Mineral filler solutions for coating applications

APYRAL[®] APYRAL[®] AOH ACTILOX[®] B NABALOX[®]



Nabaltec's mineral filler for coating applications

Product	D50* [µm]	D90* [µm]	BET [m²/g]	Oil absorption [ml/100 g]	Whiteness [%]	Mohs hardness			
APYRAL® – aluminium hydroxide as flame retardant and TiO ₂ -extender									
APYRAL [®] 40CD	1.8	3.4	3.5	22	98				
APYRAL® 60CD	1.0	2.3	6	28	98				
APYRAL [®] 120E	0.9	2.9	11	37	98				
APYRAL® 8	20	45	1.3	24	92	3			
APYRAL [®] 15	14	27	1.7	27	93				
APYRAL [®] 16	21	52	1.8	17	93				
APYRAL [®] 24	13	28	2.5	19	94				
APYRAL® AOH / ACTIL	_OX [®] for pro	otective coa	tings in bat	teries					
APYRAL® AOH 30	2.2	4	3.2	28	94				
APYRAL [®] AOH 60	0.7	1.4	5	30	98				
APYRAL® AOH 70	0.5	0.8	7.5	31	98	3 - 4			
ACTILOX [®] 200SM	0.3	0.6	18	36	98				
NABALOX® – aluminiu	m oxide for	anti-scratc	h-coatings						
NABALOX [®] NO 725-10	2	4.8	2	n. d.**	n. d.**	0			
NABALOX [®] NO 115 TC	4	13.1	1	n. d.**	n. d.**	y			

*Laser diffraction, Microtrac S3500 **not defined

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Mineral filler for coating applications

Aluminium metal hydrates are well established as flame retardant filler in a broad range of polymeric applications. Especially in the cables industry, aluminium hydroxide (ATH) is one of the most important materials e.g., to manufacture HFFR (halogen free flame retardant) cables to meet current high safety and environmental requirements.

Another application field of ATH is the wide area of coating industry, covering the spectrum from

decorative to protective and functional coatings. Nabaltec's aluminium based functional fillers impart specific properties to coatings thanks to their versatile characteristics.

This brochure shall give an insight into the different coating application areas of the Nabaltec products **APYRAL®** (aluminium hydroxide; ATH), **ACTILOX®** / **APYRAL® AOH** (aluminium oxide hydroxide, AOH, boehmite) and **NABALOX®** (aluminium oxide).

APYRAL®

Our **APYRAL**[®] products for coating applications can be divided into two classes based on the production process:

- ground grades
- fine precipitated grades

The ground hydrates **APYRAL® 8** and **APYRAL® 15** are produced by grinding aluminium hydroxide. They have a narrow, symmetrical grain size distribution. Particle morphology is plate type with a high L/D ratio, up to splintery. In liquid systems, these products are especially characterized by very low sedimentation. Both products are used in flame-protected coatings.

APYRAL® 16 and **APYRAL®** 24 are produced by application of a special grinding process. These products have only a very low portion of splintery particles - their grain size distribution is relatively broad, why they show good viscosity characteristics.

Fine precipitated grades as **APYRAL® 40CD**, **APYRAL® 60CD** and **APYRAL® 120E** are made by controlled precipitation following complete dissolution of aluminium hydroxide. This process results in very fine, morphologically optimized types. Their key features are an even particle morphology and a narrow particle size distribution.



SEM image of APYRAL® 40CD



SEM image of APYRAL® 15

The SEM images on page 4 show the differences in particle shape of a ground hydrate (**APYRAL**[®] **15**) and a fine precipitated hydrate (**APYRAL**[®] **40CD**). For more detailed information, please refer to the appropriate product datasheets (www.nabaltec.de).

The base properties of Nabaltec's aluminium hydroxide are given in the table on the right side. All **APYRAL**[®] products have a very high chemical purity of at least 99.5 % as well as high whiteness.

