Continuous laminating

Continuous production of flat and corrugated sheets

Application data and cure data

Nouryon is the world's leading producer of organic peroxides for the curing of thermoset resins, coatings and specialty monomers. We're home to the best known brands in the thermoset market, examples include Butanox®, Cadox®, Perkadox® and Trigonox®. We also offer a range of auxiliary products, such as specialty promoters to meet your specific production requirements.

This application guide introduces you to our thermoset product portfolio and helps you find a suitable curing system for your specific application.

Application

The application concerns the continuous production of flat and corrugated sheets. This can be done at ambient temperature using a standard lamination table, or using continuous machines with an oven to cure the sheets at elevated temperature. The sheets can either be curved or straight depending on the end application.

Nouryon curing agents

The ambient temperature curing of flat sheets on a big lamination table is commonly carried out with standard peroxides like Cadox M-50, Butanox M-50, Trigonox 44B, Trigonox 63 and Trigonox 44K.

Reason for our products

- High quality
- Good aftersales and technical service
- Intensive safety research
- Worldwide distribution
- Customized application research: special formulated products for an optimal performance in this application

Continuous sheet production has high output and consequently needs elevated temperature curing and an oven for the final cure. The curing system of choice for this application is depending on the type of oven that is used. For companies working with a long oven a suitable curing system could be Cadox M-50/Butanox M-50 (medium reactivity) Trigonox 44B (AAP fast curing), or Trigonox 44K (AAP and perester) for enhanced final cure. Mixed peroxides such as Trigonox 63 (MEKP and AAP) can be used to get reactivity in-between that of a MEKP and AAP.

For companies working with a short oven the most favourable curing system could be Trigonox 44B or 44K. Trigonox 44K is fast and efficient at elevated temperature curing which is desired in high temperature ovens. It has an activation temperature of 50°C.

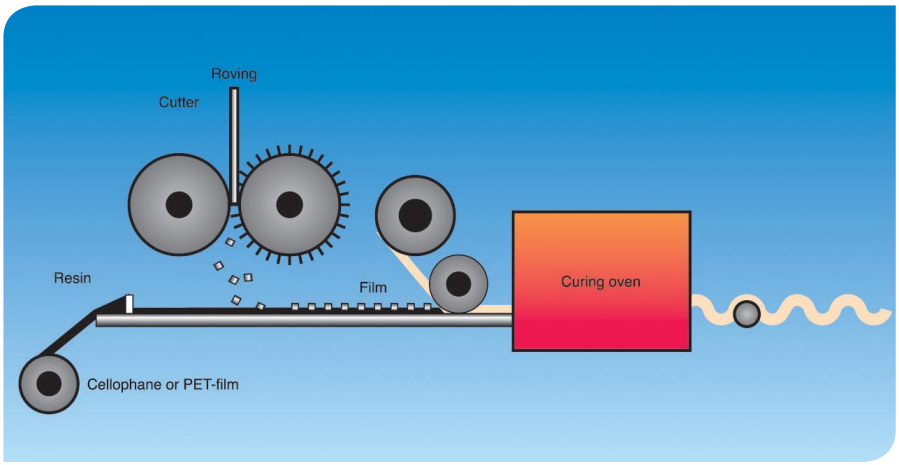
Main products

Corrugated sheets are available in a wide variety of profiles and application areas. High quality products can be gelcoated afterwards with a pure resin layer, which gives an improvement of the weatherability. Flat panels for trucks and building panels are usually pigmented.

The production process

Flat sheets production at ambient temperature on a big lamination table are commonly gelcoated and have a thickness of 0,5 - 3 mm.

Corrugated sheets are manufactured in a continuous machine. These sheets are normally not gelcoated and are in nearly all cases manufactured at elevated temperature in ovens at 80 - 110°C. The oven section is sometimes 60 - 100 meter long (long ovens), or 25 - 35 meter (short ovens).



The elevated temperature curing process is roughly described as follows.

