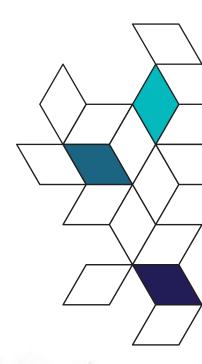
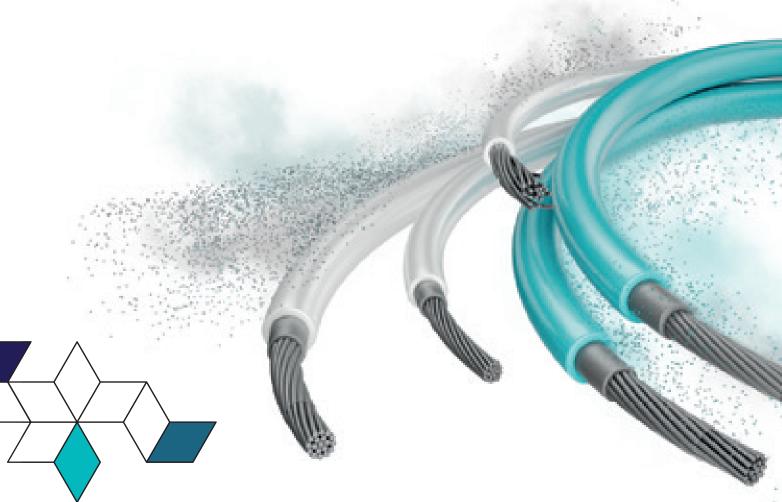
Metal hydrates for cables

APYRAL®, ACTILOX® B







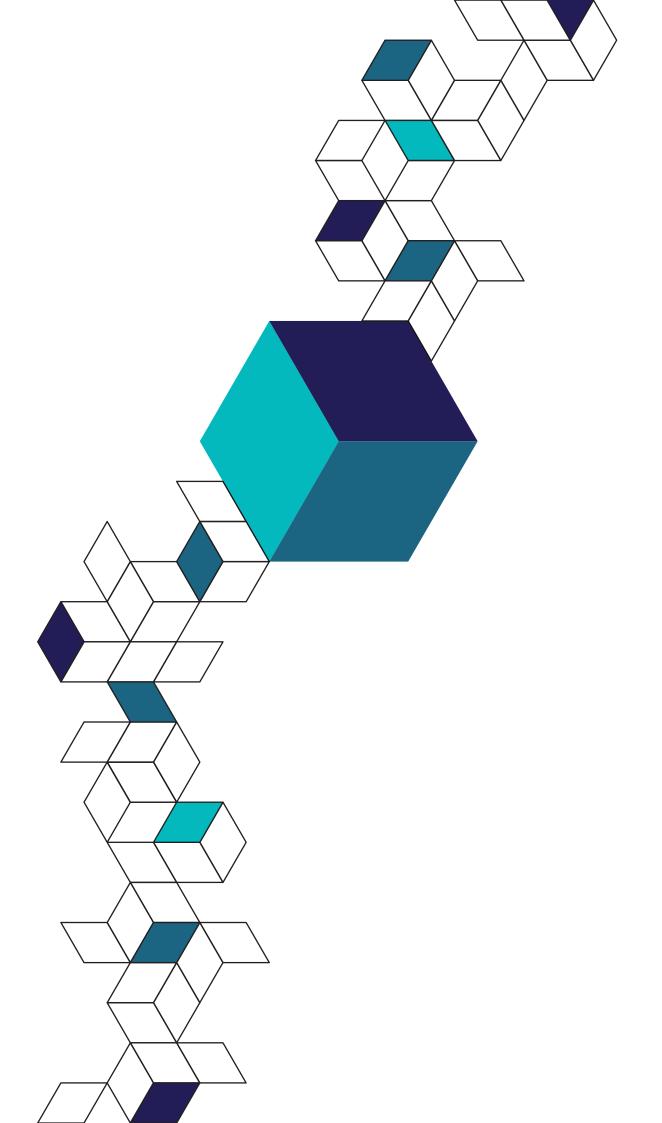
Metal hydrates for cables

Product	D50* [µm]	BET [m²/g]	Oil absorption [ml/100g]	Main application
APYRAL® – Ground				
APYRAL® 8	20	1.3	24	Bedding / Filling compounds
APYRAL® 16	21	1.8	17	Beduing / Filling Compounds
APYRAL® - Fine-pr	ecipitated			
APYRAL® 40CD	1.8	3.5	22	Thermoplastic
APYRAL® 60CD	1.0	6	28	Insulation compoundsSheathing compounds
APYRAL® 120E	0.9	11	37	Elastomers
APYRAL® 40T1	1.4	4.0	27	Sheathing compounds
APYRAL® 40 VS1	1.6	3.5	33	Crosslinked • Insulation compounds
APYRAL® 60 VS1	1.0	6	45	Silicone compounds
ACTILOX® B - Boel	nmite			
ACTILOX® B30	2.2	3	28	
ACTILOX® B60	0.7	5	30	
ACTILOX® 200SM	0.3	18	36	Insulation compoundsSheathing compounds
ACTILOX® PA-B2	Pr	opitary informat	cion	
ACTILOX® PA-14	Pr	opitary informat	cion	

^{*}Laser granulometer Microtrac S 3500

List of abbreviations

BET	Specific surface area
D50	Average particle size
E@B	Elongation at Break
EPDM	Ethylene-Propylene-Diene-Rubber
EVA	Polyethylene-co-Vinylacetate
EVM	Polyethylene-co-Vinylacetate with a high VA-content
FRLS PVC	Fire Retardant Low Smoke PVC
HFFR	Halogen Free Flame Retardant
L/D	Length versus diameter ratio (extruder parameter)
LLDPE	Linear Low Density Polyethylene
LOI	Limiting Oxygen Index
LSFOH	Low Smoke Free Of Halogen
MFI	Melt Flow Index
MPa	Megapascal
MVR	Melt Volume Rate
NBR	Nitrile-Butadiene-Rubber
PA	Polyamide
PBT	Polybutylene Terephthalate
PE	Polyethylene
PEgMA	Maleic acid anhydride grafted PE
PP	Polypropylene
PVC	Polyvinylchloride
rpm	Revolutions per min
Sb ₂ O ₃ / ATO	Antimony trioxide
SIR	Silicone elastomers (Silicone Rubber)
TPE	Thermoplastic elastomer
ТРО	Thermoplastic polyolefin
TPU	Thermoplastic polyurethane
TS	Tensile Strength
UL	Underwriters Laboratories
VA	Vinylacetate
XLPE	Crosslinked polyethylene



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Flame retardancy of cables

Wires and cables are present in our everyday life. Cables are found in buildings, cars and public transport vehicles as well as in all electric appliances.

Preventive fire protection of cables is a topic of ever increasing importance since forty years now.

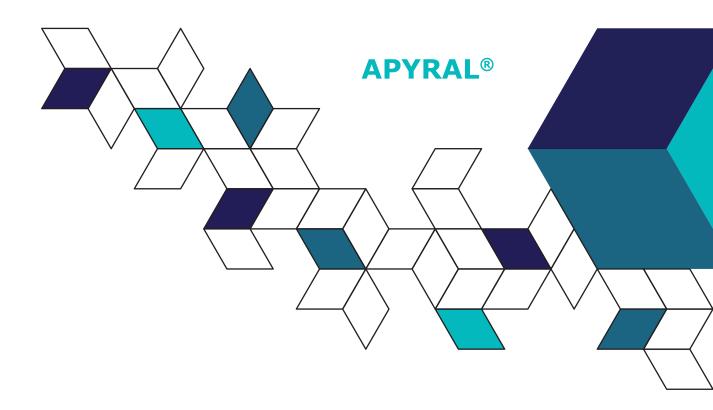
Especially in buildings, spread of fire by cables is an important issue. Electrical, machinery and data cables found in buildings can be several kilometers long. They are installed horizontally within individual levels and penetrate multistorey buildings vertically.

