OXEA
the oxo people

OXFILM 351®

Next Generation Coalescing Agent

Formulating Next Generation Low Odor, Low VOC Latex Paints with OXFILM 351®

OXFILM 351 coalescent was introduced to the coatings market about one year ago. Over the course of the year, OXEA has come to better understand the benefits, formulation requirements, and most suitable applications for OXFILM 351. The purpose of this update is to share these learnings.

OXFILM 351 is a low odor, almost zero VOC coalescing agent. By definition, a non-VOC remains in the dried film where OXFILM 351 acts as a permanent plasticizer yielding a slightly softer film. To compensate, a slightly harder, higher T_g, resin is required to obtain the desired final film hardness. OXFILM 351 cannot be substituted into an existing formulation based on a fugitive coalescent with the expectation of obtaining the same film hardness and properties. Traditional fugitive coalescents such as ester alcohols and glycol ethers have some water solubility and partition between the aqueous and polymer phases. OXFILM 351 is water insoluble and migrates completely into the polymer particle. This helps make OXFILM 351 more efficient than conventional coalescing agents. However, additional time is necessary to allow for migration into the polymer phase. All formulations must be aged overnight before evaluations begin to see the full benefits that OXFILM 351 provides.

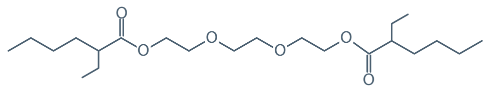
Emission cell testing per ISO 16000-100 has demonstrated that use of OXFILM 351 and careful choice of other additives can yield 28 day results below 100 $\mu g/m^3$!

OXFILM 351 has found the broadest acceptance in higher PVC matte and semi-gloss coatings. However, use in high-gloss low PVC coatings should not be precluded.

In summary, OXFILM 351 is not designed to be a drop-in replacement for traditional ester alcohol or glycol ether coalescents. It is intended for next generation low odor, low VOC coatings. Use of a slightly harder polymer is recommended to compensate for its permanence and plasticizing effect in the dried film. Low odor, extremely low 28 day emission results are achievable with OXFILM 351 when properly formulated.

Technical data

CAS Number 94-28-0
EINECS Number 202-319-2



Physical properties

Formula	$C_{22}H_{42}O_6$
Molecular Weight	402.6 g/mol
Appearance	Clear colourless liquid
Boiling point (°C)	351
Specific Gravity (20°C)	0.967
Odour	Nil
Solubility in Water (mg/L, 20°C)	1.53
Vapour pressure (hPa, 20°C)	< 0.001
Viscosity (mPa*s, 20°C)	16.3

