Metal hydrates for elastomers

APYRAL® APYRAL® AOH ACTILOX®



Metal hydrates for elastomers

Product	D50 [µm]	BET [m²/g]	Oil absorption [ml/100g]	Main application				
APYRAL® – Ground								
APYRAL® 1E	50	0.2	21	Carpet backing, e.g. EPDM				
				Cable bedding compounds				
APYRAL® 8	15	1.3	24	Roofing sheets, e.g. EPDM, Bitumen				
APYRAL® 16	16	1.8	17	Flooring, e.g. EPDM				
APYRAL® – Fine-p	recipitated	L						
APYRAL® 40CD	1.5	3.5	22	Cable Insulation and Jacketing Compounds, for: offshore cables, specialties, e.g.				
APYRAL® 60CD	1	6	28	EVA, EVM, EPDM, Silicone, TPU Sealing, for: Railcar windows, compartments,				
APYRAL® 120E	0.9	11	37	e.g. EVM, EPDM, Public Building, e.g. EPDM, Offshore-Installations, e.g. EVM				
APYRAL® 200SM	0.4	15	48	High Voltage Insulators, e.g. Silicone, EPDM Mining belts, e.g. EPDM				
APYRAL® 40 VS1	1.5	3.5	33	Materials for Offshore- Installations, e.g. EV. Insulation Foams, e.g. for				
APYRAL® 40 HS1	1.5	3.5	26	Building & Construction, e.g. PVC / NBR, Air condition pipes, e.g. EPDM, Water pipes, e.g. EPDM				
ACTILOX® - Aluminium oxide hydroxide								
ACTILOX® B30	2.3	3	28					
ACTILOX® B60	1.2	5	30	Additives for Diverse Applications				
ACTILOX® 200SM	0.3	17	36					

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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Introduction

Elastomers are polymer materials which are unique due to their special mechanical properties.

The crosslinking of the polymer chains after moulding and their low glass transition temperature enable those materials to gain back their original shape, even after mechanical deformation.

This characteristic clearly distinguishes elastomers from other polymer types such as thermoplastics (re-meltable and re-mouldable but hardly elastic) and liquid resin based thermosets (not re-meltable and not elastic).

Different types of elastomers are classified according to the chemistry of their backbone structure. The last letter of the polymer designation indicates to which class this material belongs.

Selected examples are given in the table below [1].

Group	Backbone structure	Example
М	Saturated backbone, Methylene-type	EPDM, EVM
R	Double bonds, Rubber-type	NR, CR
U	Urethane-group	AU, EU
Q	Siloxane-group	MQ
0	Oxygen	PO

The abbreviations of the monomers which were used to form the polymer are put in front of the class designation to form the polymer's name. Selected examples are given in the following table [2].

Mono- mer abbre- viation	Monomer	Rubber example			
В	Butadiene	NBR	nitrile butadiene r.*		
С	Chloroprene	CR chloroprene			
D	Diene	EPDM	ethylene propylene diene r.*		
Е	Ethylene	EPDM	ethylene propylene diene r.*		
М	Methyl- siloxane	MQ	methyl polysiloxane r*		
N	Nitrile	NBR	nitrile butadiene r.*		
Р	Propylene	EPDM	ethylene propylene diene r.*		
S	Styrene	SBR	styrene butadiene r.*		

r.* rubber

According to this, styrene butadiene rubber for example is designated as SBR.

Elastomer compounds offer an optimal property profile for several applications, for example where mechanical impact must be absorbed or where flexibility and sealing must be guaranteed.

Thus, elastomeric compounds are used for the production of very special parts, but also for products which we use every day.

In all areas where people or goods must be specially protected from harm and damage from fire, the resistance of a compound against ignition as well as a slow rate of flame spread and smoke development are decisive factors. Application areas for such materials are public transport and building products. For this purpose Nabaltec offers manufacturers of elastomeric materials a broad range of different grades of metal hydrates.

In particular, their environmental friendliness and their favourable cost performance ratio make **APYRAL®** (aluminum tri hydroxide / ATH) and **ACTILOX®** (aluminum oxide hydroxide / AOH) important sustainable flame retardants.

In the following table those mineral flame retardant product groups of Nabaltec are shown. Listed are chemical composition, mineral structure and the commonly used synonyms.

APYRAL®	AI(OH) ₃
Chemical:	Aluminium hydroxide
Mineral:	Gibbsite
Common name:	Aluminium tri hydrate
Abbreviation:	ATH
APYRAL® AOH ACTILOX®	AIOOH
Chemical:	Aluminium oxide hydroxide
Mineral:	Boehmite
Common name:	Aluminium mono hydrate
Abbreviation:	AOH

This brochure shall offer the reader an insight into the broad possibilities to develop an elastomeric compound formulation with mineral flame retardants. It complements our brochures "Mineral Based Flame Retardancy with Metal Hydrates", "Metal Hydrates for Thermosets", "Metal Hydrates for Cables", and "Metal Hydrates for PVC".

Due to the diversity of available resins, processing methods, additives, and fillers for the production of elastomeric compounds, this brochure cannot be exhaustive. It is intended to give an overview and hints for own formulation developments.



