

Pultrusion

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[®], Perkadox[®] and Trigonox[®]. We also have a whole range of specialty auxiliary products, such as cobalt free accelerators to meet specific production requirements. We also have a whole range of auxiliary products, such as accelerators and promoters, to meet your specific production requirements.

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

Application

This application guide gives a guidance on curing systems for the pultrusion process. Curing systems for both the European market as well as for the area's where the ambient temperatures can be much higher, like in the Middle East for example. Especially in the hot countries it is difficult and costly to use Perkadox 16 which is a commonly used peroxide for pultrusion. The reason is that Perkadox 16 requires cooled transportation. In this application guide we therefore also mention curing systems which can be used in the hot countries.

Nouryon curing agents

For climate conditions like in Western Europe we recommend the use of Perkadox 16, Trigonox C and Trigonox 29-C50. Normally these are used in combination to make full use of the specific reactivity of the various peroxides. Also a combination with Trigonox 21S is possible.

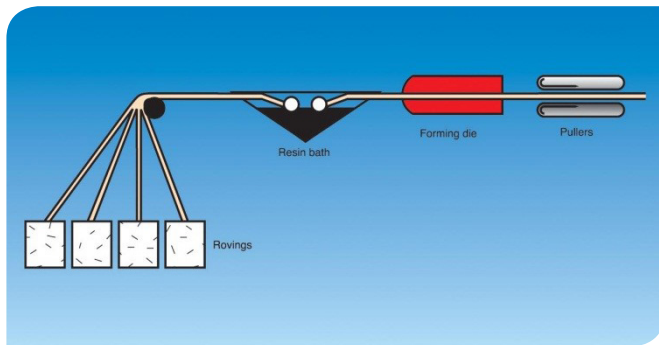
For the high temperature regions we recommend Trigonox C and Trigonox 29-C50. But also peroxides like Trigonox HMa and Perkadox CH-50X /Perkadox GB-50X can be used as kicker peroxides.

Main products

The main products which are manufactured by pultrusion process are profiles and rods which find their use in steps, grids, handles, windows etc.

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



Process equipment

The pultrusion process is a continuous production method for highly filled polyester profiles. Basically in this process: reinforcement material, impregnated with a resin is pulled through a heated die for shaping and curing. Composition: 25-50 parts high reactive UP resin and 75-50 parts glass reinforcement and optional additives like filler, pigments and flame retardants.

The glass reinforcement is in general roving or spin roving, but can also be glass mat, glass cloth or a combination. The impregnation of the reinforcement material is done by pulling the glass through a resin bath (outside) or by injecting the resin in the glass in the die. The dies are generally made of chrome plated steel with a length of about 50-150 cm.

The gelation and curing takes place in the die at a temperature of e.g. 90-170°C, with a production speed of 0.25-2 m/min. The choice of the curing system is dependent on parameters like profile shape, thickness, die-temperature, profile composition and resin reactivity.

The curing system is chosen in a way that the pot-life of the premix is long enough. This means that a balance should be found between reactivity at ambient temperature and reactivity in the die at higher temperatures. Perkadox 16 can provide such a balance and is an ideal kicker peroxide in the pultrusion process. A kicker peroxide initiates the curing, provides fast gel times and takes care of the initial temperature increase. Trigonox 21S is also known as a kicker peroxide. However these kicker peroxides are not very efficient and do not finish off the reaction. For this reason a second peroxide like Trigonox C for example is added to the formulation to complete the cure.

Consequently a curing system for pultrusion always contains a kicker peroxide and an efficient peroxide, Perkadox 16 and Trigonox C for example is commonly used. But other combinations are possible as well.

For areas with a high ambient temperature the choice of peroxides could be different because of the requirements for cooled transportation peroxides like Perkadox 16 and Trigonox 21S.

For the initial cure (kicker) in order to get a fast gel time in the die peroxide like Trigonox HMa or Perkadox CH-50X can be used. However, these peroxides are not as reactive like Perkadox 16. Trigonox HMa is more reactive as kicker peroxide than Perkadox CH-50, but a disadvantage is the short pot-life in the pre-mix. Still a combination with an efficient peroxide like Trigonox C is preferred to complete the reaction.