The resin is sprayed on a polymer sheet and the glass roving is chopped on the resin. The top is then covered with a polymer sheet and the flat package is fed to the oven where the sheet is gelled at elevated temperatures (approx.60 - 90°C). After gelation the sheet is shaped and calibrated on thickness and then cured in the second step in the oven at approx.90 -110°C to fix its final shape (corrugated, flat, curved etc.). After cooling down the sheet is cut in the desired length and packed. The sheets do not have a gelcoat finish but a coating can be applied afterwards.

Problems and solutions

Peak exotherm too high

Too high of a peak exotherm can lead to delamination of the end product. White fibers (the glass reinforcement) will then become visible. The mechanical and physical properties of such laminate will be not optimal. Reducing the exotherm can be achieved by lowering the promotion of the resin or changing the peroxide catalyst. Exotherms decrease in the following order, Trigonox 44K>Trigonox 44B>Trigonox 63>Cadox/Butanox M-50>Cadox/Butanox L-50 (lowest).

Cure data

Butanox M-50

Butanox M-50 is a general purpose methyl ethyl ketone peroxide (MEKP) for the curing of unsaturated polyester resins in the presence of a cobalt accelerator at room and elevated temperatures.

The curing system Butanox M-50/cobalt accelerator is particularly suitable for the curing of gelcoat resins, laminating resins, lacquers and castings; moreover, the manufacture of light resistant parts may be possible contrary to the curing system benzoyl peroxide/amine accelerator.

Practical experience throughout many years has proven that by the guaranteed low water content and the absence of polar compounds in Butanox M-50, this peroxide is very suitable in GRP products for e.g. marine applications.

For room temperature application it is necessary to use Butanox M-50 together with a cobalt accelerator (e.g. Accelerator NL-49PN)

Dosing

Depending on working conditions, the following peroxide and accelerator dosage levels are recommended:

Butanox M-50	Accelerator NL-49PN
1 - 4 phr *	0.5 - 3 phr

* (parts per hundred resin)

Cure characteristics

In a high reactive standard orthophthalic resin in combination with Accelerator NL-49PN (= 1% cobalt) the following application characteristics were determined:

Gel times at 20°C

2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	12 minutes
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	7 minutes

Cure of 1 mm pure resin layer at 20°C

The speed of cure is expressed as the time to reach a Persoz hardness of respectively 30, 60 and 120 s.

Persoz	30	60	120	s
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	2.4	4.1	13	h
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	1.7	3.0	9.5	h

Cure of 4 mm laminates at 20°C

4 mm laminates have been made with a 450 g/m² glass chopped strand mat. The glass content in the laminates is 30% (w/w).

The following parameters were determined:

- Time-temperature curve.
- Speed of cure expressed as the time to achieve a Barcol hardness (934-1) of 0-5 and 25-30 respectively.
- Residual styrene content after 24 h at 20°C and a subsequent postcure of 8 h at 80°C.

	GEL TIME (min.)	TIME TO PEAK (min.)	PEAK EXOTHERM (°C)
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	13	36	44
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	8	26	64

	BARCOL		RESIDUAL STYRENE	
	0-5 (h)	25-30 (h)	24 h 20°C (%)	+8 h 80% (%)
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	3	15	6	0.3
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN		1	5	0.1

Pot life at 20°C

Pot lives were determined of a mixture of Butanox M-50 and a non-preaccelerated UP resin at 20°C.

2 phr Butanox M-50	12 h
4 phr Butanox M-50	7 h

Solubility

Butanox M-50 is miscible with phthalates and slightly miscible with water.

Colors

Butanox M-50 is available in the colors blue, yellow-A, red-YM and red-YM 1/6.

Trigonox 44B

Trigonox 44B is an acetyl acetone peroxide formulation for the curing of unsaturated polyester resins in the presence of a cobalt accelerator at room and elevated temperatures.

With the curing system Trigonox 44B/cobalt accelerator a much faster speed of cure may be achieved than with curing systems based on a MEKP plus cobalt accelerator, at room and elevated temperatures. Normally the gel times with Trigonox 44B are comparable to those with Butanox M-50.

Trigonox 44B is particularly suitable in those applications where a fast mold-turnover is required, e.g. for the cold press molding or resin injection molding techniques.