APYRAL®

APYRAL®

Our **APYRAL**® products for elastomers can be divided into two classes, on the basis of their property profiles:

- · Ground grades
- · Fine precipitated grades

Fine precipitated **APYRAL®** products are broadly used in elastomeric compounds for fire protection of materials which have to fulfill special mechanical and flammability requirements like in cables, seals, and many other products used e.g. for applications in public buildings, on ships, or oil rigs.

Of particular importance for use in halogen free flame retardant (HFFR) compounds are APYRAL® 40CD, APYRAL® 60CD and APYRAL® 120E.

A special material of the fine precipitated **APYRAL®** product class is **APYRAL® 200SM**. Its median particle size (D50) of only 0.4 µm offers additional possibilities for the user to optimize existing flame retardant compounds by additive use.

To improve further the mechanical properties and surface characteristics of the final compound, **APYRAL®** can be surface treated with organic substances to enhance the coupling between the polymer matrix and the surface of the mineral.

APYRAL® 40 VS1 and APYRAL® 60 VS1 are vinyl silane treated products, especially used in crosslinked LSOH (low smoke zero halogen) compounds, e.g. on silicone rubber (SiR) basis. APYRAL® 40 HS1 contains a special hydrophobic silane and is used in specialty silicone elastomers and TPU compounds. Other surface treatments of APYRAL® are also available depending on the specific requirements of the users.

For compounds in which the mechanical properties and the surface characteristics are not the main focus, e.g. in cable bedding compounds or certain roofing materials, ground APYRAL® grades are recommended, like APYRAL® 8 and APYRAL® 16.

Chemical and physical parameters

APYRAL® products have a very high chemical purity of ca. 99.5 %. The remaining constituents are mainly sodium oxide, which is part of the crystal lattice and partly adhering to the **APYRAL®** surface in hydrated form, as well as traces of iron and silicon compounds. Due to its whiteness **APYRAL®** behaves neutral to the colouring of polymers.

Its Mohs hardness of 2.5 – 3 causes no tool abrasion problems even in highly filled molten masses. Its relatively high heat capacity $c_{\rm p}$ of $1.65~\rm J/gK$ at

400 K (127 °C) has a beneficial effect on the dimensional stability under heat for **APYRAL**® filled polymers.

With a specific density of 2.4 g/cm³, APYRAL® is a medium dense mineral filler. As a result of the required filling ratios, the density of the flame retardant plastics is increased compared to the virgin polymer. APYRAL® is one of the most attractive flame retardants, even on the basis of a volume specific cost balance.



ACTILOX®

Boehmite grades produced by Nabaltec are finding a new range of application as functional fillers within the polymer industry, particularly due to their high temperature stability up to 340 °C (613 K). These grades are sold under the trade names **APYRAL® AOH** and **ACTILOX® B**.

Currently, three products of this range are available in different grades of fineness for application in elastomers: ACTILOX® B30, ACTILOX® B60, and ACTILOX® 200SM. Among them, ACTILOX® 200SM with its high fineness and optimised processing behaviour shows advantages for melt compounding processes which are used for the production of elastomeric compounds.

The flame retardancy efficiency of boehmite is lower, compared to aluminium hydroxide and magnesium hydroxide (see also our brochure "Mineral

Based Flame Retardancy with Metal Hydrates"). ACTILOX® B and ACTILOX® 200SM should therefore be used in combination with other flame retardants to comply with most severe flame resistance standards. In thermoplastic and elastomeric compounds, synergistic effects can be observed if ACTILOX® B or ACTILOX® 200SM are combined with aluminium hydroxide, magnesium hydroxide, or metal phosphinates, for example.

When ACTILOX® B and ACTILOX® 200SM are used as co-additives to APYRAL® the remaining char after combustion forms a very homogeneous, nearly fully closed surface. During the start of a fire, the char forms a protective shield against heat and it retards the release of volatile and flammable decomposition products and the incipient thermal breakdown of the polymer.

This is even more successful if the char layer is more closed and stable.

Chemical and physical parameters

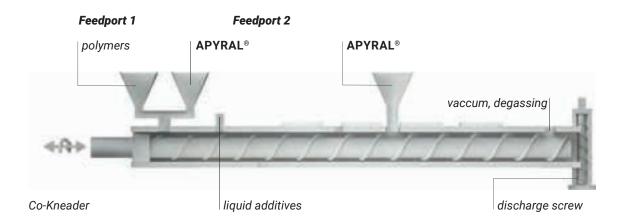
All ACTILOX® B grades and ACTILOX® 200SM are extremely pure (ca. 99 %) crystalline boehmite products with a very low aluminium hydroxide content. This ensures extraordinary temperature stability. ACTILOX® B and ACTILOX® 200SM can be easily processed up to 340 °C. Their very low electrolyte content is crucial for the application of ACTILOX® B and ACTILOX® 200SM in electrically insulating products.

ACTILOX® B and **ACTILOX® 200SM** are ideal for the use in electrically insulating heat sinks due to their high heat capacity ($c_p = 1.54 \text{ J/gK}$ at 500 K, 227 °C) combined with high temperature stability.

