials generally used for this application are silica, silicon carbide and alumina. Table 8.1 shows the ability of different resins and abrasion-resistant additives to resist surface wear.

Table 8.1 - DERAKANE epoxy vinyl ester resins taber abrasion testing²

Laminate Description	Wear Index
DERAKANE 411 Resin	388
DERAKANE 470 Resin	520
DERAKANE 8084 Resin	250
DERAKANE 411 Resin 10% Fine Silica	70
DERAKANE 411 Resin 50% Fine Silica	38
DERAKANE 411 Resin 66% Fine Silica	38
DERAKANE 411 Resin 20% Silicon Carbide	25
DERAKANE 411 Resin 40% Silicon Carbide	10
DERAKANE 411 Resin 50% Silicon Carbide	10

¹ Mallinson, John H., Corrosion-Resistant Plastic Composites in Chemical Plant Design, Marcel Dekker, Inc., New York, New York, 1988. ² CS-17 Abrasive Wheel, 1000-gram loading

Inspecting a laminate

CHECKING FOR COMPLETE CURE

Barcol hardness values are used to indicate the completeness of resin cure in FRP laminates. To test for Barcol hardness, use the Barcol Tester #934.1* to take readings on both the inside and outside surfaces of the part (10 to 12 readings in all). Reject the highest and lowest readings, and average the remaining values to get the Barcol hardness value.

Key points

- Calibrate the Barcol Tester with both high and low hardness discs, and check it frequently.
- Use the Barcol Tester at room temperature (about 77°F [25°C]).
- When using the tester, keep it level. Be sure the point is perpendicular to the laminate surface.
- Clean or replace Barcol points frequently.
- Be sure to take measurements at secondary bonds, such as flanges and internal connections. Values at these points are critical.
- Laminates with high glass filling may give abnormally high Barcol values when the point hits glass.
- Contacting a synthetic veil may cause a drop of 2 to 3 points.
- Improper catalyst ratios may cause low Barcol readings.
- Incomplete catalyst mixing may cause widely varying readings.
- Heavy residual wax on the surface will cause low Barcol readings. Lightly sand off 1 to 2 mils of wax and test again.

RECOGNIZING FRP DEFECTS

Most FRP inspection, other than hardness checking, is visual, because physical testing is generally destructive. However, defects can be identified by observing the finished construction. The list of defects found on pages 33-35 can be used to help identify problems and correct fabricating techniques.

Table 9.1 – Typical Barcol Tested Hardness

	Hardness Values		
	Typical 24-Hour RT¨ Cure	Typical Force Cure	Typical Acceptable Minimums
DERAKANE 411 Resin	20-30	35	30
DERAKANE 441-400 Resin	20-30	35	30
DERAKANE 470 Resin	30-40	45	40
DERAKANE 510 Resin	20-30	40	35
DERAKANE 8084 Resin	15-25	30	25

^{*} Product of Barber-Coleman Company

This bulletin contains tips and suggestions, but no warranty, expressed or implied, is given; fabricators are advised to determine suitability as to any product, additive, process, formula, ratio, or end-use through their own expertise or tests.



^{**} RT = Room Temperature



Defect	Nature	Possible Cause
Air bubbles, voids	Air entrapment in and between plies; non-interconnected spherical voids.	Whipping action during mixing. Poor or dirty surface. Air bubbles not rolled out during lay-up.
Blisters	Rounded, sometimes sharply defined elevations of laminate surface resembling blisters.	Too rapid cure. Moisture in resin, filler or glass.
Impact cracks	Separation of material through entire thickness and visible on surfaces.	Impact, insufficient reinforcement, or high concentration of resin in one area.
Crazing	Pattern of fine cracks on or beneath surface.	Isolated resin-rich area. Impact. Intermittent service causing temperature differences, wetting/drying cycling and resin shrinkage.

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SECTION 9

Defect	Nature	Possible Cause
Delamination	Separation of layers.	Poor glass saturation. Dirty surface. Too high glass content. Failure to remove air-inhibition coat before adding layer.
Fisheye	Small globular mass that has not blended into surrounding material. Particularly evident in transparent or translucent materials.	Dirty surface. Foreign matter falling on resin.
Dry spots	Area of reinforcement that was not wetted with resin. Usually at laminate edge.	Not enough resin used. Applies mostly to molding operations.
Pimple	Small sharp or conical pimple-like elevation on surface. Usually resin-rich.	Dipped resin or small bubble. Working on area after resin has started to cure.

9

Defect	Nature	Possible Cause	
Pit or pinhole	Small regular or irregular crater on surface, usually with nearly equal width and depth.	Air bubbles that rise to surface, which cure before smoothing out.	
Resin pocket	Apparent accumulation of excess resin in a small localized area.	Improper lay-up (glass separates and void fills with resin.)	
Scratch	Shallow mark, groove, furrow or channel.	Improper handling or storage.	
Worm holes	Elongated void in surface or covered by thin film of cured resin.	Usually found in small-diameter pipe where cure is from the outside and progresses toward the mandrel.	
Wrinkle	Crease or wrinkle-like surface imperfection in one or more plies of molded-in reinforcement.	Improper lay-up. Too rapid cure. Use of old resin. Fold in release film on the mold.	