Flame retarded cables can prevent a small cause, such as an electrical short circuit, from becoming a major fire catastrophe which can result in significant material damages and even loss of lifes.

Over the years, metal hydrates, especially aluminium hydroxide and magnesium hydroxide have been established as the most important flame retardants for the wire and cable industry.

In particular their environmental friendliness and their favourable price performance ratio make **APYRAL**[®], aluminium hydroxide and **ACTILOX**[®] **B**, aluminium oxide hydroxide (boehmite) important and sustainable flame retardants.

APYRAL®	AI(OH) ₃
Chemical	Aluminium hydroxide
Mineral	Gibbsite
Common name	Aluminium trihydrate
Abbreviation	ATH
APYRAL® AOH ACTILOX® B	АІООН
	AIOOH Aluminium oxide hydroxide
ACTILOX® B	
ACTILOX® B Chemical	Aluminium oxide hydroxide





Our **APYRAL**® products for wire and cable applications can be divided into two classes on the basis of property profiles:

- · fine precipitated grades
- · ground grades

Fine precipitated **APYRAL**® products are broadly used to manufacture flame retardant insulation and especially sheathing compounds.

Of highest importance are **APYRAL® 40CD**, **APYRAL® 60CD** and **APYRAL® 120E** used in halogen free flame retardant (HFFR) cable compounds, also called LSFOH (Low Smoke, Free Of Halogen) cable compounds.

APYRAL® 40 VS1 and **APYRAL® 60 VS1** are vinylsilane treated products, especially used in crosslinked LSFOH compounds based on EPDM, EVM and SIR (silicone rubber).

Additionally, increasing the flame retardancy of PVC cable compounds and especially their smoke reduction is another important wire and cable application for fine precipitated **APYRAL**® products.

The ground grades **APYRAL® 8** and **APYRAL® 16** find widespread use in highly filled flame retardant bedding compounds.

APYRAL® products have a very high chemical purity of at least 99.5 %. The remaining constituents consist mainly of 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 silica. Due to its whiteness **APYRAL**® behaves neutrally to the colouring of polymers. The Mohs hardness of 2.5 – 3 causes no problem even in highly filled molten masses. The relatively high heat capacity c_p of 1.65 J/g*K 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 retarded plastics is increased compared to the unfilled polymer. However, **APYRAL**® is one of the most attractive flame retardants, even on the basis of a volume-specific cost balance.

Application				APY	RAL®			Polymers
	8	16	40CD	60CD	120E	40 VS1	60 VS1	
Bedding	•	•						EPDM, EVM, EVA, TPO
Insulation			•	•		•	•	PE, XLPE, EVA, PVC
Sheathing			•	•	•	•	•	PE, EVA, TPO, TPE, TPU, PVC, EVM, EPDM, NBR, SIR

Compounding

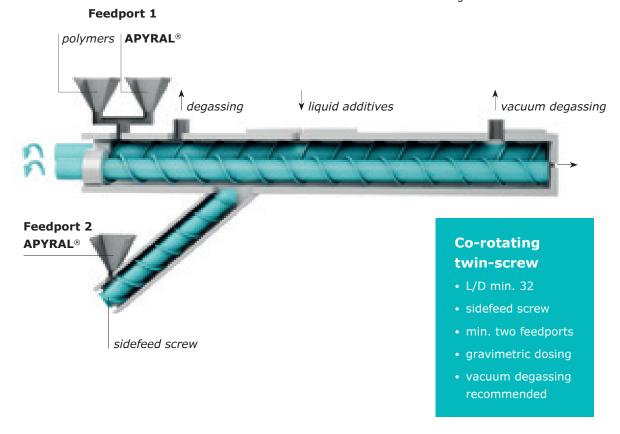
When producing **APYRAL**®-filled thermoplastics via melt compounding, special attention must be given to the process temperature limit of 200 °C.

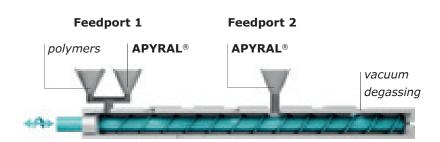
The temperature limit may vary in a marginal way depending on the residence time and the applied shear force during compounding in different compounding machines (see our brochure "Mineral Based Flame Retardancy with Metal Hydrates"). Therefore, it is recommended that the temperature of the molten mass is kept below 190 °C. In the case of continuous processing, higher temperatures are possible for short residence times. However, this option must be tested depending on the individual compound formulation and machinery settings. In general, temperature control must be as exact as possible.

In the manufacture of filled elastomers as well as thermoplastics, the internal mixer (kneader) has proved most flexible.

The continuous processes of the co-kneader and the co-rotating twin screw extruder offer better results in terms of dispersion and quality consistency. These compounding machines have excellent dispersing abilities thanks to their special designed screw geometry and special kneading elements, even in highly filled compounds.

The twin screw extruder should have an L/D ratio of at least 32 and two feeding ports. In order to ensure constant filling levels, gravimetric dosing of **APYRAL®** is recommended. The bulk of the filler should be applied via Feedport 1. Vacuum degassing combined with a number of ventilation openings ensure a good ventilation of the molten mass and the removal of volatile organic substances.





Co-Kneader

- L/D min. 11
- min. two feedports
- gravimetric dosing
- vacuum degassing recommended

Contrary to the co-rotating twin screw extruder, the co-kneader features one single screw which is put into an axial oscillation additional to the screw rotation. The compound is kneaded and transported through intermitting kneading elements and hence dispersed in a smooth oscillating manner. The co-kneader should have an L/D ratio of min. 11 and two feeding ports.

2/3 of the **APYRAL**® is generally applied via the first port along with the polymer. For the dosing of fluid additives the injecting between the first and the second feed port through a hollow kneading bolt has proved most effective.

Vacuum degassing before entry of the molten mass into the discharge screw is recommended, particularly when using silanes. In principle, screw geometries with low compression should be used for both continuous compounding processes. Specific screw designs are based on the compound formula, degree of filling and the **APYRAL**® grade used.

Recently, it became more common to use co-kneaders with higher L/D ratios (up to 22), especially for the production of silane crosslinkable HFFR compounds in a onestep procedure.

LSFOH compounds

Low smoke free of halogen (LSFOH) compounds are sometimes based on elastomers such as EPDM or NBR, but much more frequently on polyolefines, in particular PE and its copolymers.

In Europe permanently installed cables are classified according to CPR (Construction Products Regulation, EU 305/2011). For cables, EN 50575 defines the different classifications. The most important test method is EN 50399, which is a vertical cable bundle test with integrated measurement of temperature, heat and smoke evolution. Reaction to fire, resistance to fire and release of dangerous

substances are the main characteristics in the European as well as in international standards for flame retardant cables.

In order to pass these tests, compounds filled with 60 – 65 wt.-% of **APYRAL**[®] must be used.

Alongside the material properties, determined by the polymer and flame retardant used, the combustion behaviour in these cable tests, however, depends to a great extent on the respective cable geometry and cable design. Thus, the quoted filling levels are only guidelines.

Compounds for insulation and sheathing

Because of its excellent insulation properties, crosslinked low density polyethylene (XLPE) is by far the most important coating material used to prevent short circuiting of electrical conductors. XLPE is also the most used insulation material for flame retardant cable constructions. Anyhow, LSFOH compounds based on fine precipitated **APYRAL**® are also applied in wire insulation.

