

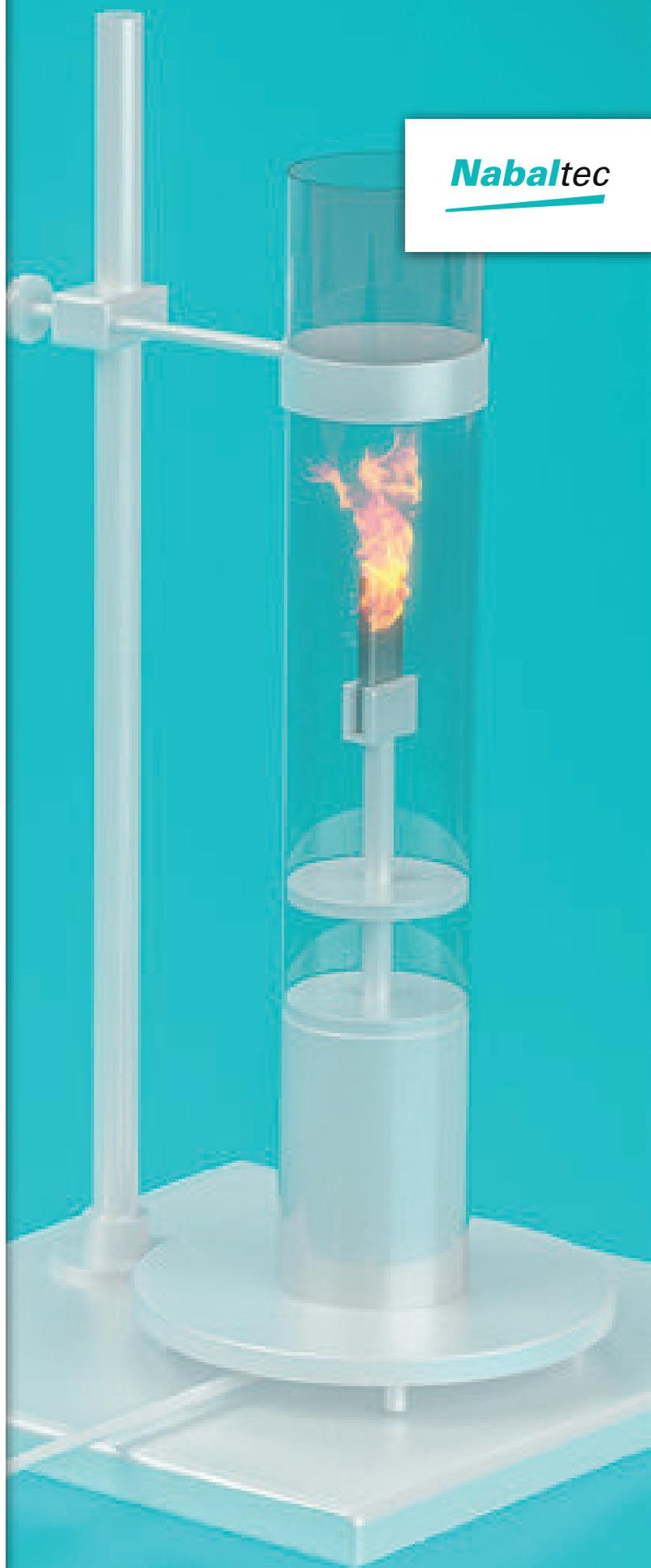
**Mineral based
flame retardancy
with metal
hydrates**

APYRAL[®]

APYRAL[®] AOH

ACTILOX[®] B

Nabaltec



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General considerations on flame retardancy

Why flame retardancy?

Due to their wide range of properties and the high degree of versatility they offer in terms of processing and moulding, plastics are now widely used in everything from everyday to high-tech applications. As most polymers are based on hydrocarbon framework, however, they are highly flammable. Thus, alongside the physical characteristics necessary for the specific application, fire protection requirements also have to be fulfilled. For this purpose flame retardant additives are added to the plastics during processing. Flame retardants protect modern polymer materials such as building insulation material, circuit boards and cables against combustion and the spread of fire by preventing or retarding the occurrence and propagation of flames. The decisive factors for flame retarded materials are a low combustibility, a late ignition point, the slowest possible propagation of flames and a low release of heat and smoke. This gives people more time to escape and flash-over can be avoided. Flame retarded plastics can prevent a small cause, such as an electrical short circuit, from becoming a major fire catastrophe which can result in major material damages and even loss of life.

| APYRAL® | Al(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 |
| pH | 8 - 9 |

Halogen free flame retardancy with metal hydrates

Flame retardants on the basis of metal hydrates, in particular aluminium and magnesium hydroxide, have established themselves over the years as by far the most important fire retardants.

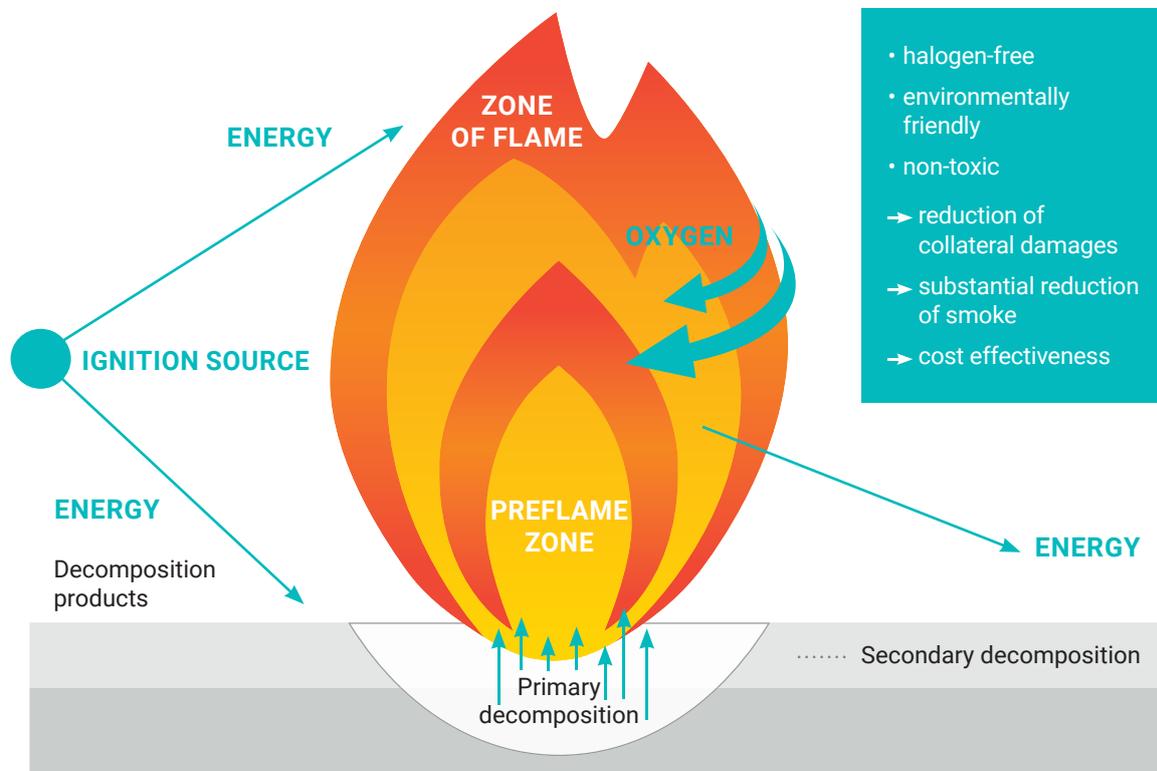
In particular their environment friendliness and their favourable price-performance ratio make **APYRAL®** (aluminium hydroxide), **APYRAL® AOH** and **ACTILOX®** (aluminium oxide hydroxide) sustainable flame retardants.