In addition, **ACTILOX® B** and **ACTILOX® 200SM** can be used as white pigments due to their high whiteness in conjunction with extreme fineness and good dispersion properties.



Processing guidelines



There are several possibilities for the production of elastomeric compounds which are filled with **APYRAL®**, **ACTILOX®** B or **ACTILOX®** 200SM.

Batch processing in internal mixers (see right sketch) and/or on two roll mills are often used if good dispersion of large amounts of fillers is necessary and if regular product changes occur.

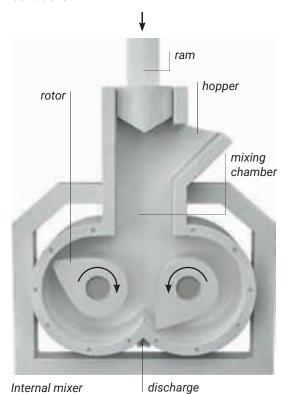
Continuous extrusion via twin screw extruders or co-kneaders (see sketch above) is recommended for large volume products and if the dispersion of the filler can be guaranteed during the residence time of the melt in the extruder without overheating the materials.

To garantee a sufficient dispersion and to ease the incorporation of the metal hydrates into the polymer, the twin screw extruder or co-kneader should have at least two feedports.

Subsequently, the compounds are shaped, for example via extrusion, and crosslinked. The crosslinking can be initiated chemically, such as for sulphur or peroxide crosslinking or energetically like in E-beam crosslinking.

The temperature limits for **APYRAL**® (200 °C) and for **ACTILOX**® **B**, and **ACTILOX**® **200SM** (340 °C)

must be considered. It is recommended to keep the temperature of the molten mass below 190 °C while producing **APYRAL**® filled elastomers via batch compounding, for example. In the case of continuous processing, higher temperatures are possible for short residence times. But this must be tested depending on the individual process conditions.





Application examples

The following chapters will demonstrate the performance of **APYRAL**® and **ACTILOX**® in different elastomeric compounds.

The influence of a certain type of mineral flame retardant on the properties of the final compound

shall be demonstrated. Mechanical properties and flame retardancy performance of the materials will be compared.

EPDM based compounds

EPDM (Ethylene-propylene-diene-elastomer) is a versatile polymer which combines high elasticity and good resistancy against moisture, ozone and other media. Thus, it can be used for the production of a broad range of compounds which can be adjusted to diverse requirements and applications using additives and fillers.

EPDM is recommended as base polymer for the production of HFFR compounds with mineral flame retardants as EPDM can hold even large amounts of minerals and still retains good mechanical properties and processing behaviour.

High filling levels are necessary for mineral based flame retardant compounds as the flame resistance of a compound increases with growing amounts of minerals (see also our brochure "Mineral Based Flame Retardancy with Metal Hydrates").

Two examples for EPDM based formulations are shown on the following pages. One of them is a black sulphur cured EPDM compound and the second example is a bright peroxide crosslinked material.

Fine precipitated minerals were used as flame retardant fillers. These were **APYRAL® 40CD**, **APYRAL® 60CD**, **APYRAL® 120E**, **APYRAL® 200SM** with a median particle size (D50) ranging from 1.3 down to 0.4 µm. Additionally, the submicron sized boehmite **APYRAL® 200SM** was used as filler.

The compounds were produced on a laboratory scale two roll mill according to the formulations in the following tables. Subsequently, plaques were pressed out of these masses and they were cured at 180 °C. The specimens for mechanical analyses and flame retardant tests were cut out of these cured plaques.

Black, sulphur cured EPDM compounds

Black, sulphur cured EPDM compound examples

Component [phr]		APYRAL® 60CD	APYRAL® 120E	APYRAL® 200SM	
EPDM, Keltan® 8340		100	100	100	
Additives		7	7	7	
Curing		14.55	14.55	14.55	
Plasticiser		65	65	65	
Carbon black		60	60	60	
APYRAL® 60CD		155	_	-	
APYRAL® 120E		_	155	-	
APYRAL® 200SM		_	_	155	
Total		401.55	401.55	401.55	
Characteristic data		APYRAL® 60CD	APYRAL® 120E	APYRAL® 200SM	
Mooney viscosity (MI (ML (1+4) 100 °C)	U)	64	75	80	
Tensile Strength	[MPa]	9	8.2	8.1	
M 500	[MPa]	6.7	6.5	6.2	
Elongation at Break [%]		616	641	654	
Shore A, 23 °C	(dimensionless)	56	63	63	
LOI	[% O ₂]	26	27	27	

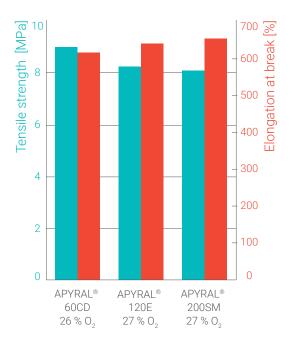
Compounding on laboratory scale two roll mill, specimens made of compression moulded and cured (180 °C) plaques.

The compound above represents a starting formulation for an electrical insulating sealing profile with a mineral content of 155 phr.

