Flame retardants for engineering plastics

ACTILOX® ACTILOX® B NABALOX® HC



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Co-flame retardants for engineering plastics

The unique features of Nabaltec Boehmites offer a wide range of possibilities in demanding high temperature applications

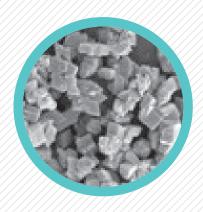
ACTILOX® and **ACTILOX**® **B** products are extremely pure, crystalline boehmites with a very low ATH residue (purity min. 99 %). This guarantees an extraordinarily high temperature stability.

ACTILOX® and **ACTILOX**® **B** can be processed up to 340 °C. These products have a very good chemical resistance, especially a very high acid resistance.

Nomenclature and properties of aluminium hydroxide

ACTILOX® ACTILOX® B	AlooH
Chemical	Aluminium oxide hydroxide
Mineral	Boehmite
Common name	Aluminium monohydrate
Abbreviation	АОН
Loss on ignition	17 %
Density	3.0 g/cm ³
Mohs hardness	3 - 4
рН	7 - 8

Product advantages of ACTILOX® / ACTILOX® B



- high thermal stability, processing up to 340 °C
- synergistic flame retarding effects
- high purity
- low electrolyte content
- · high acid-resistance
- · high thermal conductivity
- low Mohs hardness

- decomposition only slow up to approx. 400 °C
- excellent resin compatibility
- moderate alkalinity
- low water uptake
- scavenging of corrosive by-products
- high thermal capacity (c_p= 1.54 J/gK at 500 K (227 °C)

ACTILOX® / **ACTILOX® B** combine properties like inert chemical behaviour, good flow properties and low abrasion tendency and therefore act as a very effective co-flame retardant. **ACTILOX®** / **ACTILOX® B** functions together with many flame retardants commonly used for engineering plastics.

Especially in combination with phosphorous (PFR) and nitrogen based halogen free flame

retardants (NFR) like diethylphosphinate aluminium salt (DEPAL) or melamine polyphosphate (MPP), **ACTILOX® / ACTILOX® B** can satisfy highest performance requirements.

Contrary to many other mineral flame retardants, Boehmite is advantageous for applications in high shear compounding or injection moulding processes. The amphoteric character supports a scavenging of corrosive by-products, protecting machines,

tools and especially the polymer matrix from corrosion or degradation in melt flow processes.

Product Parameters	Unit	ACTILOX® 200SM	ACTILOX® B60	ACTILOX® B30
AIOOH	% min	99	99	99
Moisture	% max	0.6	0.2	0.2
Spec. surface area (BET)	m²/g	18	5	3
D ₁₀	μm	0.2	0.5	0.9
D ₅₀	μm	0.3	0.7	2.2
D ₉₀	μm	0.6	1.4	4
Bulk density	kg/m³	230	400	650
Oil absorption	ml/100g	36	30	28
Whiteness	% min	97	98	94
Spec. conductivity	μS/cm max	150	80	35

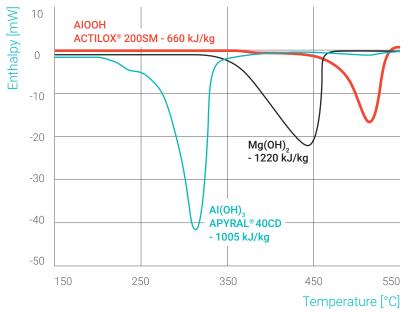


Figure 1: Differential scanning calorimeter data (DSC) of the mineral flame retardants aluminium hydroxides **APYRAL**®, magnesium hydroxide $Mg(OH)_2$ and boehmite (**ACTILOX® 200SM**).

Processing behaviour

The advantageous compounding behavior is based on the special orthorhombic crystal structure of our **ACTILOX**® products.

This cubic like structure imparts very little frictional stress to polymers. Also chemical

induced degradation of polycondensates like polyamides and polyesters is extremely limited with **ACTILOX®** / **ACTILOX®** B. This is because these boehmites combine intrinsically moderate alkalinity with a very low moisture level achieved by Nabaltec's proprietary drying technology.

Figure 2 (below) displays melt volume rates (MVR) for polybutylene therephthalate reinforced with 20 wt.-% glass fibres (PBT GF20) and nylon reinforced with 30 wt.-% glass fibres (PA66 GF30) containing different compositions of flame retardants. The formulations containing **ACTILOX**® consistently show higher MVR compared to the flame retardant reference compounds not containing any boehmite. This MVR increase is not based on any polymer chain scission, as analysis has proven.