The following sections describe the flame retardant mechanisms of **APYRAL®** and **APYRAL® AOH / ACTILOX®** and explain their effects on the main combustion characteristics of plastics such as flammability, flame propagation, rate of heat release and smoke production.

The table below shows some of Nabaltec's halogen free flame-retardant fillers with their chemical composition, mineral and common name as well some typical physical data.

| APYRAL® AOH ACTILOX® | AlOOH |
|---------------------------------|-----------------------------|
| 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 |
| pH | 7 - 8 |

Mineral based flame retardancy with metal hydrates



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

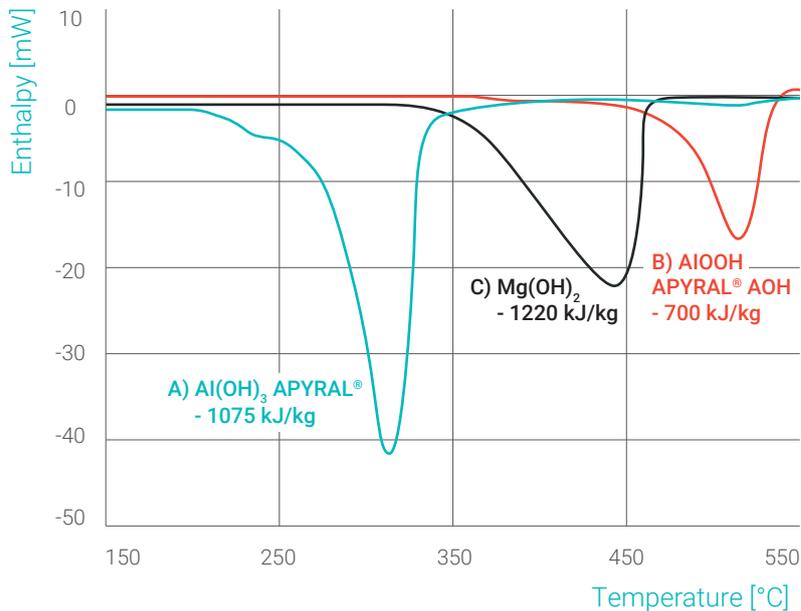
The decomposition reactions A to C including water release are characteristic for metal hydrates in the **APYRAL**[®] and **APYRAL**[®] AOH / **ACTILOX**[®] product lines. The performance of metal hydrates as flame retardants is based on physical and chemical processes. The schematic drawing above shows the involved processes of an **APYRAL**[®] / **ACTILOX**[®] filled polymer in case of a fire.

In the presence of an ignition source – a flame or a hot object – the thermal decomposition of the aluminium hydroxide into aluminium oxide and water takes place. During this process, energy is consumed from the ignition source, as the decomposition is an endothermic reaction.

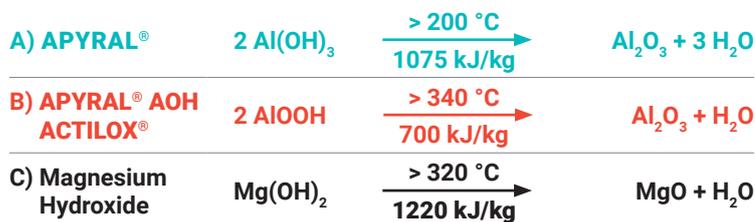
At the same time, the released water vapour is cooling the surface of the polymer and particularly dilutes the concentration of burnable gases in the surrounding area.

The remaining aluminium oxide residue has a high internal surface where sooty particles, respectively polycyclic aromatic hydrocarbons are absorbed. Additionally, the oxide layer acts as a barrier, disabling the further release of low molecular weight decomposition products as well as a heat barrier protecting the polymer against further decomposition.

Decomposition of metal hydrates



Differential scanning calorimeter data (DSC) of the mineral flame retardants **APYRAL®**, **APYRAL® AOH** and magnesium hydroxide **Mg(OH)₂**.



Decomposition reaction of metal hydrates

The diagram above shows the corresponding enthalpy curves (thermal energy consumption) during decomposition of aluminium hydroxide (**APYRAL®**), magnesium hydroxide and Boehmite (**APYRAL® AOH** / **ACTILOX®**) (recorded as DSC).

With the endothermic phase transition beginning at around 200 °C, **APYRAL®**, aluminium hydroxide, has the lowest decomposition temperature.

Magnesium hydroxide and Boehmite, **APYRAL® AOH** / **ACTILOX®**, have a considerably higher temperature stability. Thus, they enter into the reaction at higher temperatures and can consequently also be processed safely up to higher temperatures.

While **APYRAL®** and magnesium hydroxide consume similar quantities of energy per unit mass, Boehmite, **APYRAL® AOH** / **ACTILOX®**, has a substantially lower value.

Flammability / Limiting oxygen index

The „Limiting oxygen index“ (LOI) in accordance with DIN EN ISO 4589 is an indicative value for the assessment of flame retardancy and, in particular, the flammability of polymer materials. This describes the minimum concentration of oxygen in an oxygen/nitrogen mixture which is sufficient to support the combustion of a vertically oriented specimen.