Reactivity figures

Curing systems for regions with moderate ambient temperatures, (the results below serve only as a guideline and are for comparison purposes only. The results are measured with the SPI laboratory test).

Perkadox 16

Time-temperature curves

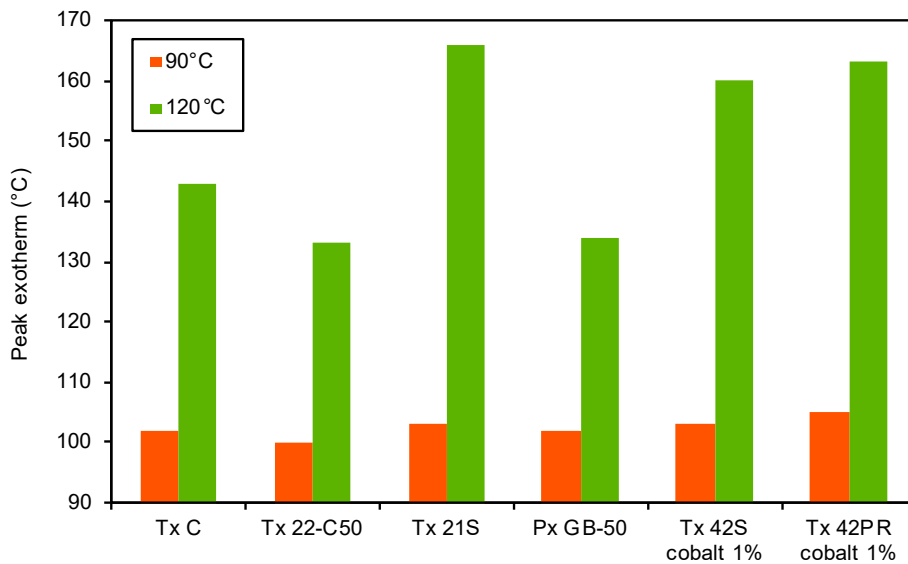
Time-temperature curves have been determined at 60°C, 100°C and 140°C to demonstrate the use of Perkadox 16 as kicker in pultrusion applications. From the curves the time to peak was calculated as an indication for the cure speed.

TIME TO PEAK

	AT 60°C (HOURS)	AT 100°C (MIN.)	AT 140°C (MIN.)
1 phr Trigonox 29-C50	5.5	7.7	2.8
1 phr Trigonox 29-C50 + 1 phr Perkadox 16	0.25	2.4	1.4
1 phr Trigonox C	24	13.5	3.5
1 phr Trigonox C + 1 phr Perkadox 16	0.25	2.1	1.4

Graph 2. The peak exotherm of the time temperature curve in 5 mm laminates cured with 0.5 phr Perkadox 16 + different finishing catalysts at 90°C and 120°C

Tx = Trigonox; Px = Perkadox



Reactivity figures

Curing systems for regions with high ambient temperatures (the results below serve only as a guideline and are for comparison purposes only. The results are measured with the SPI laboratory test).

TIME TO PEAK EXOTHERM

	AT 100°C (MIN.)	AT 140°C (MIN.)
1 phr Trigonox 22-C50	7.7	2.8
1 phr Trigonox 22-C50 + 1 phr Trigonox HMa	3.7	1.9
1 phr Trigonox C	13.5	3.5
1 phr Trigonox C + 1 phr Trigonox HMa	3.8	2.0

Perkadox CH-50X

Time-temperature curves

Time-temperature curves have been determined at 60°C, 100°C and 140°C to demonstrate the use of Perkadox CH-50X/Perkadox GB-50X as kicker in pultrusion applications. From the curves the time to peak was calculated as an indication for the cure speed.

	TIME TO PEAK		
	AT 60°C (HOURS.)	AT 100°C (MIN.)	AT 140°C (MIN.)
1 phr Trigonox C	24	13.5	3.5
1 phr Trigonox C + 0.5 phr Perkadox CH-50X/GB-50X	12.6	9.0	2.1

A summary containing the standard reactivity data of the most used products in this application can be found on the next pages.