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SECTION 10

Other processing techniques

As we stated in Sections 1 and 2, FRP is made with reinforcement material, such as glass or carbon fibers, along with resin and additives. The reinforcement material provides strength and structure, while the resin holds the fibers in place and protects them from corrosion and abrasion.

The fabricator applies the resin and fiber materials in layers by one of several fabricating techniques. This "layering" process gives the fabricator freedom to mold intricate shapes.

Fabricators today have the technical sophistication to custom design and build pieces of equipment tailored to demanding design considerations. The fabricating technique you choose depends on factors like the size, shape and complexity of the equipment; performance and appearance requirements; volume; and cost. This section describes the most commonly employed fabricating methods used with vinyl ester resin systems.

FORMULATING FOR SPRAY-UP APPLICATIONS

One-part system

For spray-up and chopper gun applications, accelerators and promoters are added directly to a 55-gallon drum and mixed thoroughly into the resin with an air-driven bunghole mixer. Collapsing-propeller type agitators give good results. A minimum of 25 minutes mixing is needed to ensure homogeneity. Typical percentages of promoters and accelerators for DERAKANE 411-45 resins are listed below.

This system can then be catalyzed with 1 to 2% MEKP (9% active oxygen) with an injection-type catalyst gun and will have a 10- to 20-minute pot life. There should be no moisture or oil in either the air line or the catalyst line. The operator should run a small amount of resin through the gun to check for gel time before fabricating parts.

Product	Percentage ¹	Oz per 55-gal drum¹	
Cobalt Naphthenate 6%	0.3	18.8	
Dimethylaniline	0.1	6.3	

Fabricators should confirm through their own tests.

Note: Shelf life is several weeks.

Two-part system

An alternative technique is to work with a two-part system using equal portions of prepromoted resin and catalyzed resin. In one pot, double quantities of CoNap promoter and dimethylaniline are added to resin; in the second pot, a double quantity of peroxide is mixed into the resin. Mixing the components of both pots provides the proper amount of promoter and catalyst.



Product	Percentage	Oz per 55-gal drum		
Part A, Drum DERAKANE 411-45 Resin Dimethylaniline 100% Note: Shelf life is several weeks.	0.1-0.3	6.3-18.8		
Product (active)	Percentage (active)	Oz per 55-gal drum		
Part B, Drum DERAKANE 411-45 Resin Benzoyl Peroxide	2.0-4.0	126-252		

Note: Mix only as much as will be used within 4 hours. Please follow suppliers' recommendations for use, maintenance, purging, etc. of spray guns and other equipment.

FILAMENT WINDING

Filament winding is a process in which continuous rovings are wetted out in a resin bath, then wound (usually in a helical pattern) onto a rotating mold called a mandrel. Glass veils and mats are commonly used on the inside surface of filament-wound pipe to provide a resin-rich, corrosion-resistant liner.

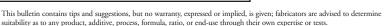
- 1. Resins DERAKANE epoxy vinyl ester resins are formulated for optimum filament winding viscosity. Filament winding usually requires a resin formulation with a viscosity between 300 and 2,000 cP. If the viscosity is too low, the resin may drain away from the fibers after application, leaving the laminate dry, especially on the inside surface. If resin viscosity is too high, the fibers will be difficult to wet out.
- 2. Glass Continuous roving is the primary reinforcement used for filament winding. Available weights (yields) of continuous rovings range from 113 yd/lb to 675 yd/lb. The following continuous rovings have been found satisfactory for use with DERAKANE epoxy vinyl ester resins:
 - Hybon¹ 2000 Series
 - Type 30
 - R099 625 & 673

Table 10.1 – Viscosities of DERAKANE epoxy vinyl ester resins for filament winding

Product	Typical Viscosity² (cP @ 77°F [25°C])
DERAKANE MOMENTUM™ 411-350 Resin	370
DERAKANE 411-45 Resin	500
DERAKANE 441-400 Resin	430
DERAKANE MOMENTUM 510C-350 Resin	380
DERAKANE 510N Resin	275
DERAKANE 510A Resin	400
DERAKANE MOMENTUM 470-300 Resin	325
DERAKANE 470-36 Resin	240
DERAKANE 8084 Resin	360

¹ PPG Industries, Ohio

² Not a sales specification



DERAKANE"

SECTION 10

Higher yield rovings generally wet out more easily, but more strands are required to achieve the desired glass content. Continuous rovings are sized to reduce damage to the glass during fabrication. Glass suppliers also add coupling agents to improve compatibility and adhesion of resin to the glass.





Surfacing veils and chopped strand mats used on the inside surface of filament-wound pipe to provide a resin-rich liner for enhanced chemical resistance are usually applied to the mandrel by hand. The technique here is similar to hand lay-up on a flat surface. The resin is applied to the mandrel surface first; then the glass veil or mat is applied and wetted-through by the resin. Applying the liner usually requires two people who unroll the veil or mat onto the mandrel in a helical pattern.