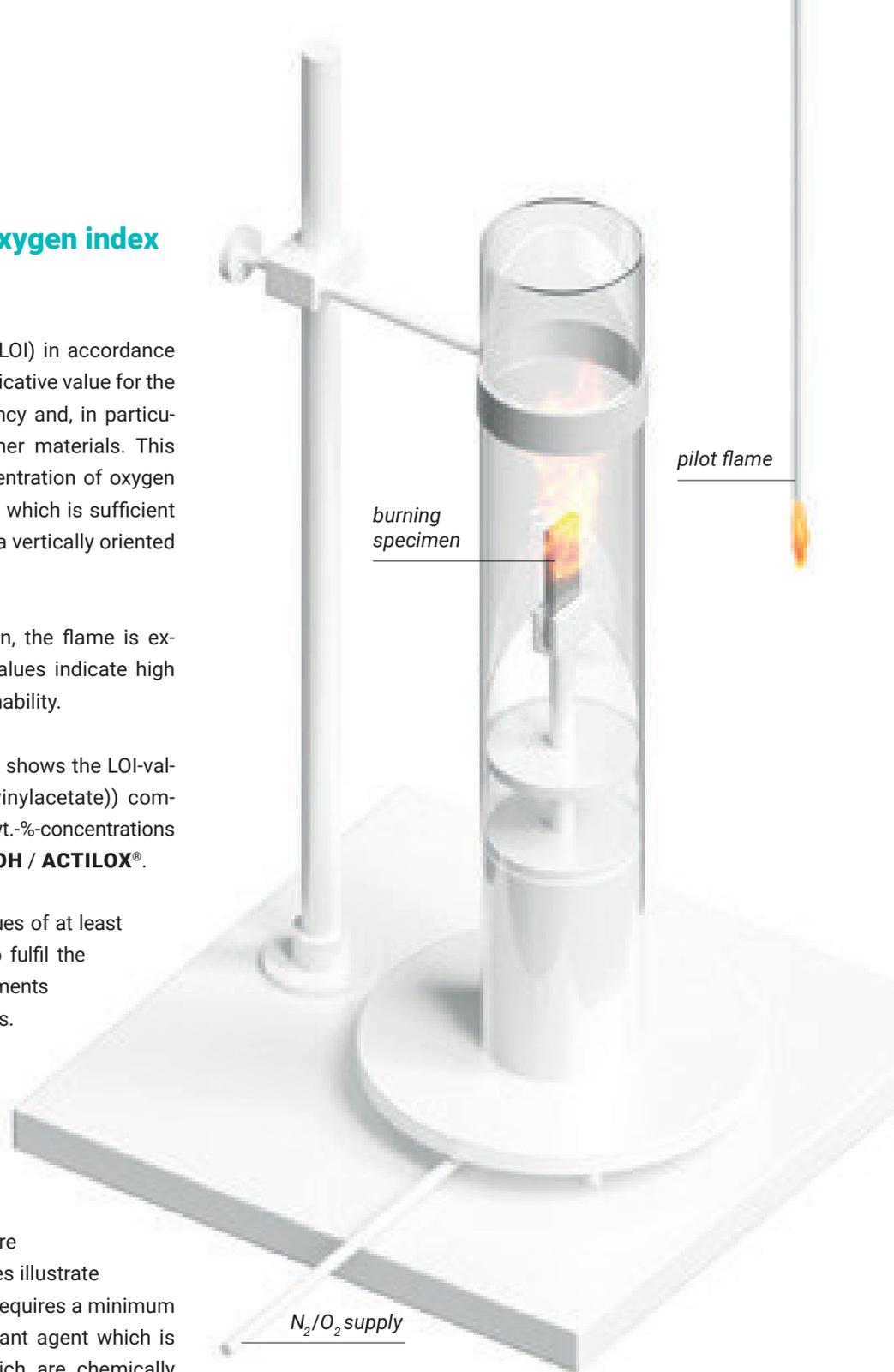
At lower oxygen concentration, the flame is extinguished. Hence, high LOI-values indicate high flame retardancy or low flammability.

The diagram on the next page shows the LOI-values for EVA (Poly(etyhyl-co-vinylacetate)) compounds filled with varying wt.-%-concentrations of **APYRAL®** and **APYRAL® AOH / ACTILOX®**.

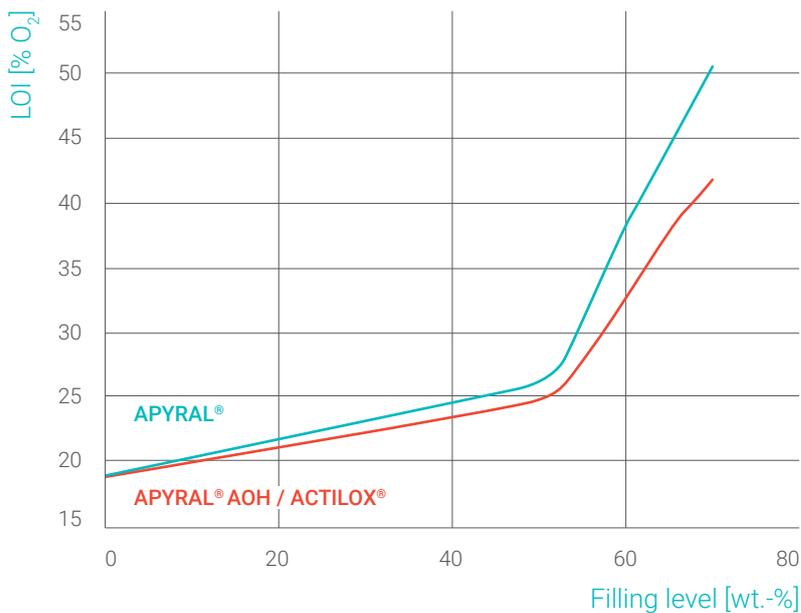
Experience shows that LOI-values of at least 30 % oxygen are necessary to fulfil the basic flame retardancy requirements in a wide range of applications. In the example given here this corresponds to a filling level of around 55 wt.-% in EVA.

To meet more stringent standards, far higher values are required. The initially flat curves illustrate that the physical mechanism requires a minimum concentration of flame retardant agent which is higher than for products which are chemically effective in the gas phase such as halogen and phosphorous-containing flame retardants.

If the filling level exceeds this minimum, even very small increases in filler loading cause remarkable increases of the LOI-values.