SEM image of ACTILOX® B60

Composition of test compounds

	Unit	Refe	Reference PFR		Synergistic combinations with boemite						
		PBT	PA66	PBT	PA66		PBT			PA66	
Polymer	wt%	80	70	60	50	60	60	60	50	50	50
GF	wt%	20	30	20	30	20	20	20	30	30	30
PFR	wt%	-	-	20	20	15	12	10	15	12	10
ACTILOX® 200SM	wt%	_	_	-	_	5	8	10	5	8	10

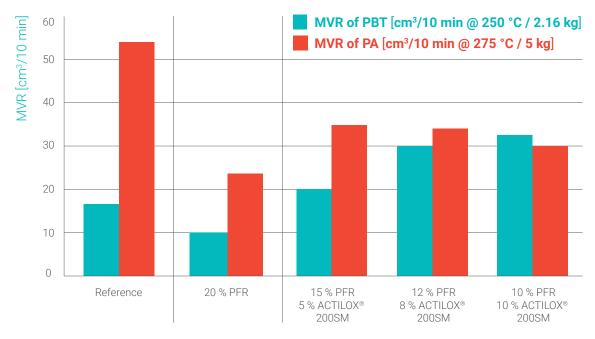


Figure 2: Melt volume rate (MVR) of PBT GF20- and PA66 GF30-compounds containing different amounts of PFR and boehmite **ACTILOX® 200M**. PFR = $Exolit^{TM}$ OP 1200; $Exolit^{TM}$ OP 1312.



Flame retardancy performance

ACTILOX® / **ACTILOX® B** reveals the optimal synergistic performance in combination with commercially available halogen free flame retardants (HFFR) on a phosphorous (PFR) and nitrogen (NFR) base. A required UL 94 classification can be achieved in various formulations.

The usage of **ACTILOX® ACTILOX® B** in such systems sets an optimal balance between heat release, char formation and generation of inert gas-

es to extinguish flames most effectively. Its ability to reduce smoke production is one further benefit of **ACTILOX**® **/ ACTILOX**® **B**.

The diagram below (figure 3) shows the limiting oxygen index (LOI) as an indication for ignitability for the two compound series based on PBT GF20 and PA66 GF30 introduced before. Additionally to **ACTILOX® 200SM**, synergistic combinations were made using **ACTILOX® B60**.



Figure 3: LOI values of PBT and PA66-compounds filled with different amounts of PFR in combination with boehmite (**ACTILOX® 200SM** or **ACTILOX® B60**).

The following tables display the corresponding UL94-V test results determined on 1.6 mm thick specimen using **ACTILOX® 200SM** as synergist (specimen have been prepared by injection moulding).

PA 66 GF30 PFR = Exolit™ OP 1312				
Ratio PFR / ACTILOX® 200SM	UL 94-V @ 1.6 mm			
20 / 0	V0			
15 / 5	V0			
12 / 8	V0			
10 / 10	V0			

PBT GF20 PFR = Exolit™ OP 1200					
Ratio PFR / ACTILOX® 200SM	UL 94-V @ 1.6 mm				
20 / 0	V0				
15 / 5	V0				
12 / 8	V0				
10 / 10	V2				

The glow wire test procedure simulates misuse, failure or malfunction of an end product. The results of glow wire ignition temperature (GWIT) of **ACTILOX®** / **ACTILOX®** B containing specimen produced by injection molding indicate very good performance. This is especially important for applications in connectors and switches.

The electrical insulation performance of such parts can be improved by the ability of **ACTILOX**®

/ **ACTILOX® B** to neutralize acidic by-products released by flame retardants (FR).

This effect is demonstrated by Comparative Tracking Index (CTI) measurements. CTI and Glow Wire Ignition Temperature (GWIT) for the different PBT GF20 formulations are both plotted in the following diagram (figure 4). Especially CTI can be increased when using **ACTILOX® 200SM** in combination with PFR.