3. Peroxide initiators – Elevated temperature peroxide initiators are used when the fabrication equipment is capable of applying heat to the uncured laminate. Many peroxides are commercially available for use in elevated temperature curing of vinyl ester resins.

Peroxide initiators are generally chosen for their ability to provide the best combination of rapid cycle time and laminate quality. The activation temperature of the peroxide is an indication of how rapidly it will catalyze a resin. Table 10.2 gives 10-hour half-life temperatures for four of the peroxide initiators commonly used with DERAKANE epoxy vinyl ester resins. Ideally, peroxides with a lower 10-hour half-life temperature will give shorter gel times at any given resin temperature.

By use of different catalyst systems, the fabricator can achieve a wide range of gel times over a broad temperature range. In addition, certain blends of peroxides may offer advantages, such as reduction of peak exotherm temperature or other special properties.

- **4. Room temperature cure** DERAKANE epoxy vinyl ester resins also can be formulated for room temperature cure. Catalyst systems recommended for these formulations are essentially the same as those used for hand lay-up. 2,4-Pentanedione may be used in combination with a cobalt-promoted system to extend room temperature pot life. (See Section 1 for further information.)
- 5. Internal mold release agents Internal mold release agents may be used (typically at a use level of 0.5 percent) in conjunction with waxed surfaces in specialized (elevated temperature) molding and filament winding applications. One brand that works well with DERAKANE resins is Zelec⁴ UN release agent.

Table 10.2 – 10-hour half-life temperatures of four peroxide initiators

- a) Perkadox 16N (bis(4-tert-butyl cyclohexyl) peroxydicarbonate)
 - 10-hour half-life temperature = 109°F (43°C)
- b) Dibenzoyl peroxide (BPO)
 - 10-hour half-life temperature = 162°F (72°C)
- c) Tert-butyl perbenzoate (TBPB)
 - 10-hour half-life temperature = 223°F (106°C)
- d) Cumene hydroperoxide (CHP)
 - 10-hour half-life temperature = 318°F (159°C)



PULTRUSION

Pultrusion is a process used to produce parts of constant cross section. It is a continuous process in which various reinforcements are first pulled through a resin wet-out bath and then through a heated die where the resin is allowed to cure, and a saw located down-stream from the pulling device cuts the part to the desired length.

- 1. Resins The use of DERAKANE epoxy vinyl ester resins helps assure production of highquality pultruded parts. The primary component of formulations used for pultrusion is the resin itself. DERAKANE resins are well suited for this process, because they provide excellent mechanical properties, toughness, thermal resistance and chemical resistance. The following DERAKANE resins are used for pultrusion:
 - DERAKANE MOMENTUM™ 411-350
 - DERAKANE 411-45
 - DERAKANE 441-400
 - DERAKANE 470-36
 - DERAKANE MOMENTUM 470-300
 - DERAKANE MOMENTUM 510C-350
 - DERAKANE MOMENTUM 640-900
 - DERAKANE 8084

Pultrusion formulations also include initiators, fillers, internal mold release additives and sometimes flame-retardant additives and pigments. A typical pultrusion formulation will have a viscosity of between 700 and 5,000 cP. The appropriate viscosity is dependent primarily on the type of reinforcement used in the laminate. When a glass mat or veil is used, a higher viscosity mix will be required to prevent excessive resin runoff.

- 2. Fillers Fillers are commonly used in pultrusion formulations. Those most frequently used are bentonite clays such as aluminum silicate or calcium carbonate. The level of filler to be used is greatly dependent on the desired mix viscosity. Fillers not only make the overall system more economical, they also help in the production of parts that undergo large exotherms in the die. For corrosion resistance applications, the effect of the filler on the corrosion resistance of the laminate should be well characterized before specific recommendations are made.
- 3. Catalyst system Various catalyst systems are used with DERAKANE epoxy vinyl ester resins for pultrusion. Often two or more initiators will be used together in a formulation. Typically, a low-temperature activating catalyst is used to initiate an early reaction in the die with an elevated-temperature catalyst to assure complete cure.

Many different peroxides may be used, depending on the resin, the part geometry and equipment parameters. Typical initiators used for pultrusion are Perkadox⁵ 16N, tert-butyl peroctoate, benzoyl peroxide and tert-butyl perbenzoate. (For specific starting formulations, refer to www.derakane.com.)

- 4. Internal mold release An internal mold release is required for pultrusion. These additives are used at concentrations ranging from 0.5 to 1.5 parts per hundred parts resin. Commercial mold releases that perform well with DERAKANE epoxy vinyl ester resins include the following:
 - Molgard⁶
 - Ortholeum⁷ 162
 - NT PS 1258
- 5. Reinforcement The reinforcement most often used for pultrusion is continuous roving, generally a heavier roving (greater than 250 yield). Good quality roving with epoxy vinyl ester resin compatible sizing are available from Owens Corning, PPG and Saint-Gobain Vetrotex.



Akzo Nobel Polymer Chemicals, LLC

E. I. DuPont de Nemours & Co., Inc 6 Whittaker Corporation

SECTION 10

The type of glass mat most commonly used in pultrusion is continuous strand mat. These mats are readily cut to the desired width and can be formed to the shape of intricate parts. Perforated polyester and other synthetic veils are used to produce a resin-rich surface on pultruded parts.