Sales Specifications

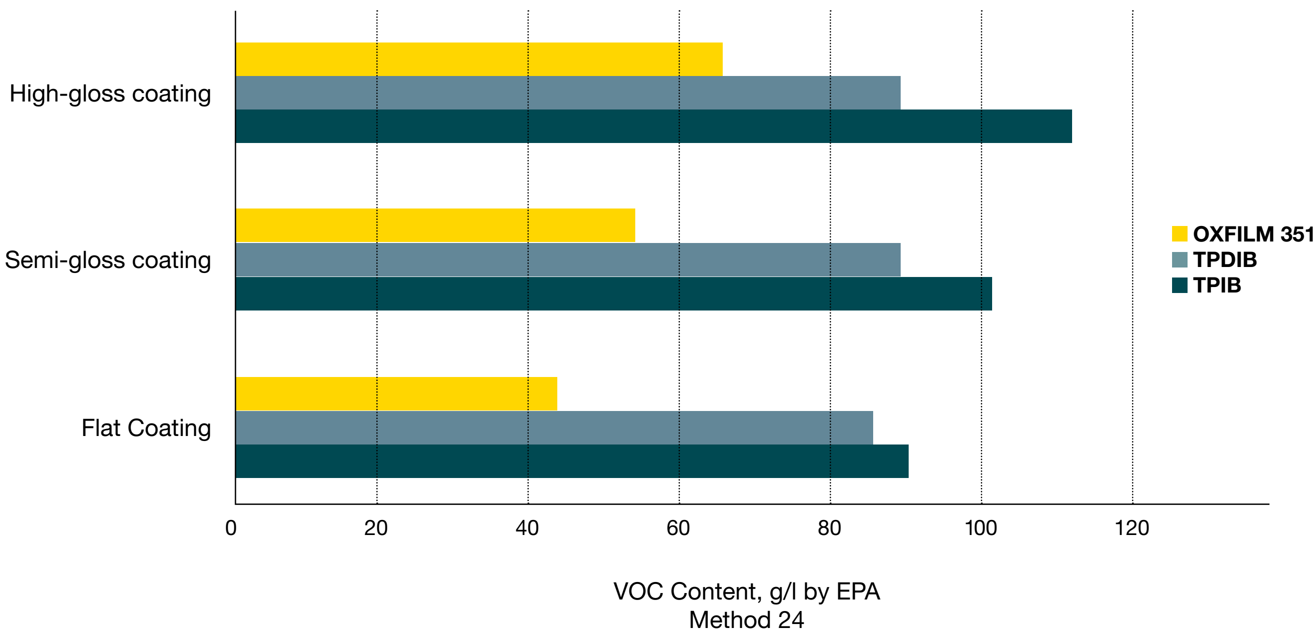
Property	Limit	Unit	Test Method	PQR
Appearance	Clear Liquid		Visual Examination	
OXFILM 351	min. 97	% (a/a)	DIN 51405 (GC)	x
Triethylenglycol-mono-2-ethyl-hexanoate (Monoester)	max. 1.5	% (a/a)	DIN 51405 (GC)	x
Diethylenglycol-bis-2-ethyl-hexanoate	max. 0.5	% (a/a)	DIN 51405 (GC)	x
Acid Value	max. 0.10	mg KOH/g	DIN EN ISO 3682 / ASTM D 1613	x
Peroxide Value	max. 1.5	mäq O/kg	RCH – AL079	x
Ester Value	236 – 279	mg KOH/g	DIN 53401	
Hydroxyl Value	max. 5.0	mg KOH/g	DIN 53240	
Water	max. 0.07	% (w/w)	DIN 51777 Part I	x
BHT Stabilizer	50	mg/kg	DIN 51405 (GC), qual.	x
Platinum/Cobalt Colour (Hazen/APHA Colour)	max. 30		DIN ISO 6271	x
Density (20°C)	0.962 – 0.972	g/cm^3	DIN 51757 Verf. D	
Refractive Index nD25	1.441 – 1.447		DIN 51 423 / ASTM D 1747	

VOC Definition and Status

VOC definition
VOC means Volatile Organic Compound. There are different ways to define and determine if a substance is a VOC.

European Decopaint directive (2004/42/EC)
VOC is any organic chemical with boiling point below 250°C at a standard atmospheric pressure of 101.3 kPa. To be given as grams VOC per liter product.

EPA Method 24
In the U.S., VOC content is determined using EPA Method 24 rather than a statutory definition. When tested, neat OXFILM 351 has a VOC content less than 0.5 %. Thus it will have negligible contribution to a coating's VOC content.