APYRAL[®] grades have a very low electrolyte content which guarantees for the lowest possible impact on compound insulation properties.

Most important is the use of LSFOH compounds as outer sheathing of cable constructions. This enables very effective fire prevention against external ignition sources.

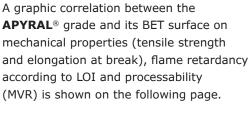
Influence of BET-surface area

The table next page shows the formulation as well as the main characteristic data for three fine precipitated **APYRAL**® grades in a test compound based on an EVA typ with a VA-content of 26 %.

The most important parameter to distinguish between the fine precipitated **APYRAL**® grades is the specific surface area according to BET. Ranging from 3.5 m²/g for **APYRAL® 40CD** and 6 m²/g for **APYRAL® 60CD** up to 11 m²/g for **APYRAL® 120E**, the BET surface area is covering a broad span.

With increasing specific surface area of the mineral flame retardant a decrease in elongation at break but an increase in tensile strength is observed.

The melt index, cited in the table as Melt Volume Rate (MVR), is an important value to estimate the processing behavior of compounds during extrusion of insulated conductors or cables. The high value for APYRAL® 60CD and especially APYRAL® 40CD is remarkable and allows the design of fast extrudable LSFOH compounds.



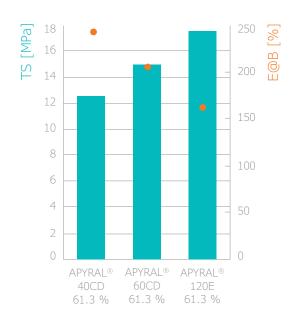


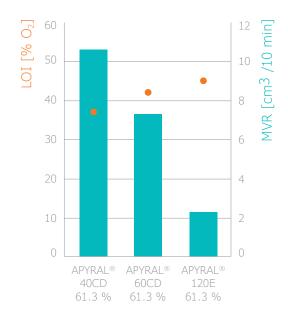
EVA (26 % VA-content) and Aminosilane.

Component [wt%]		APYRAL® 40CD	APYRAL® 60CD	APYRAL® 120E
EVA, Escorene™ UL 00226	38.3	38.3	38.3	
Aminosilane, Dynasylan® AMI	0.4	0.4	0.4	
APYRAL® 40CD		61.3	_	-
APYRAL® 60CD		-	61.3	-
APYRAL® 120E		-	_	61.3
Characteristic data				
Tensile Strength (TS)	[MPa]	12.6	15	17.6
Elongation at Break (E@B)	[%]	243	206	163
MVR at 190 °C / 21.6 kg	[cm ³ /10 min]	10.6	7.3	2.3
LOI	[% O ₂]	37	42	45

Compounding on internal lab kneader. Samples made of compression moulded plaques.

Mechanical properties, MVR and LOI (EVA with 26 % VA-content)





Mechanical properties with increasing BET

- increase in tensile strength
- decrease in elongation at break

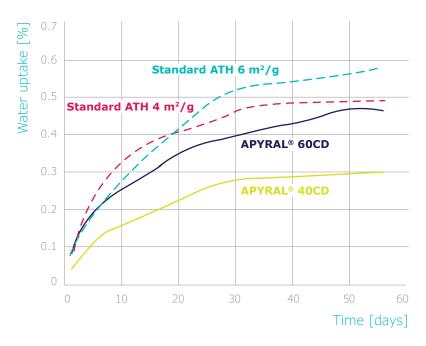
Flame retardancy and processability with increasing BET

- increase in LOI
- decrease in MVR

Water uptake

Water uptake of LSFOH compounds is essential for insulated wires. But also when used as sheathing material, a low water uptake is crucial to pass critical ageing tests performed under water bath conditions.

The diagram on the left displays the water uptake for an EVA compound based on **APYRAL® CD** grades and standard ATH grades of similar fineness.



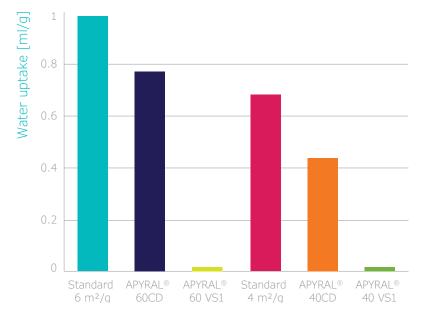
APYRAL® CD – technology

 low water uptake without surface coating

Water uptake of EVA, Escorene UL00119 filled with 61.3 wt.-% ATH / APYRAL®; T = 23 °C.

APYRAL® CD results in significantly reduced water uptake compared to standard ATH grades of comparable BET surface. The reason for this is related to

the comparably low water uptake of the **APYRAL® CD** powders, which is shown in the second graph.



With decreasing BET, decrease of filler water uptake

By hydrophobic coating further decrease possible

APYRAL® VS1

 lowest water uptake by vinylsilane coating

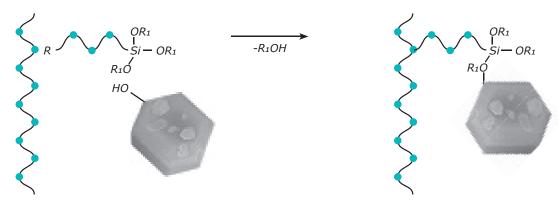
Water uptake of ATH / APYRAL® powder according to Baumann values of saturation after 30 min.

Coupling agents

The use of coupling or bonding agents to achieve good physical properties in highly filled LSFOH compounds is elementary. Coupling agents enable a binding between inorganic **APYRAL**® filler and polymer. The most preferred way of binding is by chemical reaction, but strong enough physico-chemical reaction is also feasible.

Coupling with maleic-acid-anhydride grafted polymers

Coupling via bifunctional organosilanes



 $R = NH_2$ (H-bridging and covalent bonding on polar groups within polymer) R = (covalent bonding on polymer, peroxide initiated) $R_1 = CH_3$, C_2H_5

Alongside the use of polyolefines grafted with maleic anhydride (in case of PE abbreviated as PEgMA), the use of bifunctional organosilanes, in particular with amino or vinyl end group, is the most widely used technology.

Both product groups function as chemical mediators between the mineral with a polar, hydroxide groups carrying surface and the polymer. The illustration above shows the basic chemical mechanisms.

The reaction of the silane with the hydroxide surface releases ethyl- and/or methyl alcohol which must be removed.

Organosilanes are usually used in an amount of 1 % of the overall filler mass. In the case of PEgMA the dose is a few percent of the overall composition.

While PEgMA products are generally supplied in pellet form, organosilanes are liquid. If dosing of very small quantities of fluids is non favourable, they can also be used as a master batch on a porous polymer carrier.

Alternatively, compounders may use precoated fillers, like **APYRAL® 40 VS1** and **APYRAL® 60 VS1**. In this case the silane is already bound to the metal hydrate surface.

Exemplary formulations

PE / EVA and vinylsilane / peroxide

The compound formulation listed below shows a simple thermoplastic recipe based on PE / EVA and vinylsilane. Peroxide concentration is high enough to graft the vinyl groups onto the polymer backbone, but small enough to avoid crosslinking of polymer chains.

predecessor product (standard 4 m²/g ATH). The significantly reduced values for diehead pressure and diehead melt temperature for APYRAL® 40CD enable an approximate 30 % increase in extrusion speed.

APYRAL® CD technology

This compound formulation is used to demonstrate the benefits of APYRAL® CD technology during cable extrusion.

The diagram on page 16 shows important parameters for the extrusion of an insulated copper wire.