6. Die temperature settings – In the production of high-quality parts, die temperature settings are critical. The choice of catalyst, catalyst level, reinforcement level and resin type will all affect the proper die temperature setting. When trying a new system, it is best to begin with a relatively low die temperature setting and gradually raise the temperature to find the optimum operating conditions.

For producing thick parts, it may become necessary to use a radio frequency (RF) preheater. The RF preheater is used to heat the inside of a part before the part enters the die. Preheating increases line speed and helps prevent internal cracking of the part.

RESIN TRANSFER MOLDING

Resin transfer molding (RTM) is a process that uses a mechanical pumping apparatus to transfer catalyst and resin from holding tanks, through a mixing device, into a closed mold containing reinforcement material.

- 1. Resin DERAKANE epoxy vinyl ester resins are available at appropriate viscosities for resin transfer molding. RTM requires a low viscosity resin to allow for a rapid filling of the tool (mold), low pumping pressures and good wet-out of the reinforcement. Resins with viscosities of less than 250 cP are generally best. The following DERAKANE epoxy vinyl ester resins have been used successfully in RTM:
 - DERAKANE 411-45
 - DERAKANE MOMENTUM™ 411-350
 - DERAKANE MOMENTUM 510C-350
 - DERAKANE 470-36
 - DERAKANE 8084
- 2. Reinforcements Many types of reinforcements may be used in the RTM process; the most common is continuous strand mat, such as OC M8608°. Continuous strand mat is available in several different weights. Directional stitched mat is often used to provide specific mechanical properties in a given direction.

Preformable mats also are available. A thermoplastic binder in these mats allows the reinforcement to be heated and then formed into the desired shape in a separate operation. Preforms allow the reinforcement to fit into the mold quickly. This results in faster cycle times, because it eliminates the need for time-consuming placement of the fibers at the production press.

3. Catalyst formulations – Both elevated-temperature and room-temperature catalyst formulations can be used in the RTM process. To be suitable for this process, the catalyst must be a pumpable liquid. Elevated-temperature initiators and resins are placed in separate tanks and mixed before being pumped into a heated mold.

Room-temperature catalyst formulations are similar in composition to formulations used in hand lay-up, except that the resin is usually divided into two streams to be mixed in equal amounts. Half the resin is mixed with a peroxide catalyst, and the other half is mixed with the promoter and accelerator. The two streams are pumped through the mixer into either a heated mold or a room-temperature mold.

Catalyst formulations similar to those used at room temperature also can be used at intermediate temperatures (between 100°F [38°C] and 180°F [82°C]) to improve the cycle time. These systems can be very useful for obtaining rapid cycle times at relatively low temperatures. The catalyst formulation and mold temperature must be adjusted to assure a full cure.

- **4. Mold releases** Internal or external mold releases may be used in RTM. A common technique is to maintain easy part removal by use of an internal mold release and occasionally apply a coat of external mold release as needed. The following mold releases have worked well with DERAKANE resins:
 - Kantsik¹⁰ FX9
 - INT11 EQ-6
 - Zelec¹² UN

The use of some silicone-based mold release agents has been shown to inhibit the cure of DERAKANE resins.

NOTE: Use an external mold release with room-temperature catalyst systems. Gel times of room-temperature catalyst systems are greatly increased by the presence of internal mold releases, including those mentioned above.

5. Equipment – RTM equipment includes a metering and pumping device for the resin formulation. This usually consists of two positive displacement piston pumps, which deliver the same ratio of each component for every shot. RTM also requires molds designed to assure complete air release and complete wet-out of the reinforcement.

A clamping mechanism for holding the mold closed during injection and curing of the resin is also required. Clamping can be accomplished by the use of bag molds, mechanical presses or hydraulic presses. For high production volume applications, an automated press may be used to achieve shorter cycle times.

6. Tooling – Tools for RTM may be made from several different types of materials, which vary greatly in cost and durability.

Composite tooling or epoxy tooling is inexpensive and easy to obtain. Epoxy tools are an excellent choice for making prototype parts or for low-volume production. However, they have a limited life and may only be heated to moderate temperatures.

Aluminum or steel tools should be used for processes requiring high pressures, high temperatures and high volume production. Metal tools can be designed for heating with hot waters, steam, or oil. Aluminum tools are less expensive than steel tools, but also are less durable.



¹⁰ Specialty Products Co.

[&]quot; Axel Plastics Research Labs., Inc.

¹² E.I. DuPont de Nemours & Co., Inc.

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SECTION 11

Section 11 - Industrial health information

SAFETY DATA SHEETS

The information provided in Safety Data Sheets applies only to DERAKANE and DERAKANE MOMENTUM™ resins and not to other products used in formulating, including initiators, promoters, accelerators, solvents and other additives. Specific safety and handling procedures for these additives should be requested from the product manufacturer. When various materials are used in combination, formulators should follow the most stringent safety and handling procedures applicable to the products being used.