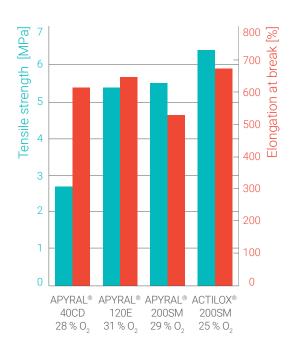
The Mooney viscosity of these mixtures increases if finer **APYRAL**® particles with a higher specific surface area are used.

The mechanical properties of the cured compounds are influenced by the type of mineral which is used, as demonstrated in the left chart next page. However, the LOI values are only slightly affected by the type of **APYRAL®** at this moderate filling level.

The user can easily adjust the mechanical characteristics of the compound to the requirements for a certain application by the choice of an appropriate **APYRAL®** product with rather stable flame retardancy properties.



Mechanical properties and LOI of the **black** EPDM compounds



Mechanical properties and LOI of the **brigth** EPDM compounds

Bright, peroxide cured EPDM compounds

A bright peroxide cured EPDM formulation with the higher filling level of 250 phr is exemplary for the application building profile (see table on following page).

For the given compound, the use of APYRAL® 40CD gives only poor results, as one can see in the right chart above. APYRAL® 40CD has the lowest specific surface area of all products compared here and is not the appropriate APYRAL® product for this kind of compound. APYRAL® 120E by contrast results in an optimal balance of tensile strength and elongation at break. The use of APYRAL® 200SM leads to similar values in tensile strength and shows a higher shore hardness, but the elongation at break decreases significantly when using this submicron sized filler.

The LOI values of the compounds which contain **APYRAL®** as mineral flame retardant are around $30 \% O_2$.

When using the boehmite **ACTILOX® 200SM** a very good tensile strength elongation at break balance combined with a relatively low Mooney viscosity can be achieved. Unfortunately at the cost of flame retardancy. The LOI drops to only 25 % $\rm O_2$ (see right diagram above).

If the mechanical properties of the compound are most important, **ACTILOX® 200SM** is superior to the aluminium hydroxide products in this compound. But if a certain level of flame retardancy is required, **APYRAL®** should be used or boehmite should be combined with aluminium hydroxide or other flame retardants. (See also our brochure "Mineral Based Flame Retardancy with Metal Hydrates").

Bright, peroxide cured EPDM compound examples

Component [phr]		APYRAL® 60CD	APYRAL® 120E	APYRAL® 200SM	ACTILOX®200SM	
EPDM, Vistalon™ 7500 [A]		100	100	100	100	
Additives [A]		12	12 21		21	
Curing [A]		16	16	16	16	
Plasticiser [A]		60	60	60	60	
APYRAL® 40CD		250	_	-	-	
APYRAL® 120E		-	250	-	-	
APYRAL® 200SI	VI	-	_	250	-	
ACTILOX® 200S	ACTILOX® 200SM		_	-	250	
Total		438	438	438	438	
Characteristic	Characteristic data		APYRAL® 120E	APYRAL® 200SM	ACTILOX® 200SM	
Mooney viscosity (ML (1+4) 100 °C		52	126	133	92	
Tensile strength	[MPa]	2.7	5.4	5.5	6.4	
M 500	[MPa]	1.4	3.7	4.5	3.8	
Elongation at Break	[%]	615	648	530	675	
Shore A, 23 °C (dimensionless)		51	64	67	62	
LOI [% O ₂]		28	31	29	25	

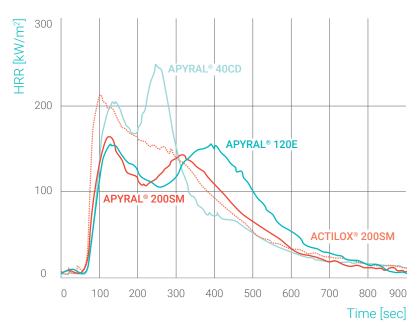
Compounding on laboratory scale two roll mill, specimens made of compression moulded and cured (180 $^{\circ}$ C) plaques.

Heat release rate (HRR) and smoke density (rate of smoke release, RSR) of EPDM compounds have been measured by Cone Calorimeter at a heat irradiation of 50 kW/m² (for more information on Cone Calorimetry see brochure "Mineral Based Flame Retardancywith metal hydrates"). The ratio at which heat is released is a good indication whether a fire will grow and how quickly. Keeping smoke density as low as possible during the infant state of a fire is critical for people to escape. Hence, both rates (HRR and RSR) should grow as slowly as possible and their peak values PHRR (Peak of Heat Release) and PRSR (Peak Rate of Smoke Release) should be as small as possible.

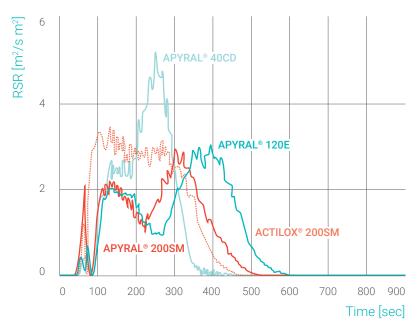
In the following diagrams (page 17, 18) the HRR and the RSR for the bright EPDM compounds are shown as an example. The test results differ de-

pending on the mineral flame retardant used. In general, the compounds with the finest aluminium hydroxide products and the highest specific surface areas (BET), APYRAL® 120E and APYRAL® 200SM, lead to a significantly less intense heat release and smoke emission compared to the reference compound with APYRAL® 40CD.