APYRAL® 40CD is compared to its

diehead [m/min] extruder die head

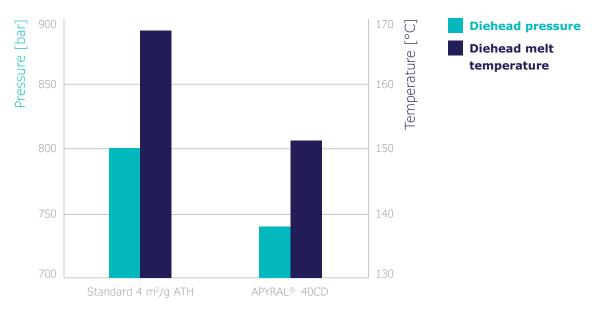
polymer extrudate

Compounding on Buss-Co-Kneader. Samples made of extruded tapes.

Component	[wt%]	Trade name
LLDPE	9.66	ExxonMobile™ LL 1004 YB
EVA	29	Escorene™ UL 00226
Vinylsilane	0.8	Silquest™ FR-693
Process aid	0.3	Silquest™ PA-826
Peroxide	0.04	Interox® TMCH-75-Al
Stabiliser	0.2	Irganox® 1010
Aluminium hydroxide	60	APYRAL® 40CD
Characteristic data		

Characteristic data

Tensile Strength	[MPa]	11
Elongation at Break	[%]	260
MVR at 160 °C / 21.6 kg	[cm³/10 min]	9.4
LOI	[% O ₂]	37



Diehead pressure and diehead melt temperature at:

- 0.5 mm² copper wire (single, round)
- die diameter 1.4 mm
- insulation thickness approx. 0.35 mm
- line speed 650 m/min

PE / EVA with PEgMA

Compounding in Werner & Pfleiderer dispersion kneader (kneading chamber); Specimen made of compression moulded plaques.

Component		[wt%]	Trade name
LLDPE		10	ExxonMobile™ LL 1004 YB
EVA		24.8	Escorene™ UL 00226
PEgMA		5	Compoline CO/LL
Stabiliser		0.2	Irganox® 1010
Aluminium hydroxide		60	APYRAL® 40CD / 60CD
Characteristic data		APYRAL® 40CD	APYRAL® 60CD
Tensile Strength	[MPa]	12.6	13.6
Tensile Strength Elongation at Break	[MPa] [%]	12.6 227	13.6 214
-		-	

Just like the previous exemplary LSFOH compound, the formulation listed on page 16 is based on a blend of PE and EVA. In this case, maleic anhydride grafted PE (PEgMA) was used as coupling agent.

This compound results in very good balanced physical properties for both **APYRAL® CD** grades. The LOI of the PEgMA coupled compound is slightly reduced in comparison to the vinylsilane coupled formulation on page 15.

Furthermore, differences in the melt viscosity (given as MVR) of the compounds are observable using the same ATH grade but different coupling agents. Besides the fundamental difference in coupling agent and the comparison of the characteristic compound properties, one has to consider that the above values have been examined on compounds produced on a discontinuous lab kneader and specimen, which have been cut out of compression moulded

plaques instead of extruded tapes. This important influence of the compounding technology and specimen preparation is demonstrated for the following basic formulation below.

A polymer blend of PE and a PE-copolymer (TPO) was used, comprising a filler loading level of 65 wt.-% and a PEgMA coupling agent. The characteristic data generated on specimen made of compression moulded plaques and compounds produced on the lab kneader are excellent for both APYRAL® CD grades.

For APYRAL® 40CD it is demonstrated that compounding equipment and technology used for specimen preparation have a significant influence on the final properties. The extruded tapes result in further improved tensile and elongation balance and compound pellets produced by co-kneader give higher MVR values.

PE / TPO with PEgMA

Influence of compounding equipment on mechanical properties of LSFOH compounds.

Component		Trade	name	[wt%]
LLDPE		ExxonMobile™	[™] LL 1004 YB	13.3
TPO (PE-co-octene)		Engage™ 8452		17.5
PEgMA		Fusabond® E MB 226 D		4
Stabiliser		Irganox	® 1010	0.2
Aluminium hydroxide		APYRAL® 40	OCD / 60CD	65
Compounding		Buss-Co-Kneader	Internal mixer	Internal mixer
Specimen		Extruded tape	Plaques	Plaques
Characteristic data		APYRAL	.® 40CD	APYRAL® 60CD
Tensile Strength	[MPa]	16.8	13.8	14
Elongation at Break	[%]	268	244	188
MVR at 160 °C / 21.6 kg	[cm ³ /10 min]	4.2	2.6	2.5
LOI	[% O ₂]	34	33	33

EVA / EVM with aminosilane (non crosslinked)

The compound below is based on an EVA with 26 % VA-content blended with an EVM grade. EVM are also copolymers of VA and ethylene, but comprising very

high VA-contents (in this case 60 %). These copolymers show rubber like properties. EVM accepts very high filler loads, but has a plasticizing effect.

Examples for non crosslinked flame retardant EVA / EVM compounds with aminosilane.

Component		Trade	name	[wt%]
EVA		Escorene [⊤]	[™] UL 00226	29
EVM		Levamelt® 600		5.6
Aminosilane		Dynasyla	an® AMEO	0.4
Aluminium hydroxide		APYRAL® 40CD	/ 60CD / 120E	60
Characteristic data				
Characteristic data		APYRAL® 40CD	APYRAL® 60CD	APYRAL® 120E
Characteristic data Tensile Strength	[MPa]	APYRAL® 40CD 6.1	APYRAL® 60CD 10.3	APYRAL® 120E 14.3
	[MPa] [%]			
Tensile Strength		6.1	10.3	14.3

Compounding on lab dispersion kneader. Specimen made of compression moulded plaques.



APYRAL® 120E most preferred for rubers and rubber-like compounds.

Consequently, compounds containing high contents of EVM have to be crosslinked to achieve sufficiently good tensile properties. Therefore, mostly silica and/or carbon black are added as reinforcing fillers.

With a moderate level of EVM it is sufficient to counterbalance by the right choice of **APYRAL®** grade.

When using **APYRAL® 40CD** the tensile strength is far too low for the non-crosslinked compound.

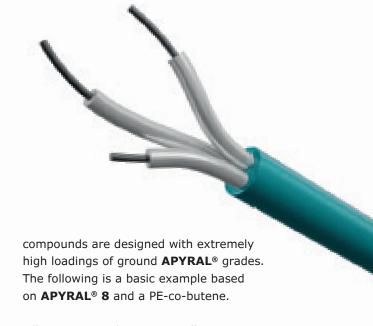
In contrast, for APYRAL® 60CD and especially APYRAL® 120E the higher specific surface area of these products shows big advantages for such types of LSFOH compounds.

APYRAL® 60CD and APYRAL® 120E are the most preferred grades for elastomer compounds on the basis of NBR, EPDM, and EVM as well as extremely soft thermoplastic polymer blends e.g. based on copolymers of acrylic esters and ethylene.

Bedding / Filling compounds

Electrical cables are usually designed with a filling mass surrounding the individual insulated conductors and thus filling the empty space between the insulated conductors and the cable sheath. These bedding compounds are also mineral filled. In order to design flame retarded cables with lowest possible fire load, APYRAL® is also used here, partially or fully substituting calcium carbonate.

The mechanical requirements for a filling mass are very low so that such



Filling compounds are generally based on PE-co-alkenes, EPDM or EVA with a high VA-content.

Examples for flame retardant bedding compounds.

Component	[wt%]	MVR (190 °C/21.6 kg)	LOI	
PE-co-butene (Engage™ ENR 7380)	15			
PE-Wax (Licowax® PE 520)	4	10.6 cm ³ /10 min	62 % O ₂	
Stearic acid	1	10.6 cm ³ /10 min		
APYRAL® 8	80			

Compounding on lab dispersion kneader. Specimen made of compression moulded plaques.