Safety Data Sheets for DERAKANE and DERAKANE MOMENTUM epoxy vinyl ester resins are available from Ashland to help users of DERAKANE resins meet their own handling needs and the requirements of OSHA and other governmental agencies. The following comments pertain to all DERAKANE resin products and are general guidelines for handling these resins safely. The Safety Data Sheets for the specific DERAKANE resin product should be consulted for more detailed health hazard and handling information before using the product.

CAUTION: Do not blend promoters directly with peroxide, since rapid decomposition or explosion could occur.

BASE RESINS AND STYRENE DILUTIONS

DERAKANE epoxy vinyl ester resins are made from epoxy resin methacrylates. The base resins themselves do not pose any significant health or handling difficulties. These base resins, however, are diluted with styrene monomer. Because of their styrene monomer content and the recent focus on health issues concerning styrene monomer, it may be advisable to review exposure levels and the recommended procedures for handling DERAKANE resins in your facility. (Recommended procedures for storing DERAKANE resins are provided in Section 12 of this bulletin.)

PHYSICAL PROPERTIES

The notation "based on styrene" appears beside several of the physical properties listed in Table 11.1. This means that the value reported applies to styrene itself, not to the resin, and therefore represents a "worst case" scenario.

The boiling point of styrene is lower than the exotherm temperature often reached during resin polymerization. For this reason, secondary laminating sequences should be scheduled so that exotherm occurs at nearly the same time in all the resin or after exotherm in the primary laminate is finished.

Table 11.1 – Liquid property data for typical epoxy vinyl ester resin¹

Boiling point	294°F (146°C) (based on styrene)
Vapor Pressure	7 mm Hg @ 68°F (20°C) (based on styrene)
Vapor Density	3.6 (based on styrene)
Solubility in water	Insoluble
Specific gravity	1.02 - 1.22
Appearance	Yellow to amber viscous liquid
Odor	Pungent styrene odor

¹ Not to be construed as specifications



Pure styrene vapor is heavier than air; thus, if ventilation is poor, the vapor evolved may not completely mix with air. This may cause a condition where styrene fumes may be more concentrated at floor level than at the ceiling. This point is important to keep in mind in designing general or local exhaust ventilation systems for FRP shops.

The odor threshold for styrene has been reported to be less than 1 ppm. That is why even cured pieces of FRP may still have a faint styrene odor.

FIRE AND EXPLOSION HAZARD DATA

DERAKANE resins are classified as flammable materials under NFPA 30. Keep it away from heat, flames and spark-producing equipment.

The burning characteristics of the liquid resin are comparable to those of styrene. The red FLAMMABLE label is required on drums, since the resins have a flash point under 100°F (38°C). Keep away from all sources of ignition. There should be absolutely no smoking in the work or storage areas. Caution should be exercised with respect to the location of flame-fired equipment such as hot water heaters, space heaters, etc.

The flash point is the minimum temperature at which the material gives off a vapor in sufficient quantity to ignite in air, as tested by one of the approved methods. Since the flash point is less than 80°F (27°C), flammable concentrations of vapors may be present when liquid DERAKANE resins are being used in the FRP shop.

Table 11.2 - Flash point and flammable limits in air

Flash point	74-84°F (23-29°C)
Flammable limits in air Lower limit Upper limit	1.1% (based on styrene 6.1% (based on styrene)

The lower flammable limit is the minimum concentration of a vapor in air which will burn when exposed to an ignition source. Conversely, the upper flammable limit is the maximum concentration of a vapor that will burn when exposed to an ignition source. These limits are specific for a given temperature and are usually given for 77°F (25°C). It is important to prevent the formation of explosive or combustible mixtures and to take precautions to avoid ignition of any such mixtures formed. Resin handling areas should be well ventilated and motors must be explosion-proof or totally enclosed. All equipment, drums, tank trucks and hose connections must be grounded for the safe discharge of static electricity.

IN CASE OF FIRE

Fires involving DERAKANE resins can be extinguished safely with foam, dry powder or carbon dioxide. Water is not normally an effective extinguishing agent for use with these water-insoluble resins. The use of foam requires caution if motors, hot plates, fans or other kinds of electrical equipment are involved in the fire. When these resins burn, they may give off toxic by-products such as carbon monoxide. For this reason, breathing of fumes, smoke and vapors liberated by a resin fire should be avoided. If there is a possibility of gross exposure to these toxic by-products, a positive-pressure, self-contained breathing apparatus, or equivalent, may be required.

Extra care should be taken in fire situations in which other resin drums are present in the area. When the drums are exposed to heat or flame, an exothermic reaction can develop, possibly accompanied by hazardous decomposition of the product. The gases generated by decomposition may include carbon monoxide. Decomposition of brominated resins may release hydrogen bromide.



SECTION 11

REACTIVITY DATA

DERAKANE resins are reactive and can polymerize even without the addition of peroxides. Polymerization initiated without peroxides is usually the result of inhibitor depletion and the reaction proceeds slowly. However, exposure to high temperature, sunlight, or certain other chemicals can result in rapid polymerization with significant generation of heat. When this happens, decomposition gases such as carbon monoxide may be formed. It is best to store drums of DERAKANE resins indoors or away from direct sunlight. However, when the material is being used on a field job, this is not always possible. For that reason, the drums are painted white to limit the amount of heat they will absorb.