The system Trigonox 44B/cobalt accelerator will give a higher peak exotherm than a standard MEKP/cobalt accelerator system. Due to this fact, is it recommendable to avoid the production of too thick laminates in one operation. At low temperatures a reasonable speed of cure is still obtained when Trigonox 44B is used in combination with large amounts of cobalt accelerator possibly in combination with N,N Dimethylaniline as promotor.

Dosing

Depending on working conditions, the following peroxide and accelerator dosage levels are recommended:

Trigonox 44B

1 - 2 phr *

Accelerator NL-49PN

0.5 - 3 phr

* (parts per hundred resin)

In a high reactive standard orthophthalic resin in combination with Accelerator NL-49PN (= 1% cobalt) the following application characteristics were determined:

Gel times at 20°C

2 phr Trigonox 44B + 0.5 phr Acc. NL-49PN	15 minutes
2 phr Butanox M-50 + 0.5 phr Acc. NL-49PN	12 minutes
2 phr Trigonox 44B + 1.0 phr Acc. NL-49PN	8 minutes
2 phr Butanox M-50 + 1.0 phr Acc. NL-49PN	7 minutes

Cure of 1 mm pure resin layer at 20°C

The speed of cure is expressed as the time to reach a Persoz hardness of respectively 30, 60 and 120 s.

Persoz	30	60	120	s
2 phr Trigonox 44B + 0.5 phr Accelerator NL-49PN	<1	1.5	5	h
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	2.4	4.1	13	h
2 phr Trigonox 44B + 1.0 phr Accelerator NL-49PN	<<1	1	4	h
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	1.7	3	10	h

Cure of 4 mm laminates at 20°C

4 mm laminates have been made with a 450 g/m² glass chopped strand mat. The glass content in the laminates is 30% (w/w).

The following parameters were determined:

- Time-temperature curve.
- Speed of cure expressed as the time to achieve a Barcol hardness (934-1) of 25-30.
- Residual styrene content after 24 h at 20°C and a subsequent postcure of 8 h at 80°C.

	GEL TIME (min.)	TIME TO PEAK (min.)	PEAK EXOTHERM (°C)
2 phr Trigonox 44B + 0.5 phr Accelerator NL-49PN	15	28	67
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	13	36	44
2 phr Trigonox 44B + 1.0 phr Accelerator NL-49PN	8	18	97
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	8	26	64

	BARCOL	RESIDUAL STYRENE	
	25-30 (h)	24 h 20°C (%)	+8 h 80% (%)
2 phr Trigonox 44B + 0.5 phr Accelerator NL-49PN	<1	4.4	0.1
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	15	6	0.3
2 phr Trigonox 44B + 1.0 phr Accelerator NL-49PN	<<1	0.9	0.2
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	1	5	0.1

Pot life at 20°C

Pot lives were determined of a mixture of Trigonox 44B and a non-preaccelerated UP resin at 20°C.

2 phr Trigonox 44B	20 h
4 phr Trigonox 44B	11 h

Trigonox 63

Trigonox 63 is an organic peroxide mixture with methyl ethyl ketone peroxide and acetylacetone peroxide for the curing unsaturated polyester resins in the presence of a cobalt accelerator at room and elevated temperatures.

With the curing system Trigonox 63/cobalt accelerator a faster speed of cure can be obtained than with Butanox M-50; however the high cure rates achieved with Trigonox 44B are not attainable. The gel times with Trigonox 63 are in general similar to those with Butanox M-50.

The curing system Trigonox 63/cobalt accelerator is particularly suitable for the curing of laminating resins, and for applications where a shorter demoulding time is required than obtained with Butanox M-50. Moreover, the manufacture of light resistant parts may be possible contrary to the curing system benzoyl peroxide/amine accelerator.

For room temperature application it is necessary to use Trigonox 63 together with a cobalt accelerator (e.g. Accelerator NL-49PN).