Elastomer compounds (crosslinked)

On the next page EVM based elastomer compounds with a VA-content of 70 % are exemplarily shown. Compared to the commonly used APYRAL® 60CD and APYRAL® 120E grades, submicron sized APYRAL® 200SM shows an advantageous balance of elongation at break and tensile strength combined with a high flame retardant efficiency given as LOI (see graph next page). The submicron sized particles

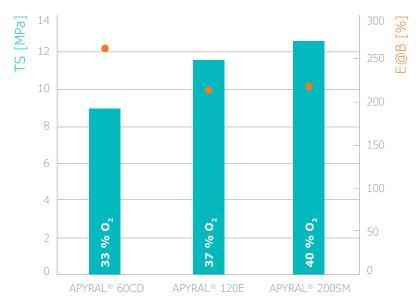
and the corresponding high specific surface area of **APYRAL® 200SM** is pivotal to a flame retardant boost effect reflected in the remarkable high LOI of 40 %.

Additional information regarding elastomers and representative compound formulations are given in our brochure "Metal Hydrates in Elastomers".

Elastomer Compound / EVM with 70 % VA-content.

Component [phr]		APYRAL® 60CD	APYRAL® 120E	APYRAL® 200SM
EVM, LEVAPRENE® 700		100	100	100
Zinc Borate		10	10	10
Plasticiser (mix)		9.9	9.9	9.9
Stabiliser (mix)		4	4	4
Crosslinker (mix)		7	7	7
Adhesive		1.6	1.6	1.6
APYRAL® 60CD		160	_	-
APYRAL® 120E		_	160	_
APYRAL® 200SM		_	-	160
Characteristic data				
Tensile Strength	[MPa]	8.9	11.6	12.6
Elongation at Break [%]		264	214	220
MVR at 160 °C / 21.6 kg [cm ³ /10 min]		32	36	45
Shore A (at 23 °C) –		75	77	81
LOI	[% O ₂]	33	37	40

Technical data given after crosslinking, except MVR viscosity.



Mechanical properties and LOI of EVM 700 compounds with 160 phr ${\bf APYRAL}^{\otimes}$.

Process aid - ACTILOX® PA-14

The benefits of APYRAL® CD technology on processability of LSFOH compounds, especially the tremendous increase in the line speed compared to standard fine precipitated ATH grades, has already been discussed on page 16. To enable even better processing of LSFOH compounds Nabaltec AG has developed a processing aid designed to ease extrusion, but avoiding an increase in diehead temperature.

ACTILOX® PA-14 is a powdery process aid based on mineral carriers and silicones added at 0.5 – 2 wt.-% during compounding.

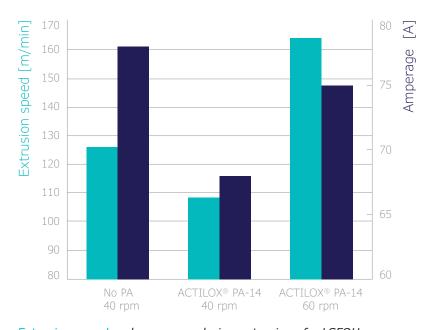
The formation of "die drool" is a principle problem of highly filled compounds. It describes the leakage of resin around

the nozzle area of the steel block through which the wire is extruded.

The diagram displays extrusion speed and amperage of the pilot plant extruder used.

When using **ACTILOX® PA-14** at 1.2 wt.-% and keeping extruder revolution at 40 rpm, amperage is significantly reduced, but unfortunately also the line speed (insulated copper with 3 mm diameter).

At increased extruder revolution (60 rpm) the potential of **ACTILOX® PA-14** is fully utilized. The line speed increases by 30 % while amperage remains at the same level as for the reference compound not containing any process aid.

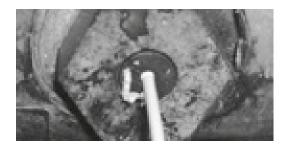


Extrusion speed and amperage during extrusion of a LSFOH compound; Left without, middle and right with ACTILOX® PA-14.

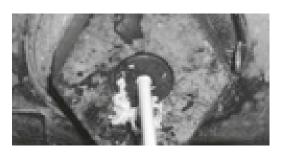
After 3 min.



After 6 min.



After 9 min.



Drifting and dropping "die drool" during extrusion of a LSFOH-compound containing **ACTILOX® PA-14**.

When the "die drool" has achieved a critical size and mass, it can be drawn away by the insulated wire and often causes failures within the insulation. Out of specification production and extruder shut downs are the unavoidable consequence.

The addition of **ACTILOX® PA-14** very effectively retards "die drool" formation. Additionally the resin residue formed is not conically closed around the nozzle, but forms on one side of the die only. When growing it slowly drifts away from the nozzle and finally drops. A potential pick up by the insulated wire can be avoided. This effect is displayed by the three images.

Flame retardant booster - ACTILOX® PA-B2

Nabaltec has developed a flame retardant synergist **ACTILOX® PA-B2** to overcome even severe flame retardancy requirements as they are described in standards like CPR (Construction Products Regulation).

ACTILOX® PA-B2 is a white, powdery masterbatch containing metal hydrate and siloxane. It can be characterized by its high temperature stability and excellent polymer compatibility. Furthermore, it is easy to blend with all known mineral fillers. In basic

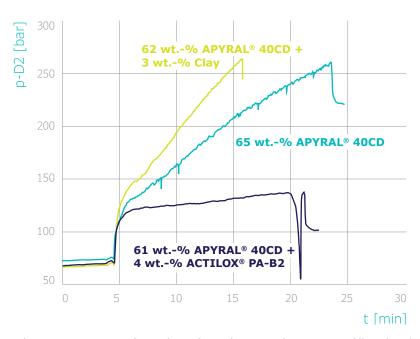
compound formulations **ACTILOX® PA-B2** showed synergistic effects in combination with ATH resulting in improved flame retardancy and hot air aging performance while providing a reduced pressure build-up during processing. The addition of **ACTILOX® PA-B2** even improves the compound processability, unlike other types of mineral synergists like organoclays.

Next figure shows the positive influence of the new **ACTILOX® PA-B2** on the

processing behavior. A filter pressure test was performed using a PE/EVA formulation with 65 wt.-% filling level. The effect of the filler was investigated using pure APYRAL® 40CD

or the following two combinations:

APYRAL® 40CD with ACTILOX® PA-B2
and APYRAL® 40CD with an organoclay.
The optimal ratios of the fillers
have been chosen for the test.