APYRAL ®	AI(OH) ₃
Chemical	Aluminium hydroxide
Mineral	Gibbsite
Common name	Aluminium trihydrate (ATH)
Loss on ignition	34.6 %
Density	2.4 g/cm ³
Mohs hardness	2.5 - 3
рН	8 - 9

APYRAL® AOH / ACTILOX®

The boehmite (aluminium oxide hydroxide, AOH) grades produced by Nabaltec AG are sold under the trade names **APYRAL® AOH** and **ACTILOX®**. All grades are extremely pure, crystalline products with a very low ATH residue (purity min. 99 %). This guarantees extraordinarily high temperature stability.



SEM image of **APYRAL®AOH 60**



SEM image of ACTILOX® 200SM

Additionally, **ACTILOX**[®] / **APYRAL**[®] **AOH** shows a very good chemical resistance, especially a very high acid resistance. The range of products includes grades with a D50 of around 3 µm down to submicron sized boehmite with D50 around 300 nm (**ACTILOX**[®] **200SM**).

Its high whiteness combined with the extreme fineness and excellent dispersion properties makes **APYRAL® AOH** an interesting whitening pigment. The uniform particle shape and especially the cubic shape of **APYRAL® AOH 30**, **AOH 60** and **AOH 70** guarantee good processing properties (see SEM image boehmites). Main properties of Nabaltec's boehmites are given in the table below.

APYRAL® AOH ACTILOX®	АЮОН
Chemical	Aluminium oxide hydroxide
Mineral	Boehmite
Common name	Aluminium monohydrate (AOH)
Loss on ignition	17 %
Density	3.0 g/cm ³
Mohs hardness	3 - 4
рН	7 - 8

NABALOX®

The high-performance aluminium oxide grades produced and sold by Nabaltec are known under the trade name **NABALOX**[®]. Important products for the coating industry are the milled hard calcined alumina **NABALOX**[®] **NO 725-10** and **NABALOX**[®] **NO 115 TC**. The products show a high purity and a defined and adjusted grain size distribution, what makes them suitable for applications like scratch resistance coatings.

Main properties as well as SEM pictures can be found in the following.

Main properties of NABALOX[®] NO 115 TC and NABALOX[®] NO 725-10.

	D50* [µm]	BET [m²/g]	Degree of calci- nation	Al ₂ O ₃ [%]	
NO 115 TC	6	1	hard	99.6	
NO 725-10	2	2	hard	99.8	



SEM image of NABALOX® NO 115 TC



SEM image of NABALOX® 725-10

Functionalities of Nabaltec's mineral filler **in coatings**

Functionalities of mineral filler in coatings



Flame retardant filler

Scheme of the processes involved during burning of an APYRAL® filled polymer.

ATH is one of the most widely used flame retardants. It starts to release water at temperatures above 200 °C. During this decomposition process, energy (heat) is consumed and, correspondingly, a temperature stable Al₂O₃-layer is generated. The particles of this layer have a high specific surface area and therefore, the development of smoke and consequential damage in case of a fire is strongly reduced. The basic mechanism can be seen in above scheme. For more details about the flame-retardant mechanism please refer to **Nabaltec's brochure "Mineral based flame retardancy with metal hydrates**"

In the past few years, ATH has experienced the highest growth among all flame retardants. Their good environmental compatibility and their favorable price-to-performance ratio make **APYRAL**[®] products sustainable flame retardants.

In case of higher required heat stability, boehmite (AOH) is the product of choice. The flame-retardant mechanism works in a similar way to ATH. To achieve particularly effective and environmentally friendly fire protection, ATH and AOH are used equally as pigments and filler in dispersions.

When considering coating applications, there are basically two main requests. One is the requirement to protect the substrates of an entire object by using a fire-protective coating. In this case, intumescent coating or ablation coating can be applied (see pages 14/15).

The second request implements the protection of the coating layer itself, which is of a decorative or functional nature. This includes for example gel coats, potting, or encapsulating compounds, sealants, and a lot of architectural coatings.

UV-transparent filler in UV cured systems

Aluminium hydroxide as well as boehmite absorbs only minor proportion of UV radiation in the wavelength range from 220 to 400 nm, which is used for most UV photo initiators.