Dosing

Depending on working conditions, the following peroxide and accelerator dosage levels are recommended:

Trigonox 63	Accelerator NL-49PN
1 - 4 phr *	0.5 - 3 phr

* (parts per hundred resin)

Cure characteristics

In a high reactive standard orthophthalic resin in combination with Accelerator NL-49P (= 1% cobalt) the following application characteristics were determined:

Gel times at 20°C

2 phr Trigonox 63 + 0.5 phr Accelerator NL-49PN 15 minutes
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN 12 minutes

2 phr Trigonox 63 + 1.0 phr Accelerator NL-49PN 9 minutes
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN 7 minutes

Cure of 1 mm pure resin layer at 20°C

The speed of cure is expressed as the time to reach a Persoz hardness of respectively 30, 60 and 120 s.

Persoz	30	60	120	s
2 phr Trigonox 63 + 0.5 phr Accelerator NL-49PN	1.4	2.5	8	h
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	2.4	4.1	13	h
2 phr Trigonox 63 + 1.0 phr Accelerator NL-49PN	0.8	1.6	5.5	h
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	1.7	3.0	9.5	h

Cure of 4 mm laminates at 20°C

4 mm Laminates have been made with a 450 g/m2 glass chopped strand mat. The glass content in the laminates is 30% (w/w).

The following parameters were determined:

- Time - temperature curve.
- Speed of cure expressed as the time to achieve a Barcol hardness (934-1) of 0-5 and 25-30 respectively.
- Residual styrene content after 24 h at 20°C and a subsequent postcure of 8 h at 80°C.

	GEL TIME (min.)	TIME TO PEAK (min.)	PEAK EXOTHERM (°C)
2 phr Trigonox 63 + 0.5 phr Accelerator NL-49PN	16	30	60
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	13	36	44
2 phr Trigonox 63 + 1.0 phr Accelerator NL-49PN	9	20	78
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	8	26	64

	BARCOL		RESIDUAL STYRENE	
	0.5-25 (h)	25-30 (h)	24 h 20°C (%)	+8 h 80% (%)
2 phr Trigonox 63 + 0.5 phr Accelerator NL-49PN	<0.5	2	5	0.3
2 phr Butanox M-50 + 0.5 phr Accelerator NL-49PN	3	15	6	0.3
2 phr Trigonox 63 + 1.0 phr Accelerator NL-49PN		<1	4	0.1
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN		1	5	0.1

Pot life at 20°C

Pot lives were determined of a mixture of Trigonox 63 and a non-preaccelerated UP resin at 20°C.

2 phr Trigonox 63	11 h
4 phr Trigonox 63	6 h

Butanox LPT-IN

Butanox LPT-IN is a methyl ethyl ketone peroxide (MEKP) for the curing of unsaturated polyester resins in the presence of a cobalt accelerator at room and elevated temperatures.

Butanox LPT-IN gives in comparison with most other ketone peroxides a significantly longer gel time and is therefore particularly suitable for those applications where a long gel time or production time is required, for instance in the production of large parts and in filament winding.

Also in areas with high ambient temperatures Butanox LPT-IN is of particular interest.

Butanox LPT-IN is especially recommended for the cure of vinyl ester resins. This MEKP formulation gives less "foaming" than standard MEKP's.

Practical experience throughout many years has proven that by the guaranteed low water content and the absence of polar compounds, Butanox LPT-IN is very suitable in GRP products for e.g. marine applications.

The low hydrogen peroxide content of Butanox LPT-IN makes this peroxide very suitable for the cure of those gelcoats, which tend to microporosity caused by the decomposition of the hydrogen peroxide. For room temperature application it is necessary to use Butanox LPT-IN together with a cobalt accelerator (e.g. Accelerator NL-49PN).

Dosing

Depending on working conditions, the following peroxide and accelerator dosage levels are recommended:

Butanox LPT-IN	Accelerator NL-49PN	Inhibitor NLC-10
1 - 4 phr *	0.5 - 3 phr	0 - 0.2 phr

* (parts per hundred resin)

Cure characteristics at ambient temperatures

In a high reactive standard orthophthalic resin in combination with Accelerator NL-49PN (= 1% cobalt) the following application characteristics were determined:

Gel times at 20°C

2 phr Butanox LPT-IN + 1.0 phr Acc. NL-49PN	20 minutes
2 phr Butanox M-50 + 1.0 phr Acc. NL-49PN	7 minutes

Cure of 4 mm laminates at 20°C

4 mm laminates have been made with a 450 g/m^2 glass chopped strand mat. The glass content in the laminates is 30% (w/w).