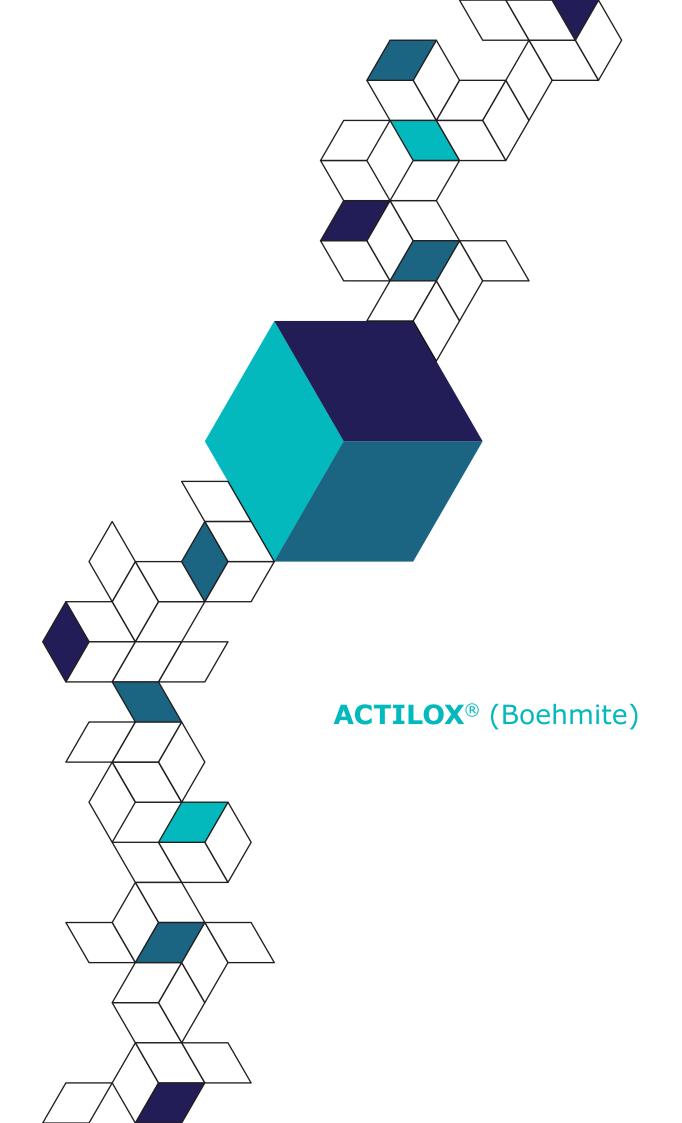


Filter pressure test of a PE/EVA formulation with 65 wt.-% filling level.

Additional to investigation of the processing behavior, the effect of **ACTILOX® PA-B2** on flame retardancy was studied. As can be seen in the table below, the PE/EVA formulation with 65 wt.-% **APYRAL® 40CD** cannot pass the UL 94 V test at 1.6 mm. To obtain a UL 94 V0 classification the filling level has to be increased up to 70 wt.-%. However, this

high filling level leads to a deterioration of the mechanical properties and compound processability (low MVR). The combination of **APYRAL® 40CD** and **ACTILOX® PA-B2** can easily be classified as UL 94 V0 while still maintaining good mechanical properties and good processing behavior. For more details please refer to our whitepaper "Processing Aid and Flame Retardancy Booster".

	Unit	65 wt% APYRAL® 40CD	70 wt% APYRAL® 40CD	61 wt% APYRAL® 40CD + 4 wt% ACTILOX® PA-B2
Tensile Strength	[MPa]	14.9	15.4	14.0#
Elongation at Break	[%]	150	112	168
MVR (190 °C/21.6 kg)	[cm ³ /10 min]	3.6	0.9	2.8
LOI	[% O ₂]	42	46	42
UL 94 V (V0/V1/V2/not classified)		NOT CLASSIFIED	V0	VO
Shore D		58	60	55



ACTILOX® (Boehmite)

ACTILOX®, boehmite, is finding an everincreasing range of new applications as functional filler, especially when higher temperature stability is required. Hence, highmelting compounds with process temperatures of up to 340 °C can be processed using **ACTILOX**®.

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

ACTILOX® can be processed up to 340 °C. Therefore it can be used in TPOs and TPEs.

Furthermore **ACTILOX**® grades are applicable in technical compounds based on PA, PBT etc. In such polycondensates strongly alkaline fillers such as magnesium hydroxide can lead to hydrolytic decomposition of the polymer chains.

Therefore **ACTILOX**® is much more favourable for polycondensates due to its relatively low basicity and moisture content.

Boehmite application in cable compounds.

	ACTILOX ®		OX®		
Application	B30	B60	200SM	Polymers	
Insulation	•	•		PP, PP-copolymers, PE, XLPE, EVA	
Sheathing	•	•	•	PP, PP-copolymers, PE, XLPE, EVA, TPO, TPE, EVM, EPDM, NBR	

Its high heat capacity (c_p =1.54 J/gK at 500 K, 227 °C) combined with high temperature stability make **ACTILOX**® boehmites ideal for use in electrical insulation heat sinks.

Our boehmites show very low electrolyte contents. This predestines **ACTILOX**[®] for electrical insulation applications.

Its Mohs hardness of 3 causes no tool abrasion problems even in highly filled molten masses. With a specific density of 3 g/cm³, **ACTILOX**® is a medium dense mineral filler.

Additionally, **ACTILOX**® shows a very good chemical resistance, especially a very high acid resistance.

Solubility in battery acid.

Mineral filler	Dissolved fraction in 34 % sulphuric acid (H ₂ SO ₄)		
APYRAL® 40CD Aluminium hydroxide	8 %		
ACTILOX® B30 Boehmite	0.2 %		

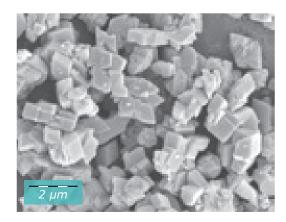
The **ACTILOX**® grades differ in their specific BET surface area and median particle size D50.

The fineness increases from ACTILOX® B30 (BET= $3 \text{ m}^2/\text{g}$, D50 = $1.7 \mu\text{m}$) via ACTILOX® B60 (BET= $5 \text{ m}^2/\text{g}$, D50 = $0.9 \mu\text{m}$) to ACTILOX® 200SM (BET= $18 \text{ m}^2/\text{g}$, D50 = $0.3 \mu\text{m}$).

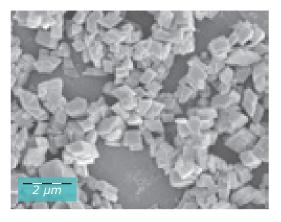
ACTILOX® B30 and ACTILOX® B60 are characterized by an excellent processability. This is due to the unique cubic like morphology of the boehmite crystals these two ACTILOX® B grades consist of. This morphology leads to low viscosities and causes a very good dispersability in the polymer matrix respectively in the cable compound. Therefore ACTILOX® B30 and ACTILOX® B60 are especially destinated for fast extrudable LSFOH compounds, whenever high line speeds and high extrusion temperatures are required.

ACTILOX® 200SM has proved being effective in increasing flame retardancy of LSFOH compounds when used together with **APYRAL®** and magnesium hydroxide. This combination of mineral fillers improves the ash or char stability as well as the LOI in a given compound (see pages 27 – 28).

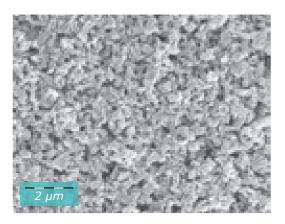
The table on page 28 shows a simple compound based on an EVA grade with a VA-content of 19 % and the most important compound data for the three different **ACTILOX**® boehmite grades.



SEM image of ACTILOX® B30.



SEM image of ACTILOX® B60.



SEM image of ACTILOX® 200SM.

EVA with aminosilanes

Examples for flame retardant EVA compounds with aminosilane.

Component [wt%]		ACTILOX® B30	ACTILOX® B60	ACTILOX® 200SM	
EVA, Escorene™ UL 00119	38.3	38.3	38.3		
Aminosilane, Dynasylan® Al	0.4	0.4	0.4		
ACTILOX® B30		61.3 –		-	
ACTILOX® B60		_	- 61.3		
ACTILOX® 200SM	_	_	61.3		
Characteristic data					
Tensile Strength [MPa]		12.5	16.8	18	
Elongation at Break [%]		219	221	165	
MVR at 190 °C / 21.6 kg [cm³/10 min]		4.5	3.5	0.5	
LOI [% O ₂]		26	27	34	

Compounding on lab dispersion kneader. Specimen made of compression moulded plaques.

The trends observed for mechanical properties and processability (MVR) are comparable to the BET dependant relationships found for fine precipitated **APYRAL**® (see page 12).

For flame retardancy according to LOI, only a small influence of BET surface difference between **ACTILOX® B30** and **ACTILOX® B60** is detectable. Anyhow, the explicitely higher BET surface of

ACTILOX[®] **200SM** results in a significant LOI increase.