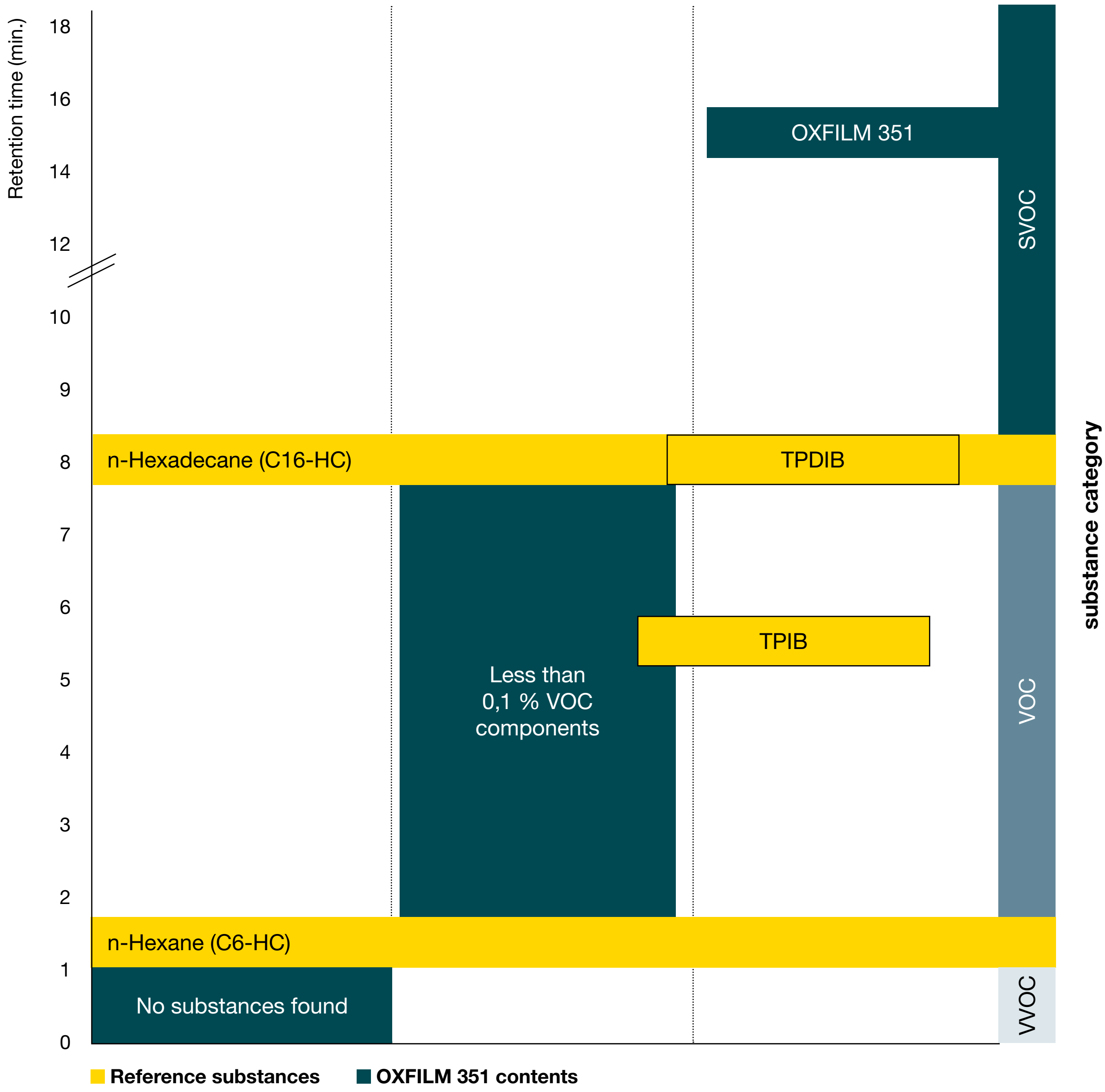


ISO 16000-6, TC 351, French VOC regulations, AgBB, AFSSET and more
Total VOC content is defined in ISO 16000-6 as the sum of volatile organic compounds, which elute from a non-polar or slightly polar gas chromatographic separation column between and including n-Hexane and n-Hexadecane (n-C6 and n-C16). This covers any organic compound with a boiling point between 69°C and 287°C. This is the definition mostly used for VOC emission testing throughout the world.

The GC analysis indicates OXFILM 351 is an non-VOC where TPIB can be considered VOC and TPDIB is right on the border.

No matter which definition or method is used OXFILM 351 is a safe choice for formulating non/low VOC formulations. OXFILM 351 outperforms the currently used coalescents and is well above any threshold and therefore is a long term solution in times where VOC regulations will further tighten.

GC Analysis



Substance classification

VVOC (very volatile organic compounds)	substances, which elute before C6-HC (n-Hexane)
VOC (volatile organic compounds)	substances, which elute between C6-HC (n-Hexane) and C16-HC (n-Hexadecane)
SVOC (semivolatile organic compounds)	substances, which elute after C16-HC (n-Hexadecane)

Minimum Film Forming Temperature (MFFT)

OXFILM 351 was initially tested for its ability to reduce the minimum film forming temperature (MFFT) of five different commercial emulsion polymer types. Results were compared to 2,2,4-trimethyl-1,3-pentanediol monoisobutyrate (TPIB[1]). TPIB has been the workhorse coalescing agent for many years but has a distinctive odor and is more volatile than OXFILM 351. In all cases tested, OXFILM 351 was more efficient than TPIB in reducing MFFT.

Further studies indicated that OXFILM 351 is 10 – 20 % more efficient than TPIB in these uncompounded latexes. In addition, film hardness tests and dry-to-touch times were equal to TPIB despite OXFILM 351's permanence in the dried films.