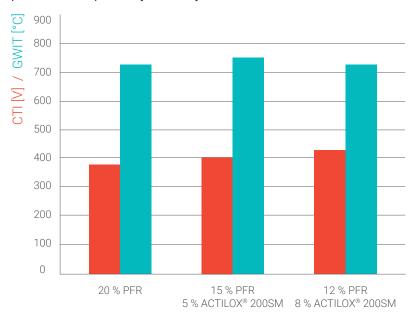


Figure 4: GWIT and CTI of PBT GF20-compounds filled with Exolit[™] OP 1200 in combination with **ACTILOX® 200SM**.

Mechanical properties

Surface treated grades of ACTILOX® / ACTILOX® B

Composition of test compounds

	Unit	Refe- rence	PFR	Synergistic combinations
PBT	wt%	80	60	60
GF	wt%	20	20	20
PFR	wt%	_	20	10
ACTILOX®	wt%	-	-	10

To achieve better homogenization in compounding processes and to improve mechanical compound performance **ACTILOX®** / **ACTILOX® B** can be surface treated. Additionally to above mentioned benefits, water uptake can be reduced when the surface of the filler is hydrophobized by organic coating.

Our know-how in surface treated **ACTILOX**[®] / **ACTILOX**[®] **B** grades help us to specially design

products to meet highest customer needs. Their different surface functionalizations range from highly hydrophobic (ACTILOX® HS1) via amino-group functionalized (ACTILOX® AS1) to vinylor epoxy-group functionalized (ACTILOX® VS1 or ACTILOX® ES1).

In the diagram below (figure 5) the effect of aminosilane coating in PBT GF20 is demonstrated. Impact strength of the flame retardant combinations based on PFR with ACTILOX® 200SM and ACTILOX® B60 and the aminosilane treated products ACTILOX® 200 AS1 and ACTILOX® B60 AS1 is shown.

As the chart shows, the surface treated products give significantly improved impact properties.

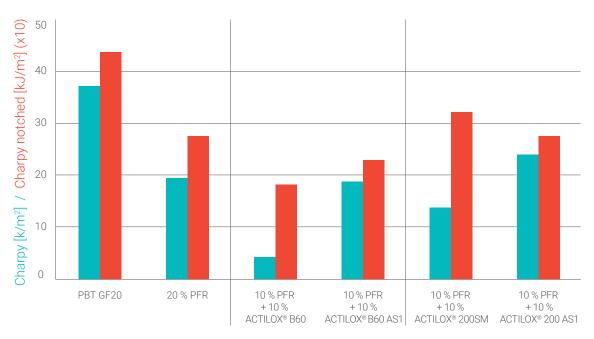


Figure 5: Effect on impact strength of aminosilane treated products **ACTILOX® B60 AS1** and **ACTILOX® 200 AS1** in PBT GF20 compounds.



Combination with halogenated flame retardants

ACTILOX® / **ACTILOX® B** reveals its excellent performance as flame retardant synergist also in halogenated flame retardant polymers. Halogenated flame retardants release acidic decomposition products during processing. Low molecular brominated flame retardants (BrFR) suffer from migration issues. Polymeric versions of BrFR reduce these migration issues. Antimony trioxide (ATO, Sb₂O₃) is commonly used as a synergist to

Compositions of test compounds.

	Unit	Refe- rence	Synergistic combinations			
PA66	wt%	50	50	50	49	47
GF	wt%	30	30	30	30	30
BrFR	wt%	15	15	15	15	15
АТО	wt%	5	_	3	1	3
ACTILOX® 200SM	wt%	-	5	2	5	5

sec UL 94 V 1.6 mm 8 **UL 94 V 0.8 mm** After burning time 7 6 5 4 3 sec sec 2 not det. 80 0 0 0 0 0 99 BrFR 5 % ACTILOX® 200SM 5 % ATO 15 % BrFR 5 % ACTILOX® 200SM 15 % BrFR, 3 % ATO 15 % BrFR, 1 % ATO 15 % BrFR, 3 % ATO 5 % ACTILOX® 200SM 2 % ACTILOX® 200SM

*not determined

Figure 6: Results of after burning time and UL 94-V classification of PA66 GF30 compounds filled with 15 % of a polymeric brominated flame retardant (Emerald Innovation 100, Lanxess) and different combinations of ATO and ACTILOX® 200SM.

BrFR. ATO has some health and safety issues and promotes the formation of smoke in the case of a fire. In addition its high fluctuating price and limited availability makes ATO a candidate for substitution.

In the two diagrams (figure 6, 7) UL94-V and glow wire test results for a flame retardant system based on a polymeric brominated flame retardant (brominated poly-styrene-co-butadiene) in PA66 GF30 is displayed.