Cure data

Perkadox 16

Perkadox 16 is a solid peroxydicarbonate which is used for the curing of unsaturated polyester resins and methacrylic resins mainly in the temperature range of 60°C and higher.

Perkadox 16 is not suitable for the production of clear - castings or - coatings, due to a slight haze in the end product. In this case, Perkadox 16S can be advised.

Perkadox 16 shows a high reactivity at elevated temperatures, which is demonstrated by its low activation temperature, in combination with a relatively long pot life at ambient temperatures.

Perkadox 16 is mostly used in combination with a low reactive peroxide to ensure a good final cure. Combinations of Perkadox 16 with e.g. Trigonox C, Trigonox 29-C50, Trigonox 21S or Perkadox CH-50X/GB-50X can therefore successfully be used for those applications where a long gel time or production time is required at room temperature in combination with a fast cure at elevated temperatures of e.g. 60-140°C. Applications area can be: pultrusion, filament winding, manufacturing of engineered stone.

Dosing

Depending on working conditions, the following application characteristics were determined:

**Perkadox 16
as such
1 - 2 phr ***

**Perkadox 16
as kicker
0.5 - 1 phr**

*(parts per hundred resin)

Cure characteristics

In a high reactive standard orthophthalic polyester resin the following application characteristics were determined:

Activation temperature

1 phr Perkadox 16 40°C

Gel times at 100°C

1 phr Trigonox C 10 min.

1 phr Trigonox C + 0.5 phr Perkadox 16 2 min.

1 phr Trigonox C + 1.0 phr Perkadox 16 1.2 min.

Pot life at 20°C

1 phr Perkadox 16 2 days

1 phr Trigonox C 56 days

1 phr Trigonox C + 0.5 phr Perkadox 16 8 days

1 phr Trigonox C + 1.0 phr Perkadox 16 2 days

Time-temperature curves at elevated temperatures

Time-temperature curves have been determined at 60°C, 100°C and 140°C to demonstrate the use of Perkadox 16 as kicker in pultrusion applications. From the curves the time to peak was calculated as an indication for the cure speed.

	TIME TO PEAK AT		
	60°C (HOURS)	100°C (MIN.)	140°C (MIN.)
1 phr Trigonox 29-C50	5.5	7.7	2.8
1 phr Trigonox 29-C50 + 1 phr Perkadox 16	0.25	2.	1.4
1 phr Trigonox C	24	13.5	3.5
1 phr Trigonox C + 1 phr Perkadox 16	0.25	2.1	1.4

Trigonox C

Trigonox C, tert-butyl peroxybenzoate, is an aromatic perester, which is used for the curing of unsaturated polyester resins at elevated temperatures.

Trigonox C is preferred for the curing of UP resin based Hot Press Molding formulations (SMC, BMC) in the temperature range of 120-170°C.

Trigonox C can also be used in combination with high reactive peroxides like Perkadox 16 or Trigonox HMa as kicker in formulations for pultrusion in the temperature range of 100-150°C.

In combination with a cobalt accelerator (e.g. Accelerator NL-53N, 10% cobalt), Trigonox C is also applicable for the cure of UP resins in the temperature range of 70°C and higher. Application area can be: air drying lacquers, diplacquers, filament winding, etc.

Dosing

Depending on application and working conditions, the following peroxide and when applicable cobalt accelerator dosage levels are recommended:

Trigonox C

1 - 2 phr *

Accelerator NL-53N

0.1 - 0.6 phr

*(parts per hundred resin)

Cure characteristics in pure UP resin

In a high reactive standard orthophthalic UP resin the following application characteristics were determined:

Activation temperature

1 phr Trigonox C	80°C
1 phr Trigonox C + 0.1 phr Acc. NL-53N	70°C

Pot life at 20°C

1 phr Trigonox C	56 days
1 phr Trigonox C + 0.1 phr Acc. NL-53N	17 days

For the application at elevated temperatures the following data determined in 25 g pure UP resin can be used as an indication of the reactivity:

Time-Temperature curves at 90°C

	GEL TIME MIN.	TIME TO PEAK MIN.	PEAK EXOTHERM (°C)
1 phr Trigonox C	9	25	236
1 phr Trigonox C + 0.1 phr Accelerator NL-53N	2	6	258

Cure characteristics in Hot Press Moulding

In a standard Hot Press Moulding compound based on a high reactive orthophthalic polyester resin with calcium carbonate as filler and magnesium oxide as thickening agent, the following application characteristics were determined:

Shelf life at 30°C

	not pigmented	+ 5 phr iron oxide black
1 phr Trigonox C	47 days	17 days

Platengel time

	Mould temperature	
	120°C	140°C
1 phr Trigonox C	85 sec.	22 sec.