Sample	Reported MFFT (°C)	Latex Type	MFFT with 1 % OXFILM 351 by wt (°C)	MFFT with 1 % TPIB by wt (°C)
UCAR® [2] Latex 357	12	Vinyl acetate-acrylic	6.7	10.1
UCAR® [2] Latex DA27	18	Styrene acrylic	7.7	14.0
NEOCAR® [2] Latex 2300	15	Vinyl acetate-VeoVa	7.5	12.0
Rovace® [3] 9100	10	Vinyl acetate-ethylene	7.7	8.2
Rhoplex® [3] CL-204	26	All acrylic	21.5	25.4

The performance properties of OXFILM 351 were further evaluated against TPIB and the low VOC coalescent 2,2,4-trimethyl-1,3-pentanediol di-isobutyrate (TPDIB). These evaluations were designed to determine the effectiveness of OXFILM 351 in three latex systems and three different coating types commonly employed in architectural paints:

1. UCAR® [2] 357, a vinyl acrylic latex was formulated into a flat coating (see Table 1, page 11)
2. UCAR® [2] 626, an all acrylic latex was formulated into a semi-gloss coating (see Table 2, page 12)
3. Carboset® [5] CR-760, a styrene acrylic latex was formulated into a high-gloss coating (see Table 3, page 13)

In all cases, OXFILM 351 and the comparison coalescents were added at 10 phr. Propylene glycol was added at 5 phr to facilitate freeze-thaw stability and aid low temperature coalescence. The formulations and coating properties are shown below.

All testing except VOC determination was performed at the University of Southern Mississippi by the Thames-Rawlins Research Group.

Low Temperature Coalescence & Gloss

Low Temperature Coalescence: The coatings were applied to Leneta Form HK penetration charts and immediately placed in a 4.4°C refrigerator. Upon drying overnight, the films were examined for continuity on both the sealed and unsealed portions of the charts according to ASTM D-3793.

OXFILM 351 displayed excellent low temperature coalescence.

Coating	Pass/Fail	Gloss @ 20°	Gloss @ 60°	Gloss @ 85°
Flat coating				
TPIB	Pass	–	2.5	4.7
TPDIB	Pass	–	2.8	5.9
OXFILM 351	Pass	–	2.5	4.5
Semi-gloss coating				
TPIB	Pass	15.5	56.1	–
TPDIB	Pass	13.2	53.0	–
OXFILM 351	Pass	16.6	56.9	–
High-gloss coating				
TPIB	Pass	22.3	62.1	–
TPDIB	Pass	46.0	79.2	–
OXFILM 351	Pass	46.2	80.0	–

Gloss: Each coating was applied to a Leneta 605C chart using a 3 mil Bird applicator. After drying overnight at ambient temperature, gloss was measured using a BYK-Chemie gloss meter.

OXFILM 351 showed a slightly higher gloss in all systems indicating enhanced leveling and flow.

Coating	Gloss @ 20°	Gloss @ 60°	Gloss @ 85°
Flat coating			
TPIB	–	2.0	4.3
TPDIB	–	2.1	8.8
OXFILM 351	–	2.2	10.9
Semi-gloss coating			
TPIB	12.8	51.7	–
TPDIB	10.3	48.0	–
OXFILM 351	19.4	58.5	–
High-gloss coating			
TPIB	51.4	82.0	–
TPDIB	51.2	82.0	–
OXFILM 351	59.2	84.9	–

Scrub & Block Resistance

Scrub Resistance: Coatings were applied to Leneta P-121-10N charts at 7 mils wet film thickness and dried for seven days in a lint-free dry box. Scrub resistance was evaluated on a scrub machine following ASTM D2486 using abrasive scrub media Type SC-2 from the Leneta Company.

In the presence of sufficient binder, OXFILM 351 has a positive effect on scrub resistance.

Coating	Cycles to Failure
Flat coating	
TPIB	417
TPDIB	323
OXFILM 351	325
Semi-gloss coating	
TPIB	664
TPDIB	578
OXFILM 351	648
High-gloss coating	
TPIB	537
TPDIB	523
OXFILM 351	646

Block resistance: Coatings were applied to Leneta 2C opacity charts and allowed to dry for one and seven days in a lint-free dry box. After drying, six 1.5” squares were cut and arranged into three sets with the coated side of the squares facing each other. #8 stoppers were placed an each set of squares and a 1000 g weight placed an each stopper. The weights were maintained on the stoppers for at least 16 hours. The weights were then removed and the squares slowly separated from each other. Coatings were rated for block resistance following ASTM D 4946. OXFILM 351 performed best at high pigment volume concentrations.