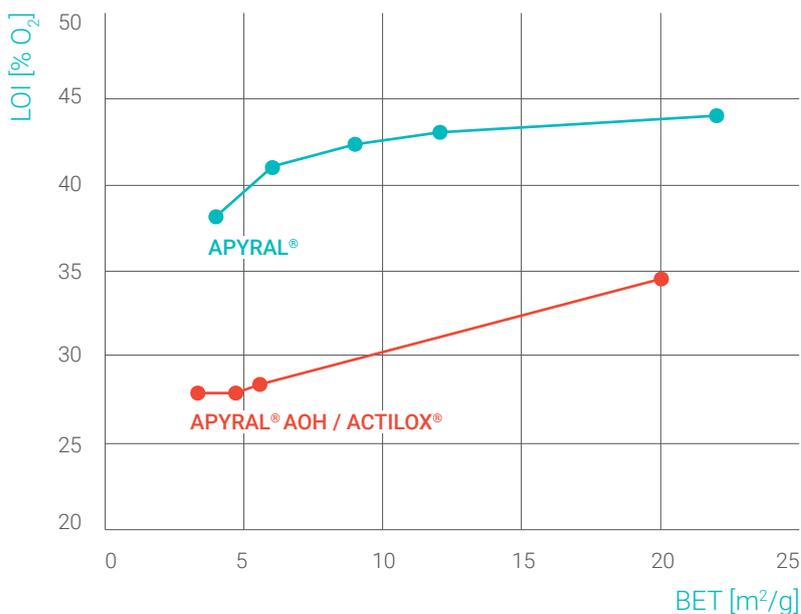


*Apparatus to measure LOI-values; (Source: Troitzsch J., *Plastics Flammability Handbook*, 3rd Edition, Hanser Publishers, Munich, 2004).*



- minimum filling load needed
- higher loading
- higher LOI
- higher loss on ignition
- higher LOI

LOI-values of EVA compounds plotted against the filler loading of metal hydrates.



- higher BET
- higher LOI
- higher loss on ignition
- higher LOI

LOI-values of EVA compounds (61.3 wt.-% metal hydrate) depending on the specific surface area of the metal hydrate.

However, the resulting oxygen index does not depend solely on the filling level or on the chemical composition of the metal hydrate (and of course the polymer matrix), but also to a large extent on the fineness and the specific surface area of the filler additive.

This relationship is shown for fine crystallized **APYRAL®** grades and for **APYRAL® AOH / ACTILOX®** types in the diagram above.

An EVA (19 % VA-content) was filled with 61.3 wt.-% of the corresponding mineral flame retardant. The LOI-values of the compounds produced via melt compounding were determined.

The smaller the particles are and the higher their specific surface area is, the higher is the LOI-value of the final compound.

This correlation is valid for all kinds of metal hydrates.



UL94 V-Test.

- high loadings
→ UL94 V0
- medium loadings
→ UL94 V1
- no correlation
between LOI and
UL94 V

Flame propagation / The UL94 V-test

Flame propagation is described by various flame retardant standards. The US standard UL94 has asserted itself for polymer materials applied in electronics. In the most common classification according to UL94 V, the specimens are tested in vertical orientation. A standard Bunsen Burner is used as ignition source. The test specimens are twice exposed to the flame and the afterburning time as well as any smouldering and dripping are used as rating criteria. The descending classification is V0, V1, V2 and non classified – UL94 V0 thus represents the highest level of fire protection. To reach the V0 classification in different polymer systems different **APYRAL**[®] filling loadings are required.

Experience shows that polymers which contain heteroatoms like oxygen in their molecular structure, are inherently flame retarded to a certain degree.

A certain level of flame retardancy can thus be reached using smaller amounts of flame retard-

ants than in pure hydrocarbons. The facts described here are summarised in the following chart (see p. 10). Different polymers were filled with **APYRAL**[®] aluminium hydroxide. The filler loading at which each polymer could be classified V0 according to UL94 (specimen thickness 3.2 mm) and the LOI at this filler loading were determined. These values are given in the table below. It is obvious that the polymers which contain heteroatoms (EP, UP, EVA) are classified V0 already at lower filler loading than the pure hydrocarbons PE and PP.

The LOI-values, also given in this table, are differing, although all compounds are classified V0. There is no direct correlation between LOI-values and UL94 classifications. A correlation between these two values is limited to evaluation within one polymer system.

Minimum APYRAL® filling levels to reach UL94 V0 (at 3.2 mm) and the LOI-values at these filling levels.

| Polymer system | Filler loading APYRAL® [wt.-%] | LOI [% O ₂] |
|------------------------------|--------------------------------|-------------------------|
| Epoxy resins | 55 | 39 - 45 |
| Unsaturated polyester resins | 58 | 35 - 50 |
| EVA | 62 | 38 - 45 |
| Polyethylene | 65 | 32 - 36 |
| Polypropylene | 67 | 30 - 32 |

Smoke suppression

All combustion is accompanied by smoke. Alongside the combustion conditions (intake of air, open fire or smouldering fire), the amount of smoke released depends to a large extent on the chemical composition of the materials involved. In the event of a fire, keeping the smoke density as low as possible can mean the difference between life and death. An escape route which remains visible for just a few more minutes can give many people the chance of saving their lives. Statistical analysis has shown that most victims die through inhalation of smoke and not as a result of the fire itself.* The lately implemented Construction Products Regulation (CPR;

EU-Regulation No. 305/2011) and the EN 45545 (fire protection in railway applications) set their additional focus on smoke toxicity to emphasize the importance of this topic.