The following parameters were determined:

- Time-temperature curve.
- Speed of cure expressed as the time to achieve a Barcol hardness (934-1) of 0-5 and 25-30 respectively.
- Residual styrene content after 24 h at 20°C and a subsequent postcure of 8 h at 80°C.

	GEL TIME (min.)	TIME TO PEAK (min.)	PEAK EXOTHERM (°C)
2 phr Butanox LPT-IN + 1.0 phr Accelerator NL-49PN	24	54	41
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN	8	26	64

	BARCOL		RESIDUAL STYRENE	
	0.5-25 (h)	25-30 (h)	24 h 20°C (%)	+8 h 80% (%)
2 phr Butanox LPT-IN + 1.0 phr Accelerator NL-49PN	3	13	6	0.1
2 phr Butanox M-50 + 1.0 phr Accelerator NL-49PN		1	5	<0.1

Cure characteristics at elevated temperatures

The fact that processing times of several hours can be achieved with low cobalt dosages and small amounts of an inhibitor makes Butanox LPT-IN very suitable for use in e.g. filament winding techniques. Simulating the manufacture of a pipe at 70°C consisting of a laminate of 4 mm with a glass content of 30% gave the following results:

Butanox LPT-IN	Accelerator NL-49PN	Inhibitor NLC-10
1 - 5 phr*	0.3 phr	0.2 phr

*(parts per hundred resin)

Gel time at 20°C: 200 minutes

Curing data at 70°C:

Gel time	7 minutes
Time to Peak	17 minutes
Peak exotherm	119°C

Barcol hardness 10 minutes after reaching the peak: 44

Pot life at 20°C

Pot lives were determined of a mixture of Butanox LPT-IN and a non-preaccelerated UP resin at 20°C.

2 phr Butanox LPT-IN	11 h
4 phr Butanox LPT-IN	6 h

Accelerator NL-49PN

The curing of unsaturated polyester resins at ambient temperatures can in general not be performed by an organic peroxide alone. The radical formation, which is necessary to start the polymerization reaction, is at ambient temperatures with most generally applied organic peroxides too slow. To speed up the radical formation in a controllable way organic peroxides must therefore be used in combination with a so called accelerator.

For ketone peroxides like methyl ethyl ketone peroxides, cyclohexanone peroxides and acetylacetone peroxide a cobalt accelerator must be used.

For this purpose the following formulations of cobalt 2 ethylhexanoate also called cobalt octoate are available:

Accelerator NL-49PN	1% cobalt in aliphatic ester
Accelerator NL-51PN	6% cobalt in aliphatic ester
Accelerator NL-53N	10% cobalt in white spirit

The reactivity of the various cobalt accelerators is directly correlated with the cobalt content. The use of a lower concentrated version increases the dosage accuracy. However, when the dosage level of e.g. Accelerator NL-49PN must be higher than approx. 3% to achieve the required cure performance, it is advised to use a higher concentrated cobalt accelerator e.g. 0.5% Accelerator NL-51PN.

The cure characteristics of an unsaturated polyester resin/ketone peroxide mixture can, apart from the choice of the ketone peroxide, very effectively be influenced by the dosage level of the cobalt accelerator. The dosage level of the cobalt accelerator expressed as Accelerator NL-53N (10% cobalt) can for this purpose be varied between e.g. 0.025% up to approximately 0.6% calculated on the UP resin.

When the right peroxide has been chosen and still the required gel time and Cure characteristics cannot be obtained with the cobalt accelerator alone, it is possible to increase the reactivity of the cobalt accelerator by the extra addition of a promotor like N,N-Dimethylaniline or Promotor D (N,N Diethylacetoacetamide).