The EPDM compound containing the boehmite ACTILOX® 200SM, even though it has the highest BET surface area of all fillers compared, releases more heat and smoke. HRR and RSR curves for this compound are between the APYRAL® 40CD containing compound and the best performing materials based on APYRAL® 120E and APYRAL® 200SM. This is due to the lower amount of crystal water released from boehmite compared to aluminium hydroxide.



HRR, EPDM bright



Smoke density, EPDM bright

Ageing of EPDM compounds

Compounds which are used for transport or building applications have to stay functional over long periods of time. Therefore, tests are done to evaluate the influences of ageing on the compound properties according to national and international standards. Depending on the application of the compound, different test media and temperatures are used to accelerate possible ageing processes of the material. This is necessary for an estimation and evaluation of the lifetime of a material under specific environmental conditions.

The effects of hot air ageing were investigated for the earlier described black and bright EPDM compounds. The specimens were stored at 100 °C for 168 hours (7 days) and after that the mechanical properties tensile strength and elongation at break were determined again. Due to the hot air ageing, the mechanical properties of the compounds are influenced, as demonstrated in the diagrams on the next page. The mechanical characteristics of the black, sulphur cured compounds with 155 phr mineral content are reduced after hot air ageing.

In the black sulphur cured EPDM compound the use of the submicron sized APYRAL® 200SM results in a better stability against ageing. The tensile strength and the elongation at break are less affected by hot air ageing if APYRAL® 200SM is used as mineral flame retardant in this compound.

The use of APYRAL® 120E in the bright peroxide cured compounds results in a better retention of the mechanical properties of the compound after ageing, compared to the other mineral flame retardants in these tests. This effect has an even stronger impact because the mechanical characteristics of the compound containing APYRAL® 120E had already been better before the ageing.

The suitability of fine precipitated **APYRAL®** products for the use in EPDM is supported by this data.

A good balance of all properties of a compound is required. Its mechanicals, flame retardancy, and ageing resistance must be adjusted according to the requirements for the intended use.

The broad range of available **APYRAL**® and **ACTILOX**® products offers the user flexibility for the formulation of an appropriate compound.



Change of the mechanical properties of the **black** EPDM conpounds after ageing (100 °C, 7 days)

Change of the mechanical properties of the **bright** EPDM compounds after ageing (100 °C, 7 days)

EVM based compounds

Ethylene-vinylacetate-co-polymers are a diverse group of materials. They can either be used as thermoplastic materials (EVA), or those grades with a high vinyl acetate content (VA) (> 40 %) can be crosslinked to form elastomeric polymers (EVM, LEVAPREN®, ARLANXEO Deutschland GmbH).

As described in the introduction of this brochure, the M in the polymer description indicates the saturated polymer backbone of the molecules, the Methylene type of EVM. EVM based materials combine extraordinary ageing and media resistance with good flame resistance. This property profile makes them suitable polymers for a wide variety of applications. EVM based compounds are used for the production of cable sheathings or sealings. End uses are in public transport, the building sector or in oil rings

and e.g. marine applications. In those sectors, ever increasing demands for fire safety have to be met.

In the following, some effects of mineral flame retardants shall be demonstrated on the basis of EVM compound formulations.

The focus shall not only be on high flame resistance of the resulting compounds, but the balance of all compound properties must be considered, depending on the intended use.

In the following chapter data of different EVM compounds are shown. The data is sorted in dependence on the VA content of the EVM type, and on the fineness and the specific surface area of the APYRAL® products.

Formulation examples with EVM

The flame retardancy can be improved with increasing surface area (BET) of **APYRAL**® or by using EVM with increasing VA content. To show these effects in particular, the following EVM formulations were used.

Three EVM types from Arlanxeo (Deutschland GmbH, tradename: LEVAPREN®) were used for

this investigation: EVM 500, EVM 600 and EVM 700 with VA contents of 50, 60 and 70 %.

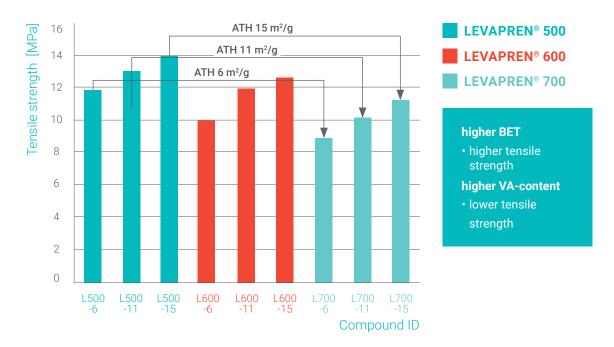
As mineral flame retardants fine precipitated APYRAL® products were chosen: APYRAL® 60 CD, APYRAL® 120E and APYRAL® 200SM.

The other components of the compound were not changed.