ACTILOX® 200SM cannot fully substitute antimony trioxide (ATO), as can be seen by the second formulation starting from the left. But 2 wt.-% of ATO based on the reference formulation (far right) can easily be substituted without any loss of performance. With increasing content of **ACTILOX® 200SM** and reduced ATO level the FR performance can be improved (increase of GWIT or reduction of afterburning time in UL94-V test).

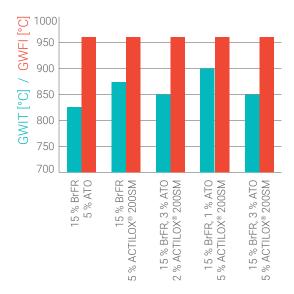
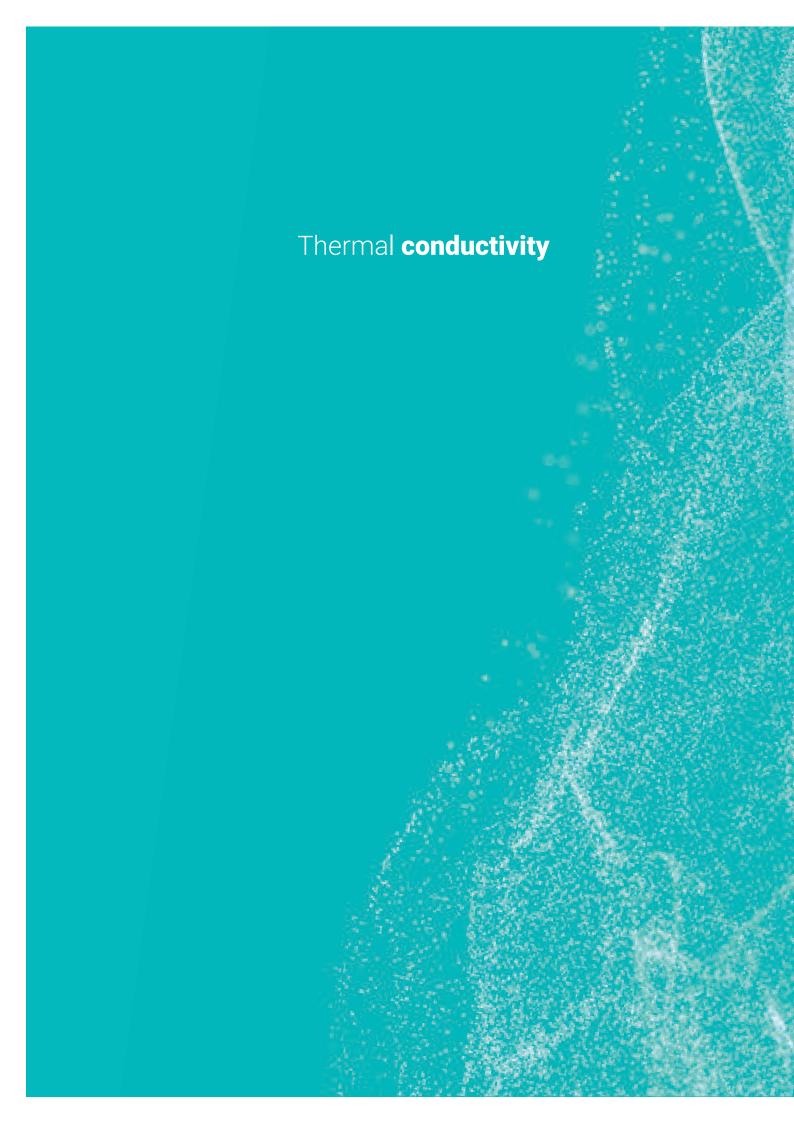


Figure 7: Results of GWIT and GWFI of PA66 GF30 compounds filled with 15 % of a polymeric brominated flame retardant (Emerald Innovation 100, Lanxess) and different combinations of ATO and ACTILOX® 200SM.



Thermal conductivity

Thermal conductivity of polymers is an important property when it comes to applications like electronic packaging and encapsulation or the more and more important becoming e-mobility sector, where good heat dissipation is needed. As polymers typically have very low intrinsic thermal conductivity, it is necessary to reinforce them with fillers. For low temperature processed plastics like polyolefins and liquid resins, Nabaltec offers a great solution with its **APYRAL® HC** grades. In regards to higher processing temperatures as

it would be necessary using engineering plastics, ATH cannot be used. For those applications our alumina grades **NABALOX® HC 170** and **NABALOX® HC 400** are highly recommended.