This adaptation of the accelerator system may be necessary when:
- a very short gel time and/or a very fast cure is required e.g. for resin transfer molding or the production of polymer concrete
- highly inhibited and/or low reactive resins must be cured e.g.
- bisphenol A/fumarate and vinylester resins.

The cure system ketone peroxide/cobalt accelerator can further be characterized by:
- the relatively low color, related to the cobalt dosage, of the cured molding
- a very good UV light resistance of the molded parts
- the long pot life of the cobalt accelerator in the polyester resin
- A possible disadvantage may be that the cure system is more sensitive for moisture, pigments and fillers than the cure system dibenzoyl peroxide/ amine accelerator.

Cobalt accelerators can also be used to increase the reactivity of organic peresters, which are applied for the cure of unsaturated polyester resins at elevated temperatures. Moreover, the use of a cobalt accelerator gives in general a lower residual styrene content in the cured molding. For this application peresters like Trigonox C, Trigonox 21S, Trigonox 42S and the special mixture Trigonox 93 can be used.

Dosage

Depending on working conditions the following accelerator dosage level is recommended:

Accelerator NL-49PN

0.25 - 3.0 phr *

* (parts per hundred resin)

Cure characteristics

In the following cure experiments the performance of cobalt 2 ethylhexanoate as accelerator will be demonstrated.

Gel times at 20°C

in a standard orthophthalic resin with various ketone peroxides

ACCELERATOR NL-49PN (PHR)	0.25	0.5	1
2 phr Butanox M 60	22	12	7
2 phr Butanox LPT-IN	65	31	20
2 phr Cyclonox LE 50	20	11	6
2 phr Trigonox 44B	24	14	8

in various resins with Dimethylaniline, 100% as promotor

standard orthophthalic resin

2 phr Butanox M 60 + 1 phr Acc. NL-49PN	7 min.
2 phr Butanox M 60 + 1 phr Acc. NL-49PN + 0.05 phr Dimethylaniline	4 min.
2 phr Butanox M 60 + 1 phr Acc. NL-49PN + 0.10 phr Dimethylaniline	2 min.
2 phr Trigonox 44B + 1 phr Acc. NL-49PN	8 min.
2 phr Trigonox 44B + 1 phr Acc. NL-49PN + 0.05 phr Dimethylaniline	5 min.
2 phr Trigonox 44B + 1 phr Acc. NL-49PN + 0.10 phr Dimethylaniline	3 min.

bisphenol A/fumarate resin

2 phr Butanox LPT-IN + 3 phr Acc. NL-49PN	145 min.
2 phr Butanox LPT-IN + 3 phr Acc. NL-49PN + 0.05 phr Dimethylaniline	65 min.
2 phr Butanox LPT-IN + 3 phr Acc. NL-49PN + 0.10 phr Dimethylaniline	34 min.

bisphenol A/vinylester resin

2 phr Butanox LPT-IN + 3 phr Acc. NL-49PN	32 min.
2 phr Butanox LPT-IN + 3 phr Acc. NL-49PN + 0.05 phr Dimethylaniline	22 min.
2 phr Butanox LPT-IN + 3 phr Acc. NL-49PN + 0.10 phr Dimethylaniline	16 min.

Time-temperature curves at elevated temperatures (70°C and 90°C)

	CURE TEMP (°C)	GEL TIME (min.)	TIME TO PEAK (min.)	PEAK EXOTHERM (°C)
1 phr Trigonox 21S	70	916	233	
1 phr Trigonox 21S + 1 phr Accelerator NL-49PN	70	35	214	
1 phr Trigonox 21S	90	16	258	
1 phr Trigonox 21S + 1 phr Accelerator NL-49PN	90	0.3	1.5	240
1 phr Trigonox C	90	925	236	
1 phr Trigonox C + 1 phr Accelerator NL-49PN	9	26	258	

Pot life at 20°C

The pot life has been determined of Accelerator NL-49PN in a standard orthophthalic polyester resin at 20°C.

1 phr Accelerator NL-49PN	6 months

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Additional information

Product Data Sheets (PDS) and Safety Data Sheets (SDS) for our polymerization initiators are available at www.nouryon.com

Nouryon

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