Generally, boehmites of the **ACTILOX**® product range result in lower LOI values compared to fine precipitated **APYRAL**® at identical loadings. For more detailed information of such general considerations on flame retardancy, we kindly request to refer to our brochure "Mineral Flame Retardancy with Metal Hydrates".

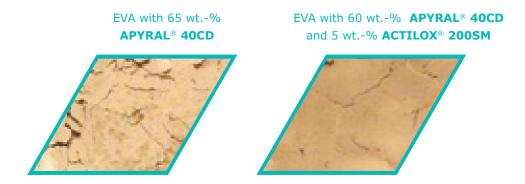
Synergistic effects with ACTILOX® 200SM

ACTILOX® 200SM as char forming agent

When using **ACTILOX® 200SM** as a co-additive to **APYRAL®**, the remaining char after combustion forms a very homogenous, nearly fully closed surface as the below comparison of the residues of two plaques tested in the cone calorimeter shows. During the start of a fire, chars serve as a

protective shield against heat and retard the release of volatile and flammable decomposition products on the incipient thermal breakdown of the polymer matrix. This shielding effect and especially transport inhibition is the more successful the more closed and stable the char layer is.

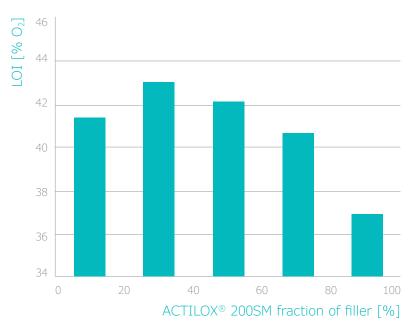
Char / ash formation after Cone-Calorimeter.



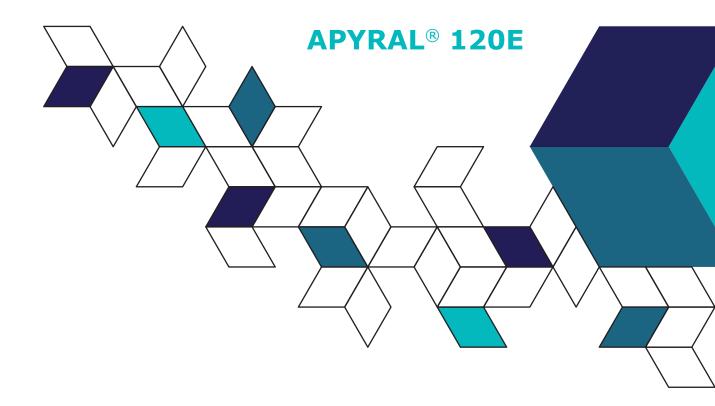
Increased LOI / Improved flame retardancy

As mentioned before, combinations of ACTILOX® 200SM and APYRAL® exhibit a flame retardant efficiency that each individual component will not achieve at comparable filling levels. This synergistic effect is given in the graph below, showing a successive substitution of APYRAL® by ACTILOX® 200SM. Obviously this

combination passes an effective maximum at a ratio of 30 % + 70 % (related to an overall filling level of 61.3 wt.-% in an EVA compound). At this ratio, the LOI increases by 3.5 % compared to the sole use of **APYRAL® 60CD**. Only when the boehmite content exeeds 80 %, the LOI drops below the reference values (ATH without boehmite).



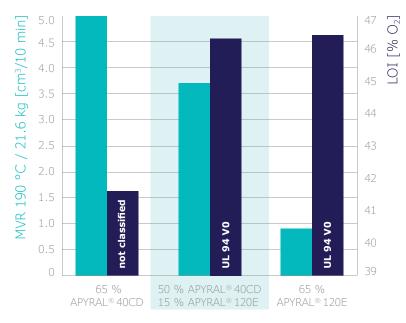
LOI as a function of the mineral filler composition (total filling level 61.3 wt.-%); LOI development by a successive exchange of APYRAL® 60CD with ACTILOX® 200SM in an EVA compound.



APYRAL® 120E as synergist

The optimal combination of specific surface area (BET) and particle size makes APYRAL® 120E an ideal synergist for the afore mentioned APYRAL® products. To achieve even better flame retardancy without the need to increase the amount of APYRAL® in the compound which often results in an unacceptable decrease in the mechanical data, APYRAL® 40CD can be combined with our APYRAL® 120E. While APYRAL® 40CD alone achieves no UL 94 V classification, both APYRAL® 120E and the combination of APYRAL® 40CD and APYRAL® 120E reach a UL 94 VO classification at 1.6 mm. However, the processability of all compounds differs significantly. While the compound with

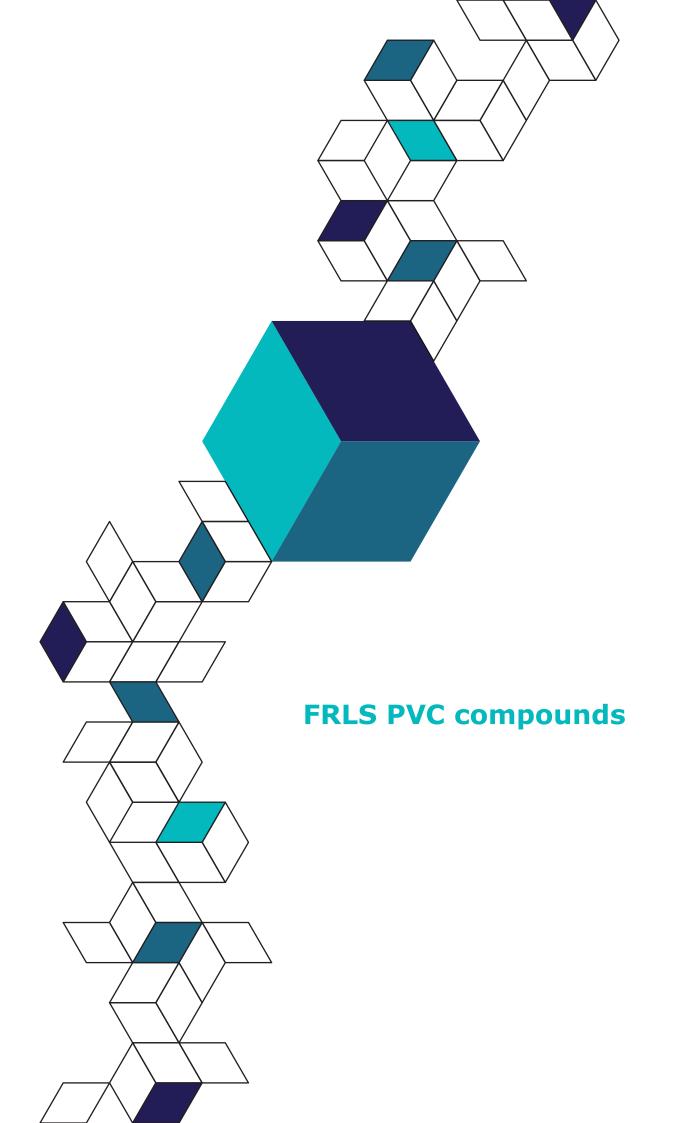
APYRAL® 40CD gives a high MVR correlating with very good processability, the compound filled with APYRAL® 120E is difficult to process, hence giving a very low value for MVR. The combination of APYRAL® 40CD and APYRAL® 120E provides a very good compromise with regards to processing. When using APYRAL® 120E the LOI is significantly increased compared to APYRAL® 40CD. However, the same LOI level can be reached when combining APYRAL® 40CD with **APYRAL® 120E**. Thus, extraordinary good flame retardancy can be realised without increasing the filling level of a compound avoiding the negative influence on the compound properties this would cause.



