



DIXIE CHEMICAL COMPANY, INC.

P.O. Box 130410
Houston, Texas 77219-0410
(713) 863-1947
Fax (713) 863-8316

NADIC□ METHYL ANHYDRIDE

Typical Properties

Physical Appearance	Colorless to very pale yellow liquid
Molecular Weight	178.2
Specific Gravity @ 25C	1.245
Weight, at 15.4°C, lb/gal	10.4
Viscosity, at 25°C, cp	175-225
Refractive index, n	1.503-1.506
Flash Point (Cleveland open cup) °C	135
Boiling Point, C	213 (115 @ 10 mm Hg)
Solubility, at room temperature	Miscible, in all proportions with: Acetone Benzene Naphtha xylene

Formulations

Properties of a cured epoxy resin depend on the starting resin, the curing agent, the accelerator, the ratio of curing agent to resin, the curing cycle, and curing temperature. No one formulation will yield a cured resin having optimum values for all properties. Therefore, it is necessary to determine the desired properties for the intended end use before choosing a formulation. In general, greater cross linking of the resin raises the HDT, hardness, and chemical resistance, but lowers the impact resistance and flexural strength of the cured product.

Calculations of required anhydride

The amount of anhydride in the formulation is generally expressed in parts per hundred (phr) parts of epoxy resin. To calculate the amount of anhydride required use the following equation:

$$\text{Anhydride, phr} = \frac{100}{\text{EEW}} \times \text{AEW} \times \text{A/E} (1)$$

Where:

EEW = epoxide equivalent weight

AEW = anhydride equivalent weight*

A/E = ratio of anhydride equivalent to epoxide equivalent

The epoxide equivalent weight, the color, and the viscosity for some representative epoxy resins is given in Table III.

* For NADIC methyl anhydride, the anhydride equivalent weight is the same as the molecular weight, or 178.2.n

Formulations and Properties of Epoxy Resins Cured with Nadic Methyl Anhydride

Formulation & Cure		
Epoxy resin	ARALDITE ^a 6004 ⁽⁵⁾	D.E.R. ^b 331 ⁽⁶⁾
NADIC methyl anhydride, phr	80	87.5
Accelerator, phr	Ciba 062 2	-
Pot life, 25°C, hr	-	-
Cure time, hr	2 + 2 100 125	2 + 4 90 165
Postcure time, hr	8 149	16 200
PROPERTY OF CURED RESIN		
HDT, °C	140	156
Tensile strength x 10 ³ psi	12	10
Elongation, %	3.5	2.5
Flexural strength x 10 ³ psi	13	14
Flexural modulus x 10 ⁵ psi	3.4	4.40
Compressive Strength x 10 ³ psi	32.5	-
Dielectric constant, at 25°C 60 Hz	3.1	-
1 MHz	3.0	-
Dissipation factor, at 25°C 60 Hz	0.001	-
1 MHz	0.010	-

Effect of Variables on Cured Resin Properties

Effect of varying cure cycles ^(3,7)

Formulation:

	<u>phr</u>
EPON 828 resin	100
NADIC methyl anhydride	90
BDMA	1

Cure cycle:

	<u>hours</u>	<u>temp, °C</u>
Cure A	4	150
Cure B	24	150
Cure C	200	150

Effect of Cure Cycles on HDT, Tensile Strength and Chemical Resistance

Property	Cure A	Cure B	Cure C
HDT, °C	112	128	144
Tensile strength, psi x 10 ³	11.6	10.5	12.1
Elongation, %	3.0	2.7	4.5
Chemical resistance, gain after 24-hour water boil, wt%	0.98	0.67	0.67
gain after 3-hour acetone boil, wt%	3.2	1.9	0.9

The longer cure increases the cross-linking and hence the HDT, tensile strength, elongation and chemical resistance.

Effect of Curing Agent Concentration

Formulation

Property	A	B	C
HDT, °C	129	132	102
Tensile strength, psi x 10 ³	11.0	11.0	11.6
Elongation, %	3.0	2.7	2.8
Flexural strength, psi x 10 ³	18.9	20.7	24.5

Formulation

Property	A	B	C
Flexural modulus x 10 ⁵ psi	5.2	5.7	5.6
Compressive Strength x 10 ³ psi	18.7	19.4	20.5
Dielectric constant, at 25°C, 1 MHz	3.6	3.8	3.7
Dissipation factor, at 25°C, 1 MHz	0.01	0.02	0.02

Maximum HDT is obtained at the 85 phr of hardener, while maximum flexural strength is obtained with the higher concentration of hardener. Elongation is slightly better at the lower concentration.