FIRST AID

Eye contact

Styrene vapors, if present at a high enough concentration in the air, may cause tears and eye irritation. If the eyes become irritated, ventilation needs to be improved. Workers should protect their eyes from splashes by using chemical goggles. If resin is accidentally sprayed into the eyes, it is important for the person to irrigate them continuously with low-pressure for at least 15 minutes. Medical personnel should be notified immediately. Include in the description of the accident other components of the mixture (peroxides, promoters and fillers), because this information may affect the treatment the physician recommends.

Skin contact

If resin comes in contact with the skin, it should be washed off with soap (if available) and water, not solvents. Prolonged or repeated contact with the skin may cause localized irritation, even a burn. Also, the sticky nature of the resins makes them very difficult to wash off exposed skin. Wearing protective body-covering clothing and impervious gloves can help minimize direct skin contact with the resin.

Inhalation

Inhalation of styrene vapors is the principal respiratory health concern associated with the use of DERAKANE resins. Exposure to high concentrations of vapors may lead to central nervous system effects (anesthesia or narcosis) and cause upper respiratory irritation.

Effective March 1989, the OSHA Permissible Exposure Limit (PEL) for styrene became 50 ppm as an 8-hour Time Weighted Average (TWA). In addition, there is a Short-Term Exposure Limit (STEL) of 100 ppm as a 15-minute TWA.

A person adversely affected by styrene vapors should be removed to fresh air, made to rest and kept warm. If the individual is not breathing, mouth-to-mouth resuscitation should be given. Medical attention should be obtained immediately.

Ingestion

If DERAKANE resins are accidentally swallowed, medical attention should be sought immediately. The physician should treat the condition symptomatically and should not induce vomiting.



PERSONNEL PROTECTION

Human contact with resins, initiators and other modifiers should be minimized. Use of chemical goggles, clean, body-covering clothing and impervious gloves will reduce the potential for skin and eye contact. Proper ventilation and any appropriate engineering procedures should be used in the work area to maintain styrene monomer vapor concentrations below the applicable government-regulated levels. Good general ventilation should be satisfactory for most conditions. However, local exhaust may be required where emissions of vapor are high and where there is little air circulation, particularly in confined areas.

If respiratory protection is required, air-purifying respirators with organic vapor cartridges may be used for styrene concentrations up to a recommended maximum of 500 ppm. The service life of these cartridges depends on vapor concentration, the user's breathing rate, and relative humidity, among other factors, and should be determined for each work environment. The cartridges should be replaced if styrene odor becomes noticeable to the respirator wearer. If there is a possibility of gross styrene exposure, a positive-pressure, self-contained breathing apparatus, or equivalent, may be required.

Cured DERAKANE resins – those that are completely polymerized – are considered to be toxicologically inert and to present no handling hazards. The finished resins, however, may represent a health hazard from inhalation of dusts generated during grinding or machining, especially if they contain glass, silica powder, asbestos or metal powders.

SPILLS

Small spills of DERAKANE resin can be handled by applying sand or other absorbent material to the spill and shoveling it into a container. The residue should be removed from the floor with hot, soapy water. Larger spills should be contained with a dike and the excess resin should be collected in containers. Again, the residue should be removed with hot, soapy water.

CAUTION: Solvents should not be used in the final cleanup, because their use could create unnecessary hazards of vapor inhalation and possible ignition.

Only trained personnel should participate in the cleanup, and they must be properly protected from skin and eye contact and breathing vapors. See "Personnel Protection" above. Before any cleanup begins, the spill area should be checked carefully and any possible sources of ignition should be removed.

ENVIRONMENTAL AND DISPOSAL INFORMATION

Any disposal practice must be in compliance with all federal, state/provincial and local laws and regulations. State/provincial and local requirements for waste disposal may be more restrictive or different from federal laws and regulations. The preferred waste management option for unused, uncontaminated, unformulated, unaltered DERAKANE resins is to send them to a licensed or permitted recycler, reclaimer or incinerator. The same waste management options are recommended for used or contaminated material, although additional evaluation is required. DERAKANE resins that are fully polymerized are considered to be toxicologically and ecologically inert and should be disposed of properly.

DERAKANE resins can be disposed through burning in an adequate incinerator. Brominated resins (DERAKANE 510A, 510N, and 510C-350) must be burned in an incinerator equipped with a halogen scrubber.



SECTION 11

Current regulations allow for cured resins to be disposed in an approved landfill in accordance with federal, state and local regulations. This is another area of emerging regulations. The regulations which may have an impact on disposal of cured waste resin include RCRA, California Proposition 65 and Rule 1162. As these rules become defined further, Ashland will be working with fabricators and distributors to keep them informed.

NOTE: In disposal of any wastes, all federal, state and local regulations must be met.