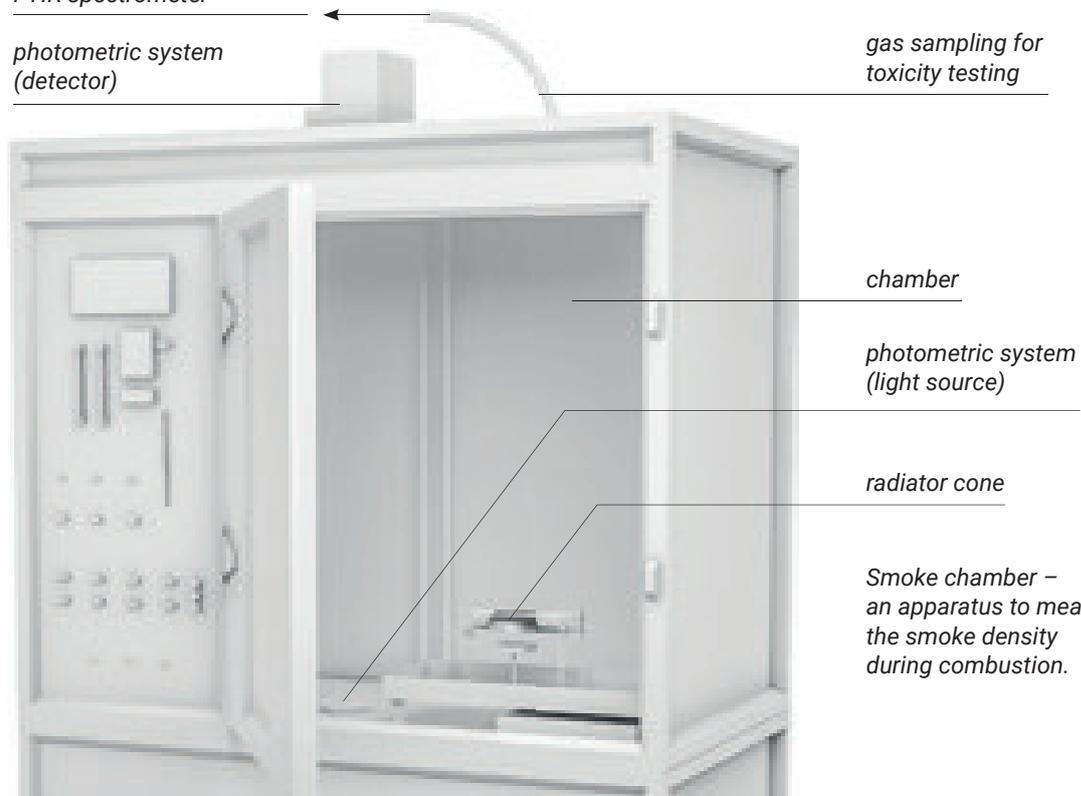
A common method for classifying materials in accordance with their smoke emissions in the event of fire is the smoke density determination in accordance with ISO 5659-2.

*Source: „Hazards of Combustion Products“; Interscience Comm. Ltd.; London; 2008 (ISBN: 978-0-9556548-2-4).

FTIR spectrometer

photometric system (detector)

gas sampling for toxicity testing



chamber

photometric system (light source)

radiator cone

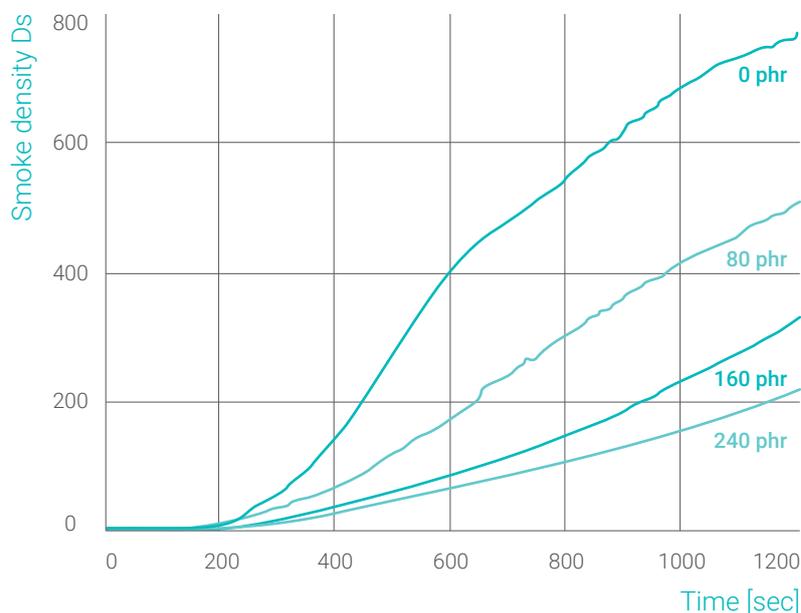
Smoke chamber – an apparatus to measure the smoke density during combustion.

This records the release of smoke over time on the basis of the optical density. The optical density is derived from the smoke released from the specimen into a sealed box which weakens the transmission of a laser beam traversing the box. The more smoke develops in the course of the test, the lower the transmission and thus higher is the optical density.

As the smoke density depends to a great extent on the fire conditions, the test materials are subjected to a double test. In the "non flaming" mode the spec-

imens are only exposed to a radiation heater which causes smouldering, while in the "flaming" mode the test is carried out using a small pilot flame.

The maximum values of the smoke density are generally taken for comparison. In addition to this the "Ds 16" is often determined. This is the time in seconds [sec] at which a "density" of "16" is reached. "16" is a specific value derived from the light absorption at which a person can still see escape routes and emergency exits.



- low maximum smoke density
- delayed smoke formation
- elongated escape time

Smoke Density versus filler loading **APYRAL® 20X** in UP-resin according to ISO 5659-2, no pilot flame.

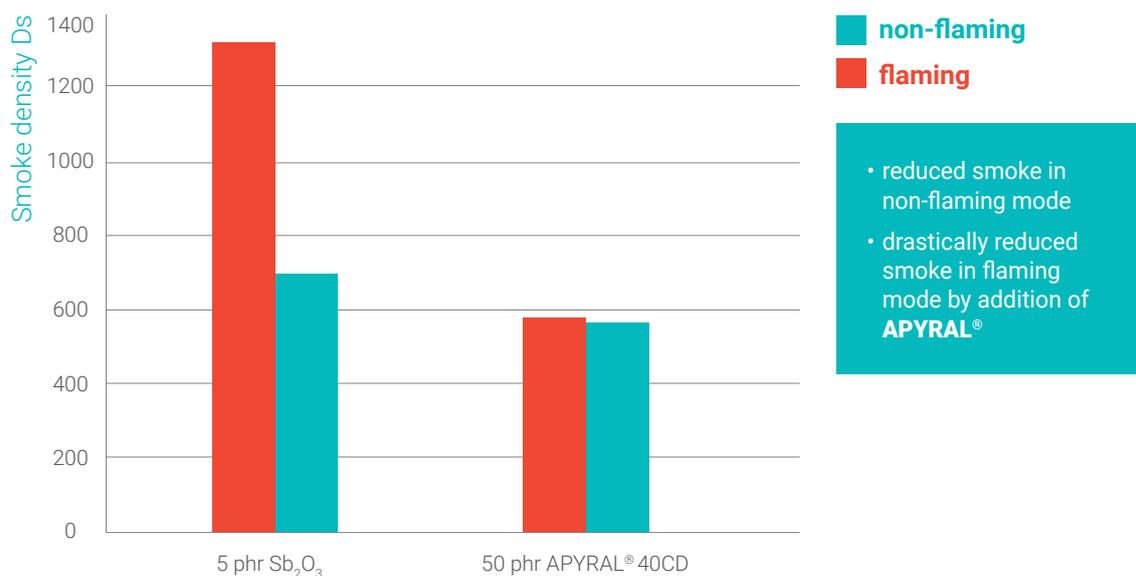
Using an unsaturated polyester resin (UP) filled with different loadings of **APYRAL® 20X**, the diagram above shows the influence of the aluminium hydroxide filling loading on the time-dependent smoke density (without pilot flame).