Coating	1 Day Block Resistance	7 Day Block Resistance
Flat coating		
TPIB	10	10
TPDIB	9	10
OXFILM 351	9	10
Semi-gloss coating		
TPIB	2	5
TPDIB	3	5
OXFILM 351	2	4
High-gloss coating		
TPIB	0	4
TPDIB	0	2
OXFILM 351	0	0

Stain Resistance & Crosshatch adhesion

Stain Resistance: Coatings were evaluated for their resistance to common household stains using method ASTM D4828. Coatings were applied to Leneta P 121-10N scrub panels and dried at ambient conditions for seven days. Ten household stains were evenly applied to the films and allowed to dry for 3 – 4 hours. A damp sponge was wetted with 15 mL of a 50:50 solution of Fantastik and tap water and inserted into a metal boat weighing 1,000 grams on the scrub machine. The panels were cleaned for 100 continuous cycles, removed from the machine, lightly rinsed and allowed to dry. Each stain was then visually rated for removal.

OXFILM 351 displayed excellent stain resistance on a par with the other coalescents tested.

	Flat Coating			Semi-gloss Coating			High-gloss Coating		
Stain	TPIB	TPDIB	**OXFILM 351**	TPIB	TPDIB	**OXFILM 351**	TPIB	TPDIB	**OXFILM 351**
Pencil	9	9	8	1	3	1	5	1	2
Ball Point Pen	0	0	0	0	0	0	0	0	0
China Marker	2	2	1	8	9	9	8	9	7
Crayon	2	2	1	6	7	7	9	8	9
Lipstick	2	2	2	6	6	6	5	4	4
Washable Marker	7	6	9	7	5	7	2	7	8
Coffee	7	7	7	9	9	9	10	10	9
Ketchup	9	9	9	9	10	9	10	10	10
Mustard	3	3	3	3	3	3	9	8	7
Motor Oil	0	0	5	10	10	10	10	10	10

Crosshatch Adhesion: Film adhesion properties were evaluated using ASTM D 3359 crosshatch method. Coatings were applied at 6 mils wet film thickness onto panels previously coated with a high-gloss alkyd film. The coatings were dried at ambient condition for one and 21 days before being evaluated for their adhesion properties.

OXFILM 351 displayed excellent adhesion to alkyd films and was superior to TPDIB in the high-gloss coating.

Coating	1 Day Adhesion	21 Days Adhesion
Flat Coating		
TPIB	4B	5B
TPDIB	4B	5B
OXFILM 351	3B	4B
Semi-gloss coating		
TPIB	3B	4B
TPDIB	5B	4B
OXFILM 351	4B	5B
High-gloss coating		
TPIB	0B	3B
TPDIB	1B	0B
OXFILM 351	0B	4B

Freeze-Thaw stability & Mudcracking

Freeze-Thaw Stability: Half-pint samples were placed in a freezer for 16 hours at -10°C. Upon removal, the coatings were allowed to thaw at ambient temperature for eight hours. If the sample was stable, the product was evaluated for cleanliness and its Stormer viscosity measured.

All the semi-gloss coatings gelled after one freeze-thaw cycle. The flat and high-gloss coatings gelled after the second freeze-thaw cycle. OXFILM 351 performance was equal to the other coalescents tested.

Mudcracking: Coatings were applied to Leneta Penopac 1B charts at 20 and 30 mils wet film thickness. Dried films were evaluated on the sealed and unsealed portions of the charts.

All samples passed mudcracking except the flat coating with TPDIB which exhibited slight cracking around the film edges at 30 mils wet film thickness.