Trigonox 29-C50

Trigonox 29-C50 is a 50% formulation of the perketale 1,1-Di(tert-butyl peroxy)cyclohexane in isododecane which is used for the curing of unsaturated polyester resins at elevated temperatures.

Trigonox 29-C50 is preferred for the curing of UP resin based Hot Press Moulding formulations (SMC, DMC, BMC) in the temperature range of 120-170°C.

Trigonox 29-C50 can also be used in combination with high reactive peroxides like Perkadox 16 or Trigonox HMA as kicker in formulations for pultrusion in the temperature range of 100-150°C.

As Trigonox 29-C50 is a perketale, the stability of this peroxide in an UP resin is hardly influenced by the presence of metal accelerators, pigments or fillers. Trigonox 29-C50 is therefore very suitable for pigmented and non-pigmented Hot Press Moulding formulations where a long shelf life of the compound is required.

Trigonox 29-C50 is restricted applicable for Low Shrink and Low Profile formulations in view of possible surface imperfections.

Dosing

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

Trigonox 29-C50

1.5 - 3 phr *

*(parts per hundred resin)

Cure characteristics in pure UP resin

In a high reactive standard orthophthalic UP resin the following application characteristics were determined:

Activation temperature

2 phr Trigonox 29-C50 70°C

Pot life at 20°C

2 phr Trigonox 29-C50 56 days

Cure characteristics in Hot Press Moulding

In a standard Hot Press Moulding compound based on a high reactive orthophthalic polyester resin with calcium carbonate as filler and magnesium oxide as thickening agent, the following application characteristics were determined:

Shelf life at 30°C

	not pigmented	+ 5 phr iron oxide black
2 phr Trigonox 29-C50	>6 months	4 months
1 phr Trigonox C	47 days	17 days

Platengel time

	Mould temperature	
	120°C	140°C
2 phr Trigonox 29-C50	30 s	10 s
1 phr Trigonox C	85 s	22 s

Minimum Moulding time

Measured in a 4 mm cup shaped moulding at 140°C.

2 phr Trigonox 29-C50	34 s
1 phr Trigonox C	60 s

Perkadox CH-50X

Perkadox CH-50X is a non-caking, fine, granular powder with excellent free flowing properties containing 50% dibenzoylperoxide.

Perkadox CH-50X is used for the curing of unsaturated polyester resins and (meth)acrylic resins at ambient and elevated temperatures. At temperatures up to 80°C, Perkadox CH-50X should be used in combination with an aromatic tertiary amine accelerator, above 80°C the use of an accelerator is not required.

Perkadox CH-50X is easy to handle, easy to disperse and dissolves very quickly in unsaturated polyester resins and (meth)acrylic resins.

The curing system Perkadox CH-50X/amine accelerator shows a very fast cure that is hardly influenced by humidity and fillers. Even at low temperatures a relatively good cure will be obtained. A disadvantage may be the yellow color and poor light resistance of the moulded product.

For ambient temperature curing the following amine accelerators are available to adjust the gel time and speed of cure of the cure system based on Perkadox CH-50X:

- Accelerator NL-65-100 (N,N-Dimethyl p toluidine) for short gel times
- N,N-Dimethylaniline for medium gel times
- Accelerator NL-64-100 (N,N-Diethylaniline) for long gel times

Dosing

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

Perkadox CH-50X

2 - 5 phr *

Amine accelerator

0.05 - 0.5 phr

*(parts per hundred resin)

Cure characteristics

In a high reactive standard orthophthalic polyester resin the following application characteristics were determined.