At only 80 parts **APYRAL® 20X** per 100 parts of the resin (phr), the optical density is significantly reduced. At higher filling ratios, the smoke-reducing properties of **APYRAL®** are particularly obvious.

Of greatest importance is the smoke reduction of metal hydrates in halogenated materials, for example products which contain either halogenated

flame retardants, or matrix polymers containing halogens, such as PVC (polyvinyl chloride). Due to their halogen content these products do not ignite easily but when they burn they produce a dense, black and harmful smoke which may contain corrosive hydrohalogen acids.

Also for these PVC compounds **APYRAL®** and **APYRAL® AOH / ACTILOX®** are very effective smoke suppressants as they reduce the corrosivity by neutralising the acidic gases. In this case we speak of „Flame Retarded Low Smoke compounds“ (FRLS, see graph on p. 12).



Maximum smoke density of plasticized PVC with different flame retardants.

APYRAL® actively reduces the corrosivity of the smoke by neutralizing the acidic gases which form during combustion of PVC compounds. The black smoke evolving by the decomposition of PVC compounds contains harmful toxic gases and corrosive hydrohalogen acids which will be adsorbed by the highly porous ash of Al₂O₃.

Furthermore, in case of an open flame, **APYRAL®** filled PVC shows a significantly reduced smoke density compared to an antimony trioxide (Sb₂O₃) filled system.

For the above described investigations, compounds of plasticized PVC have been prepared, filled with either 5 phr Sb₂O₃ or 50 phr **APYRAL® 40CD**.

The diagram above displays the optical smoke density according to ISO 5659-2, in flaming and non-flaming mode.

While the differences are rather moderate in the situation without a pilot flame, the smoke density is drastically reduced in the FRLS compound with **APYRAL® 40CD** applying the flaming mode.

Heat release / Cone Calorimeter

Fires spread through the dissipation of thermal energy which warms other materials in the direct proximity until they undergo a spontaneous combustion due to the heat itself or an igniting spark.

The ratio at which heat is released is a good indication whether a fire will grow and how quickly.

Hence, materials which release a lot of heat will contribute a significant amount to the growth of a fire and will accelerate the sudden flashover of a fire. Cone Calorimetry in accordance with ISO 5660 has established itself as a method to assess the heat emission of materials. Standardized specimens are exposed to a conical heat radiator which typically emits 30 – 100 kW/m² of thermal energy.

Low-molecular products released by the decomposing polymer are ignited by an electric igniter and consequently set the specimen on fire.

The amount of released heat is calculated using the oxygen consumption and plotted against the time (HRR). Another important aspect is the time to ignition, which should be as long as possible. Furthermore, the total amount of released heat (THR) and in particular the maximum of the heat release (PHRR) are used for the assessment. The smaller the two latter values and the later the maximum is reached, the less the tested material will contribute to the propagation of flames.

The composition as well as the smoke density of the combustion gases can also be analysed with the Cone Calorimeter accordingly to ISO 5660.

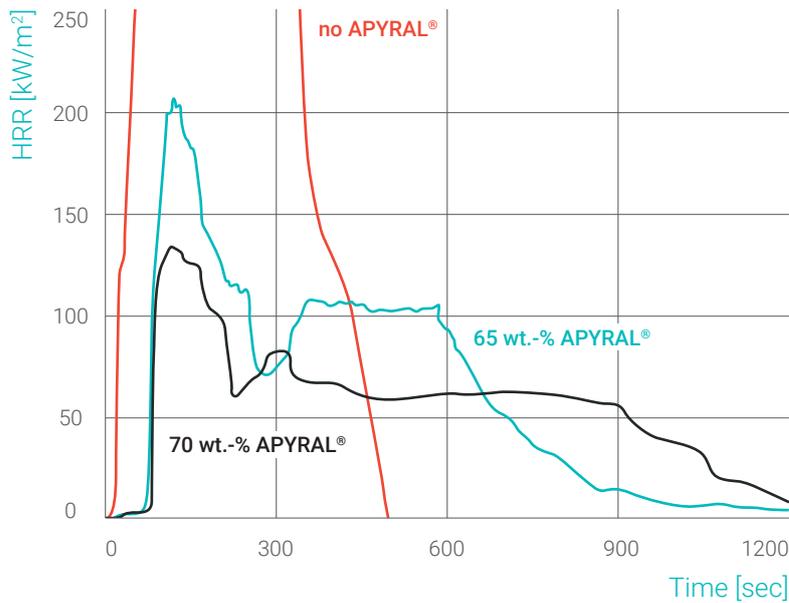
The transmission rate of a laser beam in the chimney correlates to the smoke density of the combustion gases. The less of the laser beam reaches the measuring diode, the denser is the smoke.

The content of carbon dioxide and toxic carbon monoxide in the gaseous combustion products is analysed using an IR-detector.

Calculated heat release data such as Average Rate of Heat Emitted (ARHE) and its Maximum (MAHRE), have found entry into material standards for railway (EN 45545) and cable (EN 50575) applications, making Cone Calorimetry a very useful tool for material development.



Cone Calorimeter.



- significantly retarded time to ignition
- very low peak of heat release rate
- retardation or even avoidance of flashover

Heat flux 50 kW/m², horizontal sample orientation;
Heat Release Rate (HRR) versus testing time at different **APYRAL®**
loadings; Compounds based on LLDPE / mPE / **APYRAL® 40CD**.

The illustration shows the Heat Release Rate (HRR) versus time for plastic compounds typically used for the insulation and sheathing of cables.

Using **APYRAL®**, the time to ignition of a compound can be retarded significantly and the Peak Heat Release Rate (PHRR) can be reduced dramatically which contributes both to the improvement of the flame retardant effectivity.