Component	and ID	L500 -6	L500 -11	L500 -15	L600 -6	L600 -11	L600 -15	L700 -6	L700 -11	L700 -15
LEVAPREN® 500 (VA = 50 %)	[phr]	100	100	100	_	_	_	_	_	_
LEVAPREN® 600 (VA = 60 %)	[phr]	_	_	_	100	100	100	_	_	_
LEVAPREN® 700 (VA = 70 %)	[phr]	_	_	_	_	_	_	100	100	100
APYRAL® 60 (BET = 6 m ² /g)	[phr]	160	_	_	160	_	_	160	_	-
APYRAL® 120 (BET = 11 m²/g)	[phr]	_	160	_	_	160	_	_	160	_
APYRAL® 200 (BET = 15 m ² /g)	[phr]	-	-	160	-	_	160	-	-	160
MAGLITE® DE (MgO)	[phr]	3	3	3	3	3	3	3	3	3
EDENOL® 888 (DOS)	[phr]	6	6	6	6	6	6	6	6	6
EDENOR® C 18 98-100	[phr]	1	1	1	1	1	1	1	1	1
AFLUX® 18	[phr]	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
GENIOSIL® GF 31	[phr]	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
RHENOFIT® TRIM/S	[phr]	1	1	1	1	1	1	1	1	1
PERKADOX® 14-40 B-PD	[phr]	6	6	6	6	6	6	6	6	6

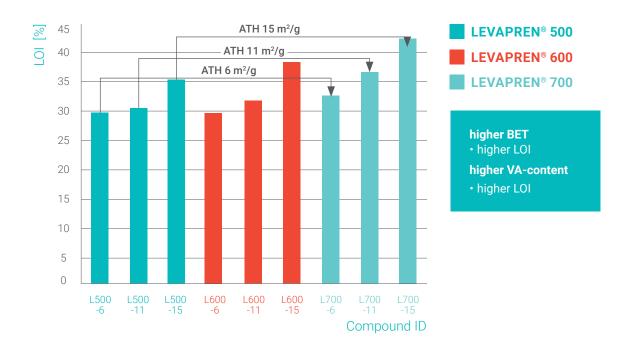
Within a certain VA (vinyl acetate) content of the polymer, the tensile strength increases with increasing specific surface area of the used **APYRAL**®.

By using a constant specific surface area, the tensile strenght decreases with increasing VA content oft the polymer.



Regarding the LOI as indicator for the resistance against ignitability of the compound, the following

chart shows interesting interrelations between BET and VA-content.



Using the same **APYRAL**® grade but different VA contents, it is shown that the inherent flame retardancy of EVM is improved, if the VA content of the polymer is higher. The same effect can be observed by increasing the specific surface area of the mineral filler. With both, increasing VA content or specific surface area of the used **APYRAL**®, the LOI can be increased.

An important fact is that even if the specific surface of **APYRAL**® increases, the media resistance of the

compound will be unaffected.

The following chart shows that the specific surface area of **APYRAL®** has no negative influence on the oil (IRM 902*, IRM 903*) swelling. To further reduce the swelling a polymer with higher VA content can be used.

Considering these correlations, it is easy to create tailor made compounds to balance the compound costs and the media resistance at given mechanical properties.



*FUCHS Lubritech GmbH, Germany

Ageing of EVM compounds

The retention of the mechanical property profile, even after long time use, is a decisive criterion for the selection of a certain compound. The required lifetime of cables, e.g. offshore cables for wind-mills or oil rigs is many years and the harsh environmental conditions (heat, cold, salt water) pose an additional challenge.

Stringent tests are necessary to evaluate the suitability of a compound for such applications.

The aging behaviour of an EVM compound with 60 % VA was analysed by means of oil- and water-based test media. Compounds made of the above used APYRAL® grades and a mixture of APYRAL® 40CD and APYRAL® 200SM were investigated. The specimens were stored for 168 hours (7 days) at 135 °C in hot air and at 100 °C in calcium bromide solution. After that, the mechanical characteristics were determined again and the changes compared to the data of the virgin compound were calculated in %.

These investigations show that the use of **APYRAL®** products with a higher specific surface area slightly reduces the ageing resistance of EVM compounds in water based media. Also the elongation at break of the compounds is reduced after ageing.

As the elongation at break was already lower for the virgin compounds which contained very fine APYRAL® products instead of APYRAL® 60CD, this effect has an even stronger impact. But this influence can be reduced, if for example **APYRAL® 40CD** with lower BET is added to the submicron sized **APYRAL® 200SM**. An optimal balance of all compound properties, even after ageing, must be fulfilled according to the actual requirements.

The broad range of available **APYRAL**® products offers the user flexibility for the formulation of an appropriate compound.



Change [%] of elongation at break after ageing (100 °C / 7 days - 135 °C / 7 days), EVM 600-compounds, 160 phr **APYRAL**®

Silicone based compounds

Silicone rubber can be classified by its appearance and its curing behavior. Several classes of silicones are defined. RTV silicones are cured at room temperature, for example whereas in HTV silicones this reaction only occurs at elevated temperatures.

Silicone rubber is a specialty elastomer and it is used when its specific properties are necessary

and offer an advantage for the user. This is true for example for special fire resistant cables in building applications which must stay functional even during and after a fire. Such cables are used for applications in elevators, alarming or fire exit lighting.