	D10 [µm]	D50 [μm]	D90 [μm]	BET [m²/g]
NABALOX® HC 170	2	4	15	2
NABALOX® HC 400	0.5	2	4	4

Powder parameters of Nabaltec's thermal conductive fillers:

Name, abbreviation	Formula	T _{decomp} .	TC [W/mK]	Density [g/cm³]	Mohs hardness
APYRAL® HC, aluminium hydroxide, ATH	AI(OH) ₃	200	20 - 25	2.4	3
NABALOX® HC, alumina, AO	Al ₂ O ₃	>> polymer	20 - 40	3.9	9

In the following diagram (figure 8) the thermal conductivity of polyamide 6 with 5 wt.-% glass fiber and different amounts of the **NABALOX® HC** grades mentioned above is shown. The specimen were made using a twin screw extruder followed by injection molding. The clear trend of higher TC (thermal conductivity) due to higher filling level can be seen. With 76 wt.-% of **NABALOX® HC 170** a great thermal conductivity of 2.7 W/mK can be achieved. By further increasing the filler load to

79 wt.-%, the TC can be improved to an excellent value of more than 3 W/mK. As **NABALOX® HC 400** is significantly finer in particle size, the filling level is limited. Nevertheless, the fine particle size distribution gives a great impact strength (charpy/charpy notched) as it is depicted in figure 9. Despite high filling levels with **NABALOX® HC 170**, good impact strength can be maintained. To further improve the processing behavior of the materials, processing aids can be added.

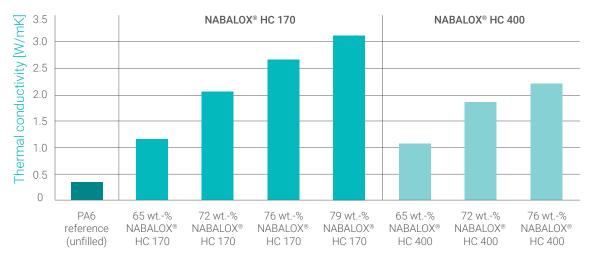


Figure 8: Thermal conductivity of PA6-compounds filled with NABALOX® HC 170 or NABALOX® HC 400.

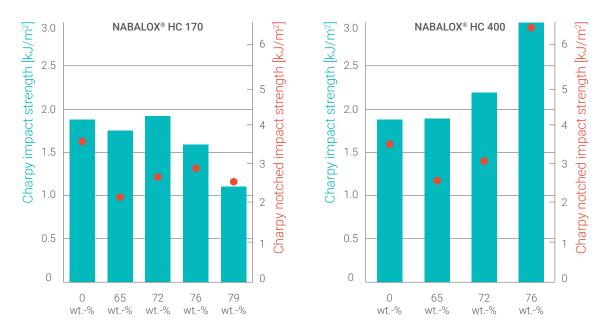


Figure 9: Impact strength of PA6 GF5-specimen filled with **NABALOX** $^{\circ}$ grades **HC 170** or **HC 400** after conditioning at 23 $^{\circ}$ C / 50 $^{\circ}$ relative humidity.

Solutions for heat barrier for E-mobility

As the number of EVs is strongly increasing, fire safety of battery casings becomes more and more important. Herein, the trend goes far beyond the well-known UL 94 V-0 classification but towards a real heat barrier which can enclose the thermal

runaway of a battery for a certain time. Solutions for heat barrier using engineering plastics are currently under development at Nabaltec. For more details please refer to our E-mobility brochure.