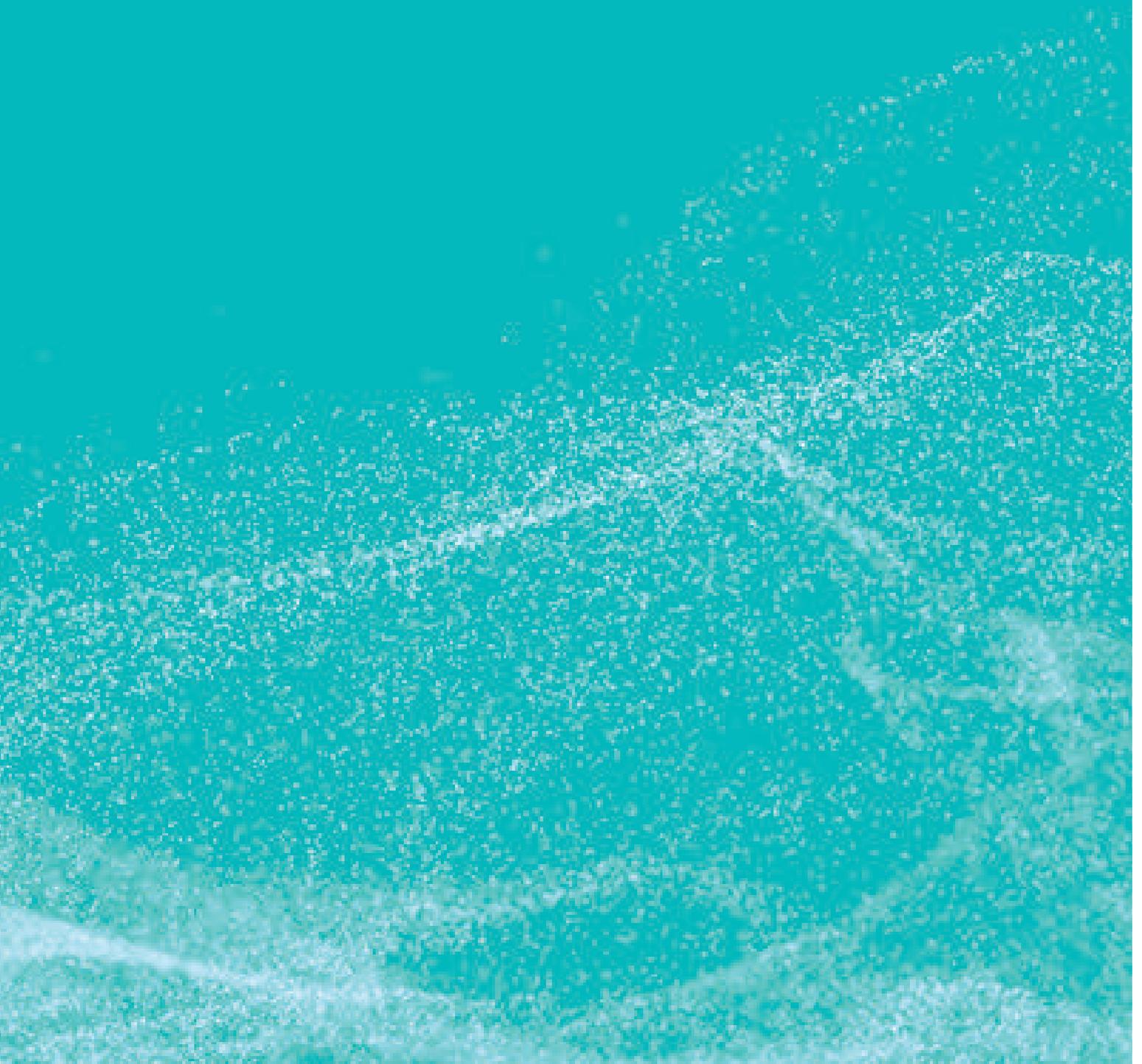
While the reference curve for the unfilled polymer blend cannot be represented on the scale used due to its high flammability and large amount of heat release (the red graph is “cut off” at the HRR-value of 250 kW/m²), the compound with 65 wt.-% of **APYRAL® 40CD** has only a very low maximum of heat release (PHRR). Although increasing the amount of **APYRAL®** further to 70 wt.-% does not retard the time to ignition further, the PHRR is substantially reduced and a considerable part of the heat is released at a later time during the measurement.

Commonly used abbreviations for the Cone Calorimeter (ISO 5660)

| | |
|--------------|-------------------------------|
| TTI | Time To Ignition |
| HRR | Heat Release Rate |
| PHRR | Peak Heat Release Rate |
| THR | Total Heat Release |
| TSR | Total Smoke Release |
| ARHE | Average Rate of Heat Emission |
| MARHE | Maximum ARHE |

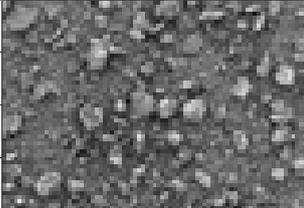
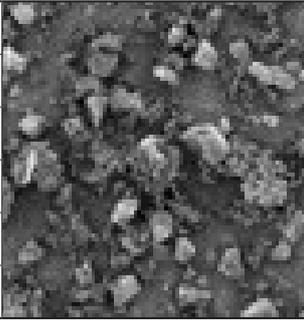
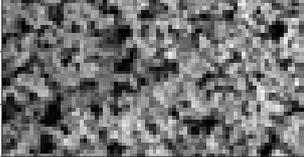
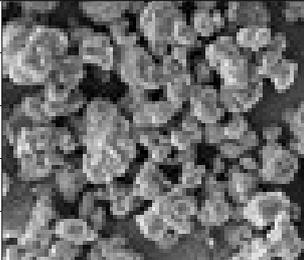
Both effects are equivalent to a further improvement of the flame retardant properties and can, in the event of an actual fire scenario – a cable fire in this case – delay or even prevent the fire from spreading to other objects (flashover).

Aluminium hydroxide **APYRAL**[®]



APYRAL® for flame retardancy

Our APYRAL® products can be – based on different characteristic properties – categorized into five product groups.

| Product | D50 [µm] | BET [m ² /g] | Oil absorption [ml/100g] | SEM |
|---------------------------------|----------|-------------------------|--------------------------|---|
| Standard | | | | |
| APYRAL® 1E | 60 | 0.5 | 21 | |
| Morphologically modified | | | | |
| APYRAL® 2E | 28 | 0.5 | 18 | |
| Ground | | | | |
| APYRAL® 8 | 20 | 1.3 | 24 |  |
| APYRAL® 15 | 14 | 1.7 | 27 | |
| APYRAL® 16 | 21 | 1.8 | 17 | |
| APYRAL® 24 | 13 | 2.5 | 19 | |
| Viscosity-optimized | | | | |
| APYRAL® 22 | 19 | 2 | 13 |  |
| APYRAL® 33 | 9 | 3 | 15 | |
| APYRAL® 44 | 10 | 2.3 | 20 | |
| APYRAL® 55 | 4 | 20 | 16 | |
| APYRAL® 20X | 30 | 1.2 | 12 | |
| APYRAL® 30X | 18 | 1.5 | 13 | |
| Fine-precipitated | | | | |
| APYRAL® 40CD | 1.8 | 3.5 | 22 |  |
| APYRAL® 60CD | 1.0 | 6 | 28 | |
| APYRAL® 120E | 0.9 | 11 | 37 | |
| Surface-modified | | | | |
| APYRAL® 40T1 | 1.4 | 4.0 | 27 |  |
| APYRAL® 40 VS1 | 1.6 | 3.5 | 33 | |
| APYRAL® 60 VS1 | 1.0 | 6 | 45 | |
| APYRAL® 40 HS1 | 1.5 | 3.5 | 26 | |
| APYRAL® 60 HS1 | 1.0 | 6 | 30 | |

General guidelines for the use of APYRAL®

Besides the necessary flame retardancy, the maintenance of certain mechanical characteristics is another important aspect of flame retardant polymers.