To achieve the required properties, ceramifying minerals can be added to the silicone during compounding, like for example alumina [3].

Another application field of silicone rubber compounds is high voltage insulators. In these compounds, aluminium hydroxide (**APYRAL**®) is used to increase the life time of the insulator by reducing the impact of overvoltage [4].

Silane treatments of the mineral can be applied to improve the compounding behavior and the compound properties further.

Vinyl silane and hydrophobic silanes are recommended as treatment for **APYRAL**® which is compounded into silicone rubber.

In a joint project with Hoffmann Mineral GmbH, Neuburg, Germany, silicone compounds were prepared and analysed. These compounds contain **APYRAL®** as well as products from Hoffmann Mineral [5].

In this brochure, those compounds shall be described and discussed which contain 100 phr of different **APYRAL®** products. Silicone rubber with a Shore A hardness of 40 was used and the compounding was done on a lab two roll mill. The peroxide curing was done at a temperature of 115 °C over 5 min and after this, the materials were tempered at 200 °C for 4 hours.

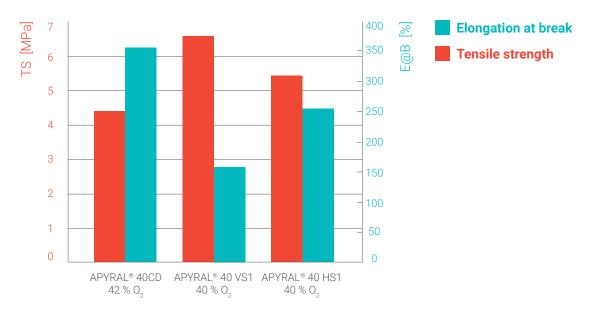
For these experiments, APYRAL® 40CD (aluminium hydroxide without silane), APYRAL® 40 VS1 (with vinyl silane), and APYRAL® 40 HS1 (with a hydrophobic silane) were used.

In the following tables and diagrams some properties of the silicone compounds are shown.

Formulation examples with silicone

Component [phr]		APYRAL® 40CD	APYRAL® 40 VS1	APYRAL® 40 HS1	
Silicone Elastosil® R 401/40		100	100	100	
Curing: Elastosil® AU	IX Vernetzer E	1.5	1.5	1.5	
APYRAL® 40CD		100	-	-	
APYRAL® 40 VS1		-	100	-	
APYRAL® 40 HS1		-	_	100	
Total		201.5	201.5	201.5	
Characteristic data		APYRAL® 40CD	APYRAL® 40 VS1	APYRAL® 40 HS1	
Mooney viscosity (M (ML (1+4) 100 °C)	IU)	25	26	23	
Tensile Strength	[MPa]	4.4	6.6	5.4	
M 500	[MPa]	2.4	3.6	2.8	
Elongation at Break	[%]	357	158	256	
Shore A, 23 °C	(dimensionless)	62	69	64	
LOI	[% O ₂]	42	40	40	

Compounding was done on a lab two roll mill. Samples were cut out of plaques which were cured at 115 $^{\circ}$ C and tempered at 200 $^{\circ}$ C.



Mechanical properties and LOI, 40 shore a silicone compounds 4h tempered

Good mechanical properties are achieved when **APYRAL® 40CD** is used. Optimisations of the me-

chanical properties can be achieved when silane coated **APYRAL®** products are used.

Ageing of silicone compounds



Change of mechanical properties of silicone compounds with 100 phr APYRAL® after hot air ageing

Hot air ageing tests were performed to evaluate the influence of ageing on the mechanical properties of the silicone compounds. The compounds were stored at 200 °C over a period of 168 h (7 days) and after this process, the mechanical properties were analysed again.

The changes of the mechanical properties due to ageing are lower when silane coated **APYRAL®** is used as mineral flame retardant. The best stability of the mechanical properties can be observed when **APYRAL® 40 VS1** is used as flame retardant filler in these compounds.