Effect of varying amount and type of accelerator

Table VII illustrates the effect of varying the type and amount of accelerator. Accelerators are used to initiate reaction and to reduce either the curing time or temperature, or sometimes both. Amines

are sometimes used as accelerators (catalysts) for anhydrides, as are Lewis acids. An example of a Lewis acid which is particularly effective is boron trifluoride monoethylamine complex^g (BF₃ © MEA).*

*Acidic catalyst should not be used where the resin will be in direct contact with metals, such as copper, as this might cause corrosion.

Formulation:

	<u>A</u>	<u>B</u>
D.E.R. 332 resin	100	100
NADIC methyl anhydride	91.5	91.5
BDMA	2	
DB-VIII		5

Cure cycle:

	<u>hours</u>	<u>temp, °C</u>
cure	4	79
postcure	15	149

Effect of Varyin the Accelerator

Formulation

Property	A	B
HDT, °C	89	93
Flexural strength, psi x 10 ³ psi	18.2	5.9
Flexural modulus x 10 ⁵ psi	5.26	4.96
Compressive strength x 10 ³ psi	22.0	22.3
Dielectric constant, at 25°C, 60 Hz	3.45	3.38
1 MHz	3.29	3.16
Dissipation factor, at 25°C, 60 Hz	0.0028	0.0047
1 MHz	0.020	0.027

The greatest difference here is in the flexural strength.

Preparing the Curing Agent/Accelerator Mixture ⁽³⁾

In addition to variables in the formulation and the curing cycle, the method of mixing the ingredients can affect the properties of the cured resin. The sequence of mixing can have a marked effect on the properties of the cured epoxy resin. Direct addition of an amine catalyst to the anhydride is not recommended. *

Epoxy resins, properly cured with NADIC methyl anhydride, are light in color and have HDT's in the region of 135-145°C. To obtain these properties, the following procedure is recommended:

Mixing method ⁽³⁾

- a) Mix anhydride and epoxy resin.
- b) Add the accelerator.
- c) Mix until completely uniform.

* If the accelerator is added to the anhydride before the resin, a darker color may result.

Curing Cycle ⁽³⁾

Curing of an epoxy resin may be carried out in one or more stages depending on the rate of reaction and the exotherm of the mixture. Better control can be achieved by curing at a lower temperature. A slow initial cure followed by a postcure at a higher temperature usually yields good results. Using this approach allows the heat to generate more slowly and thus diffuse as it is generated. This results in better color and fewer stresses in the cured epoxy resin. The postcure anneals the resin thus relieving any stresses, which may be present after the initial cure. The absence of stresses in a cured resin makes reproducibility possible.

Table VIII gives an example of the gel time and peak exotherm for NADIC methyl anhydride and a representative resin.

Gel Tim and Peak Exotherm

Formulation	Phr
ARALDITE 6020	100
NADIC methyl anhydride	82
BDMA	0.5
Anhydride mols/epoxy, A/E	1.0
Temperature at gelation, °C	137
Time, 80°C to gel, minutes	7.5
Peak exotherm temperature, °C	155
Time, 80°C to peak, minutes	11.0

Gel time vs pot life

A distinction should be made between pot life and gelation (gel) time. Pot life ⁽¹⁰⁾ is defined as the time available for use of the epoxy system after the resin, curing agent, and accelerator are mixed. Gelation time is the time required for the mixture to gel and usually takes place at elevated temperatures, as noted.

Applications

Properly cured epoxy resins have high strength, are excellent electrical insulators and are extremely resistant to solvent and chemical attack. These properties make epoxy resins outstanding as the material of engineering choice for such applications as:

- Industrial Coatings
- High voltage insulators

- Encapsulation of a sophisticated electronic circuits
- Sealants
- Adhesives
- Laminates
- Fiberglass reinforced plastics
- Filament winding
- Aircraft applications

Handling & Storage

Store NADIC methyl anhydride between the temperatures of 18°C (85°F) and 40°C (104°F) to avoid freezing, although the material is not affected by freezing. If material does freeze, thaw at a temperature below 40°C and mix well before using. Store in a dry place and keep container tightly closed to prevent absorption of atmospheric moisture or contamination. The presence of moisture could cause free acid to form in the anhydride.

Safety Precautions and First Aid

Consult product material safety data sheet.

Availability