The basic rule is that the finer the filling material used, the lower is its influence on the mechanical characteristics. The fine-precipitated qualities **APYRAL® 40CD**, **APYRAL® 60CD** and **APYRAL® 120E** are most suitable for use in thermoplastics and elastomers for end applications such as cables, conveyor belts, thermal insulation foams and housing parts.

The coarser grades are ideal when high filling levels with low viscosity are required. The standard product **APYRAL® 1E** is used in carpet backings and in construction for the manufacture of dispersion paint and filler. The morphology modified grade **APYRAL® 2E** is used mainly for the manufacture of flame retardant cast resins, particularly in the electronics sector.

The ground products **APYRAL® 8** and **APYRAL® 15** are used for a wide range of applications, but

mainly in liquid resin systems used in glass fibre reinforced BMC products and in fire-protected coatings.

APYRAL® 16 and **APYRAL® 24** are particularly suitable for glass fibre-reinforced components on a UP-resin basis manufactured using the processes SMC, BMC, RTM and pultrusion. The low viscosities at high filling levels are especially advantageous here.

In the case of extremely high filling levels they are only surpassed by the viscosity-optimised products **APYRAL® 20X**, **APYRAL® 30X**, **APYRAL® 22**, **APYRAL® 33**, **APYRAL® 44** and **APYRAL® 55**. With these products, filling levels up to 80 wt.-% are possible to fulfil most stringent flame retardant requirements on construction parts for public transportation.

The table on page 19 shows the main application areas for the respective products.

Processing guidelines for APYRAL®

In total, however, **APYRAL®** is one of the most attractive flame retardants, even on basis of a volume-specific cost balance.

With a specific density of 2.4 g/cm³, **APYRAL®** is a medium-density mineral filler. As a result of the required filling ratios, the density of the flame-retarded plastics is increased compared to the unfilled polymer.

APYRAL® products have a very high degree of chemical purity of at least 99.5 %. The remaining constituents consist mainly of sodium hydroxide from the synthesis, some of which is built into the crystal matrix, some adhering to the **APYRAL®**, as well as traces of iron and silicon compounds. Aluminium hydroxide is an amphoteric, non-soluble solid.

Due to the soluble sodium hydroxide components, a watery dispersion containing **APYRAL®** has a

pH value of 8 - 9. This must be taken into consideration in watery or other ion-sensitive solvents or dispersions – particularly where other additives are used - which are exclusively applicable within a certain pH range. In case of doubt, tests must be performed for possible interactions.

Due to the high degree of whiteness of **APYRAL**[®], there are generally no limits to pigmentation. In many cases **APYRAL**[®] can function both, as a non interacting white pigment and as flame retardant.

The Mohs hardness of 2.5 - 3 causes no problems even in highly filled molten masses. The relatively high heat capacity c_p of 1.65 J/gK at 400 K (127 °C)

has a beneficial effect on the shrinkage of materials filled with **APYRAL**[®] and this even has priority in some applications.

APYRAL[®] can be processed by all common methods of polymer processing without any problems. But due to its decomposition starting at about 200 °C, the processing temperature of aluminium hydroxide is limited. This limits the choice of polymer matrices to a certain extent. Higher processing temperatures are accessible with boehmite, **APYRAL**[®] AOH / **ACTILOX**[®]. Further information about processing and equipment can be found in our brochure "metal hydrates for cables".



APYRAL[®] filled thermoset applications.

Application examples for APYRAL®

The table gives a general indication which **APYRAL®** should be used in a specific application. Some **APYRAL®** types can be used in a wide range of applications. In contrast, some **APYRAL®** types are especially tailored for special applications. To

achieve the most optimal output of the flame-retarded compound system for each customer, the correct choice of **APYRAL®** for each application is crucial.

| Application \ APYRAL® | 1E | 2E | 8 | 15 | 16 | 24 | 20X | 30X | 22 | 33 | 44 | 40CD | 60CD | 120E | 40 VS(1) 60 VS1 | 40 H(S)1 60 HS1 | 40T1 |
|-----------------------|----|----|---|----|----|----|-----|-----|----|----|----|------|------|------|--------------------|--------------------|------|
| Carpet backings | • | | • | | • | | | | | | | | | | | | |
| PUR foams | | | | • | • | • | | | | | | • | • | | | | |
| SMC | | | • | | • | | • | • | • | • | | • | • | | | | |
| BMC | | | • | • | | • | • | | | | | | | | | | |
| Pultrusion | | | | | | • | • | | | • | • | • | | | | | |
| RTM | | | | | | • | • | • | • | • | • | • | | | | | |
| Hand lay up | | | • | | • | • | • | • | • | • | | | | | | | |
| Laminates | | | • | | • | • | | | | • | • | • | • | | | | |
| Gelcoats | | | | | | | | | | | | • | • | | | | |
| Casting resins | • | • | • | • | • | | • | • | • | • | • | • | | | | | |
| Conveyor belts | | | • | | • | | | | | | | • | • | • | | | |
| Cable beddings | | | • | • | • | | | | | | | | | | | | |
| Cable insulations | | | | | | | | | | | | • | • | | • | • | • |
| Cable jacketings | | | | | | | | | | | | • | • | • | • | • | • |
| Membranes, Tarpaulins | | | • | • | | | | | | | | • | • | | | | |
| Thermal insulations | | | | | | | | | | | | • | • | | | | |
| Profiles, Pipes | | | | | | | | | | | | • | • | | | | |
| Insulators | | • | | | | | • | • | | | | • | • | | • | • | |
| Dispersions | • | • | • | • | | • | | | | | | • | • | | | | |
| Adhesives | • | • | • | | | | | | | | | • | • | | | | |
| Coatings | | | | • | | • | | • | | | • | • | • | • | | | |



APYRAL® 120E for high-performance requirements

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**.