Appendix 1

Table 1:
Vinyl-Acrylic Flat Coating

Ingredient	Weight Percent
Grind:	
Water	16.48
Natrosol® Plus® 330 (Ashland Aqualon)	0.16
Tamol® 73 IA (Rohm and Haas)	1.24
Potassium carbonate	0.08
Kathon® LX 1.5 (Rohm and Haas)	0.12
Triton® CF-10 (Dow)	0.16
Ti-Pure® R-706 (DuPont)	23.90
Duramite® (Imerys)	9.48
Optiwhite® MX (Burgess Pigment)	9.07
Attagel® 50 (BASF)	0.66
Propylene glycol	0.52
Letdown:	
Water	16.68
UCAR® 357 (Dow)	18.24
Coalescent	1.03
Drewplus® L-475 (Ashland)	0.25
Polyphase® 663 (Troy)	0.41
Acrysol® RM-2020-NPR (Rohm and Haas)	1.24
Acrysol® RM-12W (Rohm and Haas)	0.16
Acrysol® RM-8W (Rohm and Haas)	0.12
Total	100.00
Coating Properties:	
Pigment volume concentration (%)	61.98
Volume solids (%)	33.76
Weight solids (%)	54.13

Appendix 2

Table 2:
All-Acrylic Semi-gloss Coating

Ingredient	Weight Percent
Grind:	
Water	15.81
Tamol® 2001 (Rohm and Haas)	1.10
Ammonium hydroxide	0.23
Foamstar® A-36 (Cognis)	0.05
Surfynol® CT-111 (Air Products)	0.18
Ti-Pure® R-706 (DuPont)	25.26
Polygloss® 90 (KaMin)	5.05
Kathon® LX 1.5 % (Rohm and Haas)	0.14
Propylene glycol	1.15
Letdown:	
UCAR® 626 (Dow)	45.93
Coalescent	2.30
Foamstar® A-36 (Cognis)	0.05
Ammonium hydroxide	0.14
Polyphase® 663 (Troy)	0.92
Acrysol® RM-2020-NPR (Rohm and Haas)	1.38
Acrysol® RM-12W (Rohm and Haas)	0.18
Acrysol® RM-8W (Rohm and Haas)	0.14
Total	100.00
Coating Properties:	
Pigment volume concentration (%)	29.70
Volume solids (%)	38.21
Weight solids (%)	54.64

Appendix 3

Table 3: Styrene-Acrylic High-gloss Coating

Ingredient	Weight Percent
Grind:	
Water	9.87
Tamol® 2001 (Rohm and Haas)	0.77
Ammonium hydroxide	0.25
Foamstar® A-36 (Cognis)	0.05
Surfynol® CT-111 (Air Products)	0.20
Ti-Pure® R-706 (DuPont)	20.73
Kathon® LX 1.5 % (Rohm and Haas)	0.15
Propylene glycol	1.28
Letdown:	
Carboset® CR-760 (Lubrizol)	61.12
Coalescent	2.57
Foamstar® A-36 (Cognis)	0.05
Ammonium hydroxide	0.15
Polyphase® 663 (Troy)	0.99
Acrysol® RM-2020-NPR (Rohm and Haas)	1.48
Acrysol® RM-12W (Rohm and Haas)	0.20
Acrysol® RM-8W (Rohm and Haas)	0.15
Total	100.00
Coating Properties:	
Pigment volume concentration (%)	19.17
Volume solids (%)	34.26
Weight solids (%)	47.60

Glossary of terms

%	percentage
% (w/w)	mass percent
% (a/a)	area-percent (of GC-graph)
°C	degree Celsius
APHA	American Public Health Association
ASTM	American Society for Testing Materials
CAS	Chemical Abstracts Service
cm^3	cubic centimetre
EINECS	European Inventory of Existing Commercial Chemical Substances
g	gram
GC	Gas Chromatography
hPa	hectopascal
kg	kilogramme
L	liter
max.	maximum
mg	milligram
min.	minimum
mm^2	square millimetre
mäq	mol Äquivalent
MFFT	minimum film forming temperature
mol	mole
MPa	megapascal
OXFILM	OXFILM is a trademark of OXEA GmbH
phr	parts per hundred of rubber
PQR	Product Quality Report
SVOC	semivolatile organic compounds
TPIB	TPIB is sold under various trade names including Texanol® by Eastman Chemical Company, Filmer IBT® by Dow Chemical Company, Haltanol by Johann Haltermann Ltd., NX 795 by Perstorp, CS-12 by Chisso Corporation and Kyowanol® M by Kyowa Hakko Chemical Company, Ltd.
TPDIB	TPDIB is sold under various trade names including TXIB® and Optifilm Enhancer® 300 by Eastman Chemical Company, CS-16 by Chisso Corporation and NX800 by Perstorp
VOC	volatile organic compounds
VVOC	very volatile organic compounds
wt %	percentage weight change

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