HEALTH AND HAZARD DATA

In March of 1987, The International Agency for Research on Cancer (IARC) decided to reclassify styrene from its 1982 status, "not classifiable as to carcinogenicity to humans," to Group 2B as a "possible carcinogen to humans." This decision was based on new criteria for evaluation and did not reflect the generation of any new carcinogenicity data since the previous classification five years earlier. In fact, no animal exposure studies have been identified which demonstrate conclusively that styrene is carcinogenic.



Section 12 - Resin storage

STORAGE TEMPERATURES

Storage temperatures for DERAKANE epoxy vinyl ester resins should be maintained below 77°F (25°C) for maximum shelf life. DERAKANE resins should be kept away from all sources of heat which might create localized hot spots.

DERAKANE resins are stable at ambient conditions, but, if handled improperly, may polymerize slowly. In fact, exposure to sunlight or other sources of high temperature can lead to rapid polymerization with dangerous exotherm. Fifty-five-gallon drums should be stored in a cool, dry place out of direct sunlight. The drums are painted white to minimize heat absorption from sunlight.

RESIN SHELF LIFE

DERAKANE resins provide superior performance in reinforced plastic applications. This is due, in part, to their high degree of reactivity. A side effect of high reactivity, however, is somewhat limited shelf life. The shelf life warranty for each resin is stated on the drum.

DERAKANE resins should be used within the stated shelf life. Ashland cannot honor any claims for credit on DERAKANE resin that has been stored beyond the maximum storage time. DERAKANE epoxy vinyl ester resin's shelf life is based on the date the resin was manufactured.

INVENTORY ROTATION

To minimize potential shelf life problems, you should maintain your inventory of DERAKANE resins on a first-in, first-out basis, i.e., always use your oldest resin first.

Table 12.1 - Resin shelf life

	Resin (N	nelf Life Months, '°f [25°C])
	DERAKANE 411-45	6
	DERAKANE 411-350	7
	DERAKANE 411-350PA	6
	DERAKANE MOMENTUM™ 411-35	50 12
	DERAKANE 411-700PAT	3
	DERAKANE 441-400	9
	DERAKANE MOMENTUM 640-900	6
	DERAKANE 470-36	3
	DERAKANE 470-300	4
	DERAKANE MOMENTUM 470-300	10
	DERAKANE 470HT-400	7
	DERAKANE 510A-40	4
	DERAKANE 510C-350	4
	DERAKANE 510C-350FR	4
	DERAKANE MOMENTUM 510C-35	50 12
	DERAKANE 510N	4
	DERAKANE 8084	6
	DERAKANE 8090	3
\	Experimental Products	3
`		

AERATING THE RESIN

Because inhibitors in DERAKANE resins work by reacting with oxygen to prevent premature cross-linking, periodic replenishing of the dissolved oxygen will extend the resin's shelf life. This operation should be done only in well ventilated areas free from ignition sources, because flammable vapor mixtures may be formed in the process.

The recommended way to replenish dissolved oxygen is by stirring the resin in the drums with both the bung and vent caps removed, using an air-powered drum mixer. Another possible way is to bubble small amounts of clean, dry air into the drums through a dip pipe.

NOTE: The dip pipe should not contain any zinc or copper alloy, since these materials could change the reactivity of the resin.

NOTE: DERAKANE MOMENTUM $^{\text{\tiny M}}$ resins do not require periodic aeration to maintain storage life. These resins should not be reaerated.

For increased safety, a 2:1 mixture by volume of nitrogen and air may be used to keep gas exiting the drum during sparging below the styrene/gas flammability limits. Both the drum and the sparging line should be grounded.



SECTION 12

Oxygen also can be replenished by opening the bung and the vent in the head space above the resin. After this is completed, the drum should be closed tight, then rolled on its side to mix the air into the resin.

Any steps to extend the shelf life of DERAKANE resins must be taken before the resin starts to gel. If the resin has begun to gel, it should be discarded in accordance with approved disposal procedures, since the gelled particles would not cure properly, and finished products made with the resin might have unsatisfactory chemical resistance.

BULK STORAGE

DERAKANE resins are frequently stored in bulk storage tanks. A properly designed bulk storage system should do the following:

- Eliminate flammability hazards
- Protect resin against temperature extremes
- Prevent contamination
- Retard polymerization in infrequently used lines

The following procedures must be followed carefully to ensure proper bulk storage of DERAKANE resins:

- 1. Maintain resin temperature below 77°F (25°C) in an electrically grounded vessel. (Continuous monitoring of the bulk resin temperature is recommended.)
- 2. Periodically circulate the resin in the storage tank and process lines to prevent polymer buildup in stagnant sections.
- 3. Keep resin properly aerated by circulating the contents of the storage tank to ensure complete turnover of the resin. This can be controlled with Step 2.
- 4. Use proper materials for containing, transferring and storing the resin. Suitable materials could be, for example, mild steel, black iron, or 304 stainless steel. Alloys containing copper or zinc should not be used; they can adversely affect the reactivity of the resin. Rubber parts should also be avoided, because styrene monomer is an excellent solvent for most rubbers except Viton¹ fluoroelastomer.
- 5. Check resin gel time periodically to ensure continued high resin quality.
- 6. Use the resin within the suggested storage life.