Literature shows that ATH has no interaction with transmitted UV light. It is also shown that an ATH filled system has a higher degree of crosslinking compared to not filled or SiO₂-filled systems.[1] Boehmite has a high compatibility to a lot of polymers that are used in 3D-printing and is a suitable

product as soon as higher temperature stability (> 200 °C) is needed.

Fine **APYRAL**[®] as well as **APYRAL[®] AOH** / **ACTILOX**[®] products can be used as UV-transparent filler in UV cured systems.

[1] A.A. Parker, etc. "Aluminum hydroxide: A UV transparent filler for UV-curable coating" J. of coating technol. 66 (1994), Nr. 829, S. 39-46

TiO₂ extender

 TiO_2 is a widely used whitening agent. To achieve high hiding power and high color strength, the scattering effect needs to be ideal. To reach this aim, the most common commercialized TiO_2 grades have a primary particle size around 280 nm - half of the wavelength of visible light. For an optimal scattering effect, it is most important that the TiO_2 particles are distributed uniformly over the complete system. This phenomenon is called **spacing**. To avoid agglomeration of the TiO_2 particles, suitable filler – so-called extender materials – can be used to fill the spaces between the particles.

The spacing particles need to have a particle size close to that of TiO_2 . The major reason Nabaltec's products can be used as TiO_2 extender is not only

the high whiteness but also the optimal particle size to improve TiO_2 effectivity by spacing the TiO_2 particles in an ideal way.

ACTILOX® 200SM with its D50 of around 280 nm is the ideal product to substitute up to 25 % of TiO₂ amount. Next to this, products like **APYRAL® 60CD** and **APYRAL® 120E** with a slightly larger particle size distribution can also be used to substitute TiO₂ up to a certain amount.

In the table on the next page, the results of a simple formulation using **APYRAL**[®] **120E** are given. The findings clearly show that a substitution of up to 10 % of TiO₂ by **APYRAL[®] 120E** is possible and leads to similar color of the dispersion.

	Formulation A [%]	Formulation B [%]
UP resin / P80	48.6	48.6
Dispersant / Solplus D570	0.48	0.48
Curing agent / Curox M	0.73	0.73
Accelerator	0.48	0.48
CaCO ₃	47.7	47.7
TiO ₂ /TiONA 696	1.94	1.75
APYRAL® 120E	0	0.19
Whiteness	72.8	70.7
Yellowness Index	11.9	11.3
L*	91.4	90.2
a*	0.16	0.23
b*	6.08	5.70

Simple UP formulation using 10 % of **APYRAL® 120E** as TiO₂ extender.

According to CIEL*a*b* color space.

Filler for anti-scratch coating

To increase the anti-scratching performance of a coating, aluminium oxide is in most cases the filler of choice due to its extreme Mohs' hardness of 9. An exceptionally smooth surface of the coating can be achieved by using products like **NABALOX® NO 115 TC**. To fulfil highest quality requests from the coating industry, these products undergo a special production-accompanying step, which is termed "TC" (top cut). Due to different technical requests such as stability of sedimentation, viscosity properties, effectivity of flame retardancy etc., a recommendation of the right product can only be made on an individual basis.

Nevertheless, some fundamental formulations and results are summarized in the following two tables.

Basic formulation	for	a wet	anti-scratching	coating	layer	of	12 µn	n and	a dry	layer	of 4	μm	using	NAB/	LOX ®
products.															

	Reference [%]	Formulation A [%]	Formulation B [%]
Clear Coat / Rowabase 84875	67	63	63
Crosslinker	5.8	5.44	5.44
MEK	27.2	29.22	29.22
NABALOX® 725-10	0	2.34	0
NABALOX® 115 TC	0	0	2.34

Abrasion tests have been performed on the dry coating layer. Abrasion resistance was tested for one side using an abrasive fleece from company 3M subsequently measuring the gloss retention of the surface in %. Via scratch hardness tester Erichsen 413 the resistance of a sample to a certain pressure while being mounted on a rotating table has been evaluated. The results are given in Newton.