GEL TIMES AT 20°C

UP resin	100	100	100	100	100	100	100
Perkadox CH-50X	3.0	3.0	3.0	3.0	3.0	3.0	3.0
N,N-Dimethylaniline					0.1	0.4	
Accelerator NL-64-100			0.1	0.5			
Accelerator NL-65-100					0.05	0.1	0.4
Gel time (minutes)	22	6	160	20	20	5	1

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
3 phr Perkadox CH-50X + 0.1 phr N,N-Dimethylaniline		0.5	0.8	2	h
3 phr Perkadox CH-50X + 0.4 phr N,N-Dimethylaniline				<0.5	h
3 phr Perkadox CH-50X + 0.5 phr Accelerator NL-64-100			0.5	1	h
3 phr Perkadox CH-50X + 0.05 phr Accelerator NL-65-100		1	2.5	14	h
3 phr Perkadox CH-50X + 0.1 phr Accelerator NL-65-100				0.5	h

Cure of 4 mm laminates at 20°C

4 mm laminates have been made with 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 24h at 20°C and a subsequent postcure of 8h at 80°C.

	GEL TIME (min.)	TIME TO PEAK (min.)	PEAK EXOTHERM (°C)
3 phr Perkadox CH-50X + 0.1 phr N,N-Dimethylaniline	24	31	99
3 phr Perkadox CH-50X + 0.5 phr Accelerator NL-64-100	21	26	140
3 phr Perkadox CH-50X + 0.05 phr Accelerator NL-65-100	28	35	64

	BARCOL		RESIDUAL STYRENE	
	0-5 (h)	25-30 (h)	24 h 20°C (%)	+8 h 80°C (%)
3 phr Perkadox CH-50X + 0.1 phr N,N-Dimethylaniline		<1	3.2	1.0
3 phr Perkadox CH-50X + 0.5 phr Accelerator NL-64-100		<<1	2.9	2.1
3 phr Perkadox CH-50X + 0.05 phr Accelerator NL-65-100	1	8.5	6.6	0.8

Pot life at 20°C

Pot lives were determined of a mixture of Perkadox CH-50X and a non-preaccelerated resin at 20°C.

3 phr Perkadox CH-50X	21 days
6 phr Perkadox CH-50X	11 days

Contact us

For product inquiry and ordering information, please contact your Nouryon account manager or regional Nouryon sales office.

Americas

US and other countries

Citadel Center
131 S Dearborn St, Suite 1000
Chicago IL 60603-5566
USA
T +1 800 828 7929 (US only)
E polymer.amer@nouryon.com

Mexico

Av. Morelos No. 49
Col. Tecamachalco
Los Reyes La Paz Estado de Mexico
C.P. 56500 Mexico
T +52 55 5858 0700
E polymer.mx@nouryon.com

Brazil

Rodavia Nouryon no. 707
Portão A – Planta C
Bairro São Roque da Chave
13295-000 Itupeva - São Paulo
Brazil
T +55 11 4591 8800
E polymer.sa@nouryon.com

Europe, India, Middle East and Africa

France, Italy, Spain and Portugal

Autovia de Castelldefels, km 4.65
08820 El Prat de Llobregat
Barcelona
Spain
T +34 933 741991
E polymer.es@nouryon.com

India

North Block 801, Empire Tower,
Reliable Cloud City Campus,
Off Thane – Belapur Road
Airoli, Navi Mumbai - 400708
India
T +91(0) 22 68426700
E polymer.emeia@nouryon.com

Middle East

Silicon park, Building A6
Office no 402, 4th floor
Dubai Silicon Oasis
Dubai
United Arab Emirates
T +971 4 2471500
E communications.me@nouryon.com

Russia and CIS

Smolnaya Str., 24D,
Commercial Tower Meridian
125445 Moscow
Russia
T +7 495 766 16 06
E info.moscow@nouryon.com

Other countries

Zutphenseweg 10
7418 AJ Deventer
The Netherlands
E polymer.emeia@nouryon.com

Asia Pacific

Room 2501 & 26F, Building A
Caohejing Center
No. 1520 Gumei Road, Xuhui District
Shanghai 200233
P.R. China
T +86 21 2289 1000
E polymer.apac@nouryon.com

Additional information

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

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Nouryon

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