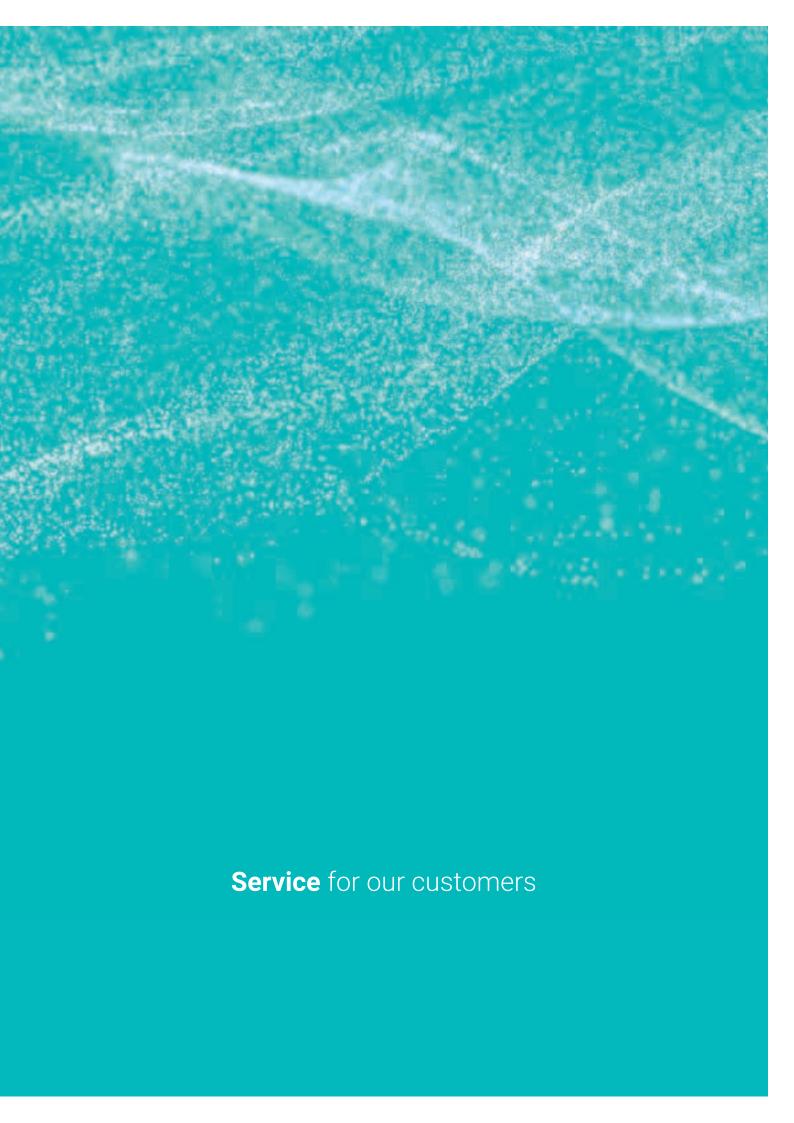
Annex

List of abbreviations

Abbreviation	Meaning
BET	specific surface area according to Brunauer, Emmett, Teller
HRR	heat release rate
HTV	high temperature vulcanizing (silicon rubber)
LOI	limiting oxygen index
phr	parts per hundred parts of resin
RTV	room temperature vulcanizing silicone (silicone rubber)
TTI	time to ignition

List of references

[1]	Ehrenstein, G.W., Pongratz, S., "Beständigkeit von Kunststoffen", Hanser-Verlag, 2007.
[2]	Schnetger J., "Lexikon Kautschuktechnik", Hüthig-Verlag, 2004.
[3]	http://www.google.com/patents/DE4437596A1?cl=en (9-2019)
[4]	Morgan, A.B., Wilkie, C.A., "Non-Halogenated Flame Retardant Handbook", Wiley, 2014, https://www.researchgate.net/publication/3258527_The_role_of_inorganic_fillers_in_silicone_rubber_for_outdoor_insulationAlumina_tri-hydrate_or_silica
[5]	http://www.hoffmann-mineral.de/content/download/25708/232929/version/12/file/NKE (9-2019)



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.

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

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Nabaltec

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

APYRAL® AOH

Boehmite, as flame retardant filler and functional filler

NABACAST®

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ACTILOX®

Boehmite, as flame retardant filler and catalyst carrier

APYRAL®

Aluminium hydroxides, as flame retardant and functional filler

GRANALOX®

Ceramic bodies, for the production of engineering ceramics

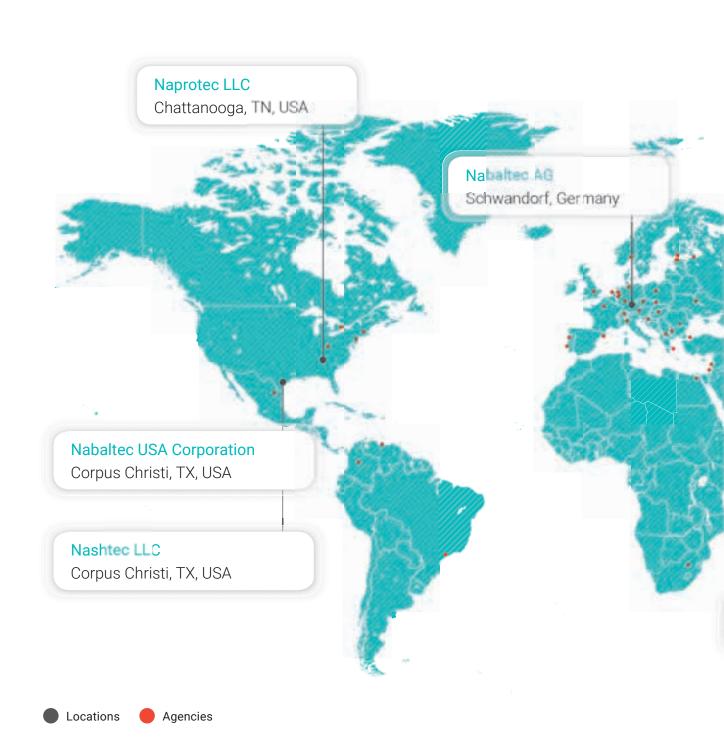
SYMULOX®

Synthetic sintered Mullite, for the production of e.g. refractory products

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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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