Annex

Abbreviation	Meaning
AO	Aluminium oxide
АОН	Aluminium oxide hydroxide
АТО	Antimony trioxide, Sb ₂ O ₃
BET	Specific surface area according to Brunauer, Emmett, Teller
BrFR	Brominated flame retardant
C _p	Thermal capacity
СТІ	Comparative tracking index
D10	10 % of particles have a smaller particle diameter/size
D50	Median particle diameter/size
D90	90 % of particles have a smaller particle diameter/size
DEPAL	Diethylphosphinate aluminium salt
DSC	Differntial scanning calorimeter
EV	Electric vehicle
FR	Flame retardant
GF	Glass fibre
GWFI	Glow wire flammability index
GWIT	Glow wire ignition temperature
HFFR	Halogen free flame retardant
LOI	Limiting oxygen index
MPP	Melamine polyphosphate
MVR	Melt volumen rates
NFR	Nitrogen based halogen free flame retardants
PA66 GF30	Glass fiber (30 %) reinforced PA66
PBT GF20	Glass fiber (20 %) reinforced PBT
PFR	Phosphorous flame retardant
SEM	Scanning electron microscope
тс	Thermal conductivity
UL 94 V	Vertical burning test according to UL 94 (Underwriter Laboratory)
Exolit™ OP 1312 (Clariant)	Flame retardant additive
Exolit™ OP 1200 (Clariant)	Flame retardant additive
Emerald Innovation™ 1000 (Lanxess)	Polymeric brominated flame retardant

Service

for our customers

Technical service development / production

Nabaltec AG develops new products and refines innovative products in close cooperation with our customers and raw material suppliers.

Here we use our own lab facilities as well as our excellent contacts to external test institutes and laboratories to offer our customers a wide range of service to support them in formulation development and test procedures.

The successful implementation of this development and the intensive customer consultations enable Nabaltec AG an interaction with our customers in a cooperative, responsible and innovative manner. This culminates in the development of high performance products at the customer as well as in our facility.

Additionally, we have the capacity to fashion tailor made products for special customer requirements and their highly sophisticated and demanding markets.

Laboratory services

Our analysis centre is responsible for independent production and quality control. It offers laboratory services for customers intending to use our large analytical equipment.

With this excellent equipment we are able to execute analytic tests in the area of inorganic solids, trace elements and water quality.

The certification in accordance with DIN EN ISO 17025 confirms the high service standards of our lab.

We will gladly inform you about our capabilities.

Nabaltec

product portfolio

ACTILOX®

Boehmite, as flame retardant filler and catalyst carrier

APYRAL® AOH

Boehmite, as flame retardant and functional filler

APYRAL®

Aluminium hydroxides, as flame retardant and functional filler

GRANALOX®

Ceramic bodies, for the production of engineering ceramics

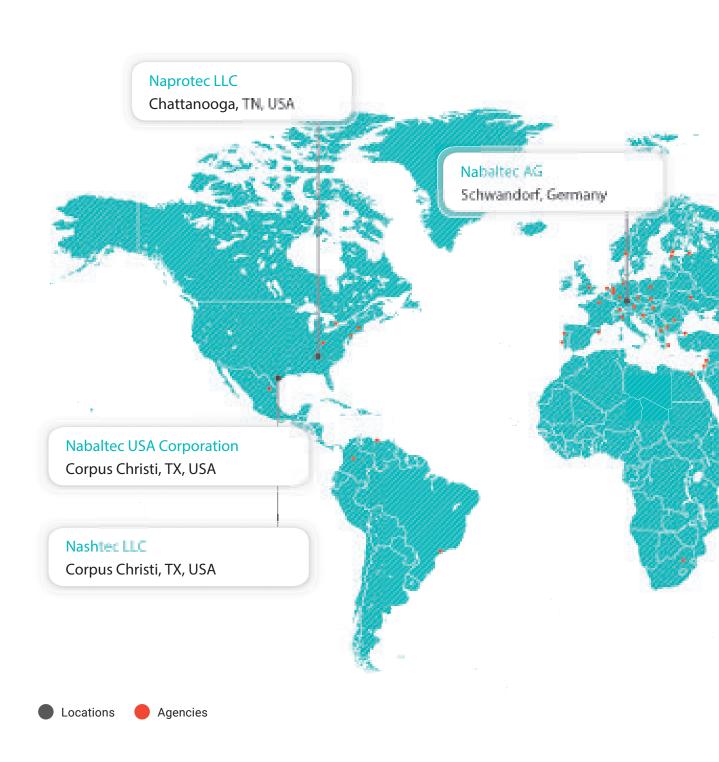
NABALOX®

Aluminium oxides, for the production of ceramic, refractory and polishing products

Nabaltec

worldwide

Visit us at our website www.nabaltec.de where you will find the latest company updates and recent versions of all available certificates free for download as PDF-documents.





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All data listed in this brochure are reference values and subject to production tolerance. These values are exclusive to the product description and no guarantee is placed on the properties. It remains the responsibility of the users to test the suitability of the product for their application.

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