High fire resistance combined with good processability. LOI, MVR and UL94-V0 at 1.6 mm for EVA filled with 65 wt.-% APYRAL®.

Combination of APYRAL® 40CD and APYRAL® 120E

- UL94 V0 at 1.6 mm
- good processability



FRLS PVC compounds

Examples for flame retardant and smoke suppressed PVC compounds.

Component		[phr]	[phr]	[phr]
PVC, K = 70 (Evipol® SH	7020)	100	100	100
Plasticizer, DINP		50	50	50
Ca/Zn-Stabilisator (Bäropan R 8850KA-5)		5	5	5
PE-Wax (A-C 6A)		0.5	0.5	0.5
Flame retardant		5	50	50
		ATO	APYRAL® 40CD	APYRAL® 60CD
Characteristic data				
Tensile Strength	[MPa]	16.5	13.8	14.0
Elongation at Break [%]		210	230	220
LOI [% O ₂]		30	30	30

Compounding on lab dispersion kneader. Specimen made of compression moulded plaques.

Smoke suppressed PVC compounds

Plasticized PVC is still by far the most widespread material in the cable sector and is used for the production of insulations but mainly for cable sheaths. PVC intrinsically has relatively low flammability and is self extinguishing when a small flame is applied. A major disadvantage with almost all halogenated materials is the generated black smoke in the case of fire.

Plasticised PVC filled with **APYRAL**® develops considerably less smoke than conventional flame retarded PVC with antimony trioxide (ATO) at the same ignitability (LOI values).

The table above shows an example of such a Fire Retardant Low Smoke PVC (FRLS-PVC). For APYRAL® 40CD and APYRAL® 60CD the characteristic values received are compared with a compound traditionally flame retarded using ATO. The mechanical properties of all compounds are very

good and easily fulfill all the requirements by common industry standards.

The diagram on the next page shows the maximum optical density of the three compounds listed in the table, received by smoke density measurements in accordance with ASTM E662.

APYRAL® filled compounds show reduced smoke density compared to ATO (Sb₂O₃) filled PVC at conditions resembling a smouldering fire (non flaming mode).

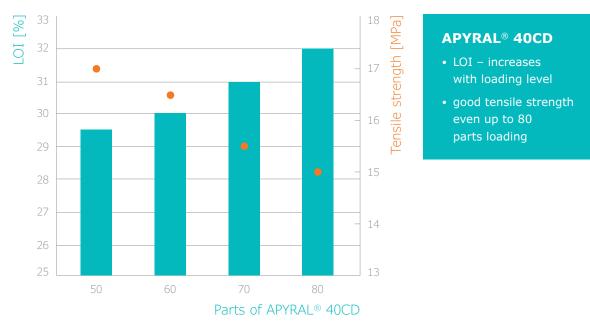
Much more considerable differences are recorded when a pilot flame is used (flaming mode, simulating an open fire). The compounds filled with **APYRAL**® metal hydrates have considerably reduced smoke values when an open fire breaks out (See more in our brochoure "Metal Hydrates for PVC").



Maximum smoke density according to ASTM E662 of plasticized PVC using different flame retardants.

The next diagram displays LOI and tensile strength for a plasticized PVC compound as a function of **APYRAL® 40CD** loading. LOI increases with increasing **APYRAL® 40CD** load.

The tensile strength of the compound drops, but remains at a very good level even up to 80 parts on 100 parts PVC and easily fulfils values required by common industry standards.



LOI and tensile strength of plasticized PVC at increasing APYRAL® 40CD loading.

Improved thermal stability of plasticized PVC compounds

A major industrial problem is the photosensitivity and low thermal stability of PVC during processing. Unfavorable processing parameters can lead not only to discoloration of the material, but also to a significant deterioration of mechanical and physical properties. Crosslinking in the polymer or even chain termination can occur and subsequently cause a change in molecular weight.

One of the most common degradation reaction is dehydrochlorination. This so called elimination reaction is autocatalytic and can be initiated by light or heat, whereupon hydrogen chloride (HCI) is released. In order to prevent thermal degradation during processing and protect the finished product against rapid aging, stabilizing agents like Ca/Zn-stabilizer need to be added.

As already highlighted in this brochure, APYRAL® can be used to improve the flame retardancy of plasticized PVC compounds. In addition, Nabaltec has developed the special surface modified APYRAL® 40T1 which combines the well-known flame retardancy with a significantly increased thermal stability of the PVC compound compared to other ATH grades.

A simple plasticized PVC formulation (see table next page) was used to demonstrate the stabilizing effect of APYRAL® 40T1 compared to APYRAL® 40CD and APYRAL® 40VS. It can be clearly seen that all three products achieve comparable good mechanical properties.

Product advantages

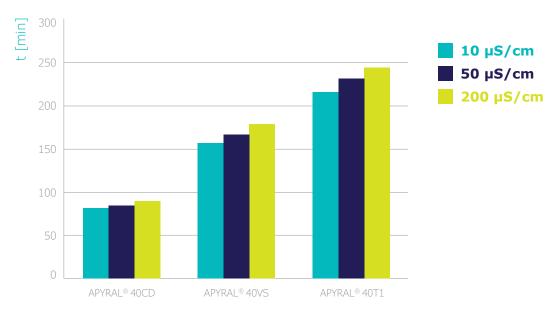
- · Good conveyance and dosing
- Very fine particles
- Excellent mechanical properties
- Improved thermal stavility in PVC
- Non-reacrtive surface treatment

The MVR shows a good processability of all three compounds however, the surface treated products perform even better than the untreated **APYRAL® 40CD**. Furthermore, the surface treatment leads to a significantly reduced water uptake of the compound.

The compound stability has been determined via dehydrochlorination test (DHC). During this test, the specimen are exposed to elevated temperature which induces the formation of HCI (dehydrochlorination reaction). The released HCl is fed into distilled water which leads to an increasing conductivity which is detected over time. So the longer it takes to reach a certain conductivity value, the better is the thermal stability of the compound. The results in the figure next page clearly show the significant stabilizing effect of APYRAL® 40T1 in plastiziced PVC compound. The stability time with APYRAL® 40T1 is almost three times higher compared to the compound with APYRAL® 40CD.

Stabilizing effect of APYRAL® 40T1 vs. APYRAL® 40CD and APYRAL® 40VS.

Components	[phr]	[phr]	[phr]	
PVC (Vinnolit S4170)	100.0	100.0	100.0	
Plasticizer, DINP	50.0	50.0	50.0	
Ca/Zn-Stabilizer (Bäropan MC 8	8.0	8.0	8.0	
APYRAL® 40CD	80.0	_	_	
APYRAL® 40VS	_	80.0	-	
APYRAL® 40T1	_	_	80.0	
Characteristic data				
Tensile strength	[MPa]	13.7	14.2	13.4
Elongation at break [%]		263	287	258
MVR (190 °C / 21.6 kg) [10 cm ³ /10min]		5.9	11.8	7.5
Water uptake (70 °C / 14 d)	1.21	1.03	0.94	



Stability measurement via dehydrochlorination (DHC) test of PVC compounds filled with 80 phr of various APYRAL® grades.

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 control and quality 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 filler 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

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Further information:

Nabaltec AG

P.O. Box 1860 · 92409 Schwandorf Phone +49 9431 53-0 www.nabaltec.de info@nabaltec.de

Customer Service

Phone +49 9431 53 910 sales@nabaltec.de

Technical Service

Phone +49 9431 53 920 tec-service@nabaltec.de

Nabaltec AG

P.O. Box 1860 · 92409 Schwandorf

Tel +49 9431 53-0 Fax +49 9431 61 557

www.nabaltec.de info@nabaltec.de