As can be seen from the findings, the scratch resistance can be improved by using one of the above given **NABALOX**[®] products. Especially with **NABALOX**[®] **NO 115 TC** highest requirements can be met.

Abrasion test results comparing the use of **NABALOX®** products via measurement of gloss retention and scratch resistance.

	Reference	Formulation A / NABALOX® NO 725-10	Formulation B / NABALOX® NO 115 TC
Dry abrasion test with 10DH abrasive fleece from 3M / Gloss retention [%]	75	91.1	95.8
Dry abrasion test via Erichsen 413 / Scratch hardness [N]	1.7	1.9	2.1

Application examples

General considerations for slurry preparation

Solid content, viscosity, and stability (sedimentation) are the main factors which need to be considered for the preparation of a slurry. Viscosity and stability in turn are highly dependent on the choice of the filler.

Sedimentation is a major issue during preparation and storage of a dispersion. It is a complex function of the particle size, the particle size distribution, the filling level, and the viscosity of the dispersion.

Considering a water-born dispersion, with increasing particle size of the filler material and especially with a D50 higher than 1 μ m, sedimentation is increasing significantly. A fine precipitated material like **APYRAL® 40 CD** (D50 = 1.8 μ m) can be easily stabilized at a high solid content of 70 % with 0.5 % dispersant in a water-based slurry for more than 48 hrs. Its viscosity is still far lower than 1 Pa*s and almost no ageing of the slurry is observed.

The situation is different when a ground ATH like **APYRAL**[®] **15** with a D50 of 14 μ m is used. The larger the particle size, the higher the sedimenta-

Chopper disc of a dissolver to create maximum shear rates (based on Wilhelm Niemann GmbH & Co Maschinenfabrik, Melle, Germany).

tion velocity of the particles. Upon reversion, viscosity will be less high as the specific surface area of these coarser products is quite small.

An important point to have in mind is the fact that a ground ATH cannot be ground to a D50 of around $1-2 \mu m$ without increasing BET values in an extreme way. This correlation is illustrated in below figure. The high surface area of these products would result in drastically high viscosity of the slurry.

The choice of the right Nabaltec product depends on the final application as well as on requirements on whiteness, surface roughness, porosity, and the final processing conditions.



Figure 1: Correlation between D50 and BET of fine precipitated and ground ATH.

Application examples

Water-born fire protection coatings

The following chapter highlights coating solutions using **APYRAL**[®], that function as fire protection coatings for wood, steel, or concrete. The aim of those coatings is to protect structural components against ignition or, in case of a fire, to act as insulative barrier and increase the fire resistance. The protective effect of these coatings is based on chemical reactions, which can be generally grouped into two different principles of action: the ablation coating and the insulation-forming fire protection coating.

Ablation coating

Ablation coatings are used wherever building components are exposed to moisture, to chemical stresses or special outdoor conditions. The coatings contain substances that undergo chemical changes in an endothermic reaction when exposed to heat. Within this reaction, the expansion is low.

When using **APYRAL**[®] products like our fine precipitated grades **APYRAL**[®] **40CD**, the ATH acts with its typical chemical and physical processes. Water that is released during burning cools the coated components and dilutes the burnable gases. After the burning, a porous, inorganic, non-flammable ceramic will remain and give a thermally insulating effect for the product.

To meet the requirements for flame retardancy according to fire tests like e.g., DIN 4102 B2 / B1, the amount of ATH must be at least 30 % up to 70 % of the total weight in **dried coating layer** – depending on the chemical composition of the polymer component in the dispersion.

A basic formulation based on sodium silicate for an ablation coating can be found in the right-hand table.

An ablation coating using above formulation was applied to an EVA sample (thickness of 2.8 mm)

Sodium silicate-based formulation for ablation coating using **APYRAL® 40CD**.