STORAGE OF PROMOTED RESIN

DERAKANE resin that has been promoted by the customer with cobalt naphthenate and/or dimethylaniline or modified with other additives may have a substantially shorter shelf life than unmodified DERAKANE resins. Because of the possibility of reduced shelf life, any formulating of resin required for fabrication should be delayed until shortly before use to ensure consistent properties. Formulated resin should be used as quickly as possible to prevent problems from occurring.

Because additives can have a destabilizing effect on DERAKANE resins, the stated storage times do not apply to resins that have been modified from their "as received" condition.

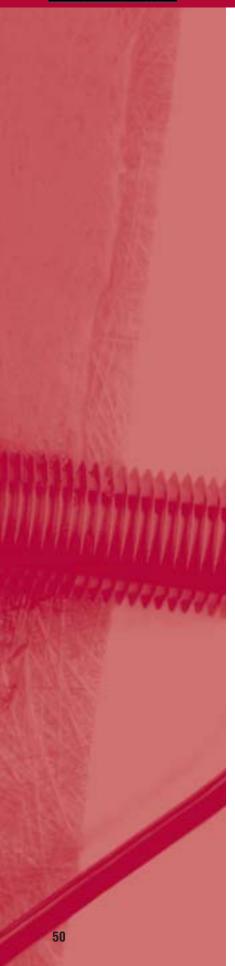


PRODUCT STEWARDSHIP

Ashland encourages its customers and potential users of Ashland products to review their applications of such products from the standpoint of human health and environmental quality. To help ensure that Ashland products are not used in ways for which they are not intended or tested, Ashland personnel will assist customers in dealing with ecological and product safety considerations. Your Ashland sales representative can arrange the proper contacts. Ashland product literature, including Safety Data Sheets, should be consulted prior to use of Ashland products. These may be obtained from your Ashland sales representative, by visiting www.derakane.com, or by sending a request by e-mail to derakane@ashland.com.



DERAKANE"



Subject Index

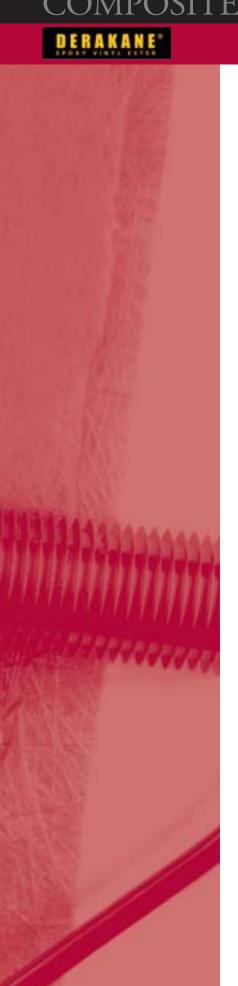
Abrasion resistance	31
Accelerators	4, 7, 9
Aerating resin	47-48
Air inhibition	
Anchors for concrete	31
Antimony compounds	
Antifoam agents	
Applying FRP	
lining	28-30
– to metal	
– to FRP	
- to concrete	
Applying a test patch	
Bulk storage	
Catalysts	
Checking for complete	.(300 111111111013)
cure	30
Conductive linings	
Conversion factors	
Defects	
Disposal	
Environmental information	45
Equipment for resin	4.4
transfer molding	41
Exposure limits	4.4
(personal protection)	
Extending resin shelf life	
FDA/USDA compliance	
Filament winding	
First aid	
Food contact	
Formulating for spray-up .	
Gel times and temperatures	
Glass fibers storage	
Glass types	3, 4, 9
Guide to making a	
laminate	
Initiators	4, 5, 9
Inspecting	
laminates	32-35
Internal mold	
releases	
Inventory rotation	47
Laminate	
construction	22-25
Making a	
laminate	22-25
Making a test laminate	

Masterbatches .	
MSDS	
Material suppliers	;
Mold release ager	nts12
Non-foaming initi	ators6, 9
Paints	
Personnel protect	ion 45
Physical propertie	es of
liquid resins	42
Polyamide epoxy	paints13
Postcuring	27
Product stewards	hip 49
Promoted resin st	torage48
	6, 9
Pultrusion	
Reactivity of resir	ıs 18, 19, 44
Reinforcement for	r
pultrusion	
Reinforcements u	
in FRP	3, 4, 9, 36, 37, 40
Resin shelf life .	47
Resin transfer mo	olding 40, 41
Retarders (gel tim	ne)7, 9
	8, 17, 42, 46
	s42
	ng 27
	45
	ures 47
Styrene, carcinog	enicity
classification .	46
	ves10-11
	41
	20-21
	ion
wax topcoats	
working life of re	sins19



Notes



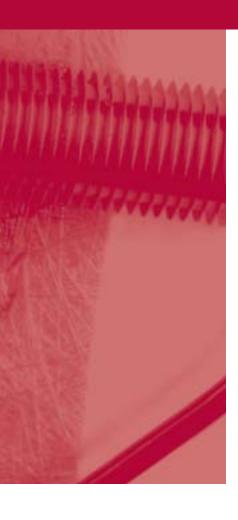


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