Formulation	[%]
Sodium silicate	20
APYRAL® 40CD	50
Wetting agent	1
Sodium polyacrylate dispersant	1
Water	18
Polyacrylate binder	10

with around 0.15 mm coating thickness. The sample was flame-treated using a Bunsen burner flame and time was measured until the specimen started to burn.

Figure 2 shall illustrate the effect of the ablation coating on to the EVA compound.

The uncoated plate (A) started to burn after 3 minutes while the coated version (B) did not burn for more than 10 minutes. The test was terminated after this time.



Figure 2: Pictures of an uncoated (A) and a coated (B) EVA sample after application of a Bunsen burner flame. Coating is an ablation coating with **APYRAL**[®] **40CD**.

Intumescent coating

A thermal insulating effect can also be created by formation of a voluminous carbon-containing layer, which isolates the building materials or surfaces underneath and protects it from heat. The formation of this insulation layer is based on various temperature-dependent chemical reactions.

Insulating layer formers generally consist of binders, gas formers, carbon formers, catalysts, and other additives. The application of an intumescent coating over large areas, e.g., on steel profiles, focusses on maintaining the functional integrity due to thermal insulation and cooling of the components. In case of bulkheads or joints, the foamed coating prevents the passage of fire and smoke over larger distances. When using fine precipitated ATH like **APYRAL® 40CD** or **APYRAL® 60CD** in such an intumescent coating, a higher char expansion can be observed due to the additional release of H₂O.

On page 16, a waterborne formulation for intumescent fire-retardant paints is given.

For mixing, the ingredients of composition I need to be premixed with a high-speed mixer for 30 minutes under cooling. Temperature should not be higher than 50 °C.

Subsequently, ingredients of composition II can be added to composition I.

Composition I	[%]	Composition II	[%]
Water	12 - 15	PVAc dispersion	24 - 26
AYPRAL® 40CD/ 60CD	1 - 2	Coalescing agent	1 - 2
Carbon donor	7 - 10	Sodium polyphosphate	0.6 - 0.8
Blowing agent	7 - 8	Water	7 - 9
Acid donor	20 - 24		
TiO ₂	4 - 6		
Dispersant	0.8 - 1.2		
Defoaming agent	0.2 - 0.3		
Hydroxyethyl cellulose	3 - 5		

Formulation of a water-borne intumescent coating using fine precipitated **APYRAL®** grades.

Protection coating for Lithium-ion batteries

A special case of protection coating is the coating of separators and electrode edges for the use in Lithium-ion batteries. Both applications will be shortly mentioned in the following. For a detailed version and sample formulations, please refer to our **brochure for E-mobility**.



Separator coating

Lithium-ion battery (LIB) technology is of central importance for the growth of electromobility and the progressive development of stationary storage for renewable energies worldwide. Nabaltec AG supports these developments in the battery industry with its boehmite product family **APYRAL® AOH / ACTILOX**.

The white boehmite powders are processed into a dispersion which is then applied to the separator either on one side or on both sides, e.g., by using a gravure roller coating or slot die system.

The separator coated in this way gains heat-resistance and prevents shrinkage of the polymer membrane. It is crucial that the coating is still permeable and remains a high porosity. Thanks to this ceramic coating the separator can withstand a temperature of up to 250 °C, depending on the substrate. In comparison, untreated separators already shrink at around 90 °C. Additionally, the coated separator is reinforced against dangerous dendrites which may be formed during charge/discharge cycles and pierce the non-coated separator causing a short circuit by contacting the cathode.

The heat stable coating with **APYRAL® AOH** and **ACTILOX®** makes the battery much safer and

more resistant to short-circuit reactions and prevents possible fires and explosion.



Figure 3: Three-dimensional structure illustration of a Lithium-ion battery cell, highlighting the coating of the separator (green).

LIB Electrode edge coating

An additional protection against internal short circuits besides separator coating is the electrode edge coating (see figure 4). The main functions of the electrode edge coating are:

1. Prevention of cutting burrs caused issues

The current collector (Al- or Cu- foil) covered with the boehmite reduces the occurrence of cutting burrs and the risk of such burrs to puncture through the separator, touching the counter electrode and causing a short circuit.

2. Isolation to avoid an internal short circuit caused by possible deviation of cell assembling.

Other than for separator coating, the industry chooses boehmite for this application right from the start. The determining metric to use boehmite is the low hardness compared to alumina. Boehmite coating improves the overall performance of the electrode cutting process and avoids a secondary contamination through abrasion of cutting tools.

Detailed information and slurry preparation can be found in our **brochure "Mineral fillers solutions for E-Mobility**", page 10 onwards.



Figure 4: A die coating system in which the boehmite dispersion is applied to the cathode edge.

Non-water-based coatings

While all formulations from previous chapter were water based, the following chapter is covering

non-water-based coatings like powder coating and PVC plastisol coating.

Powder coating

Powder coating is widely used as a method for high-quality finish of a broad range of products. Adding **APYRAL**[®] to the powder mixture, UV-resistance of the coating is enhanced due to the UV-transparent behavior of ATH. Furthermore, mechanical properties such as scratch hardness can be increased significantly with increasing ATH content. Impact resistance decreases only within 5 % of the initial resistance even with the maximum ATH loading.

A simple formulation is given in the following table.

Due to the variety of **APYRAL**[®] products, the customer has several options for the finetuning of the final gloss level. The choice of the right **APYRAL**[®] product also depends on the aimed film thickness.

Sample formulation for a powder coating mixture using **APYRAL**[®].

Formulation	[%]
Polyurethane resin	40 - 55
Crosslinker	8 - 25
Pigments	25 - 40
APYRAL ®	5 - 25
Flow modifier	1 - 2.5
Degassing agent	0.5 - 1.5

PVC plastisol coating

PVC plastisol can be found in diverse application fields like construction and transportation due to its high resistance to corrosion, against chemical exposure, environmental impacts as well as electricity.

Next to the above demands, the requirements for high fire safety need to be met. Eco-friendly and smoke suppressant flame retardants like ATH are the product of choice. Nabaltec is offering **APYRAL® 16** and **APYRAL® 24** e.g., for flooring and roofing applications. Both products not only have an attractive price, but they also meet the required conditions for the applications like high whiteness, good dispersion properties, and low oil absorption.

High surface finish requirements, good foaming properties of the product as well as a proper fabric wetting during the manufacturing process make Basic formulation of a PVC-P for fabric coating based on **APYRAL**[®]. Amount of ATH and plasticizer depends on specific performance requirements.

Formulation	phr
PVC (K = 57)	70
PVC filler resin	30
DINP	65
Ca/Zn or Ba/Zn stabilizer	3
APYRAL [®]	30 - 50
TiO ₂	5
CaCO ₃	10 - 15
Sb ₂ O ₃	3

our fine precipitated ATH grades **APYRAL® 40CD** and **APYRAL® 60CD** appropriate products to use for applications like coated fabrics and artificial leather. In the left-hand table a basic formulation of a PVC plastisol for fabric coating is given. For higher flame-retardant requirements, the use of phosphates at the cost of phthalates plasticizers is recommended.

The below diagram displays the effect of increasing ATH content on flame retardancy (measured as limiting oxygen index, LOI) and viscosity of the plastisol.



Figure 5: LOI and viscosity of ATH filled plastisol before curing (carried out with basic formulation given in above table with 15 phr CaCO₃; without Sb₂O₃)

As it was expected, LOI and viscosity increase with increasing ATH loading. The comparison between **APYRAL® 24** and **APYRAL® 40CD** shows the effect of fine filler. The finer the filler, the more plasticizer will be absorbed and the higher is the viscosity of the plastisol. Nevertheless, a loading of 50 phr **APYRAL® 24** still shows good flowing properties, which is decisive in plastisol application.

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

GRANALOX®

Ceramic bodies, for the production of engineering ceramics

APYRAL® AOH

Boehmite, as flame retardant and functional filler

NABALOX®

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

APYRAL[®]

Aluminium hydroxides, as flame retardant and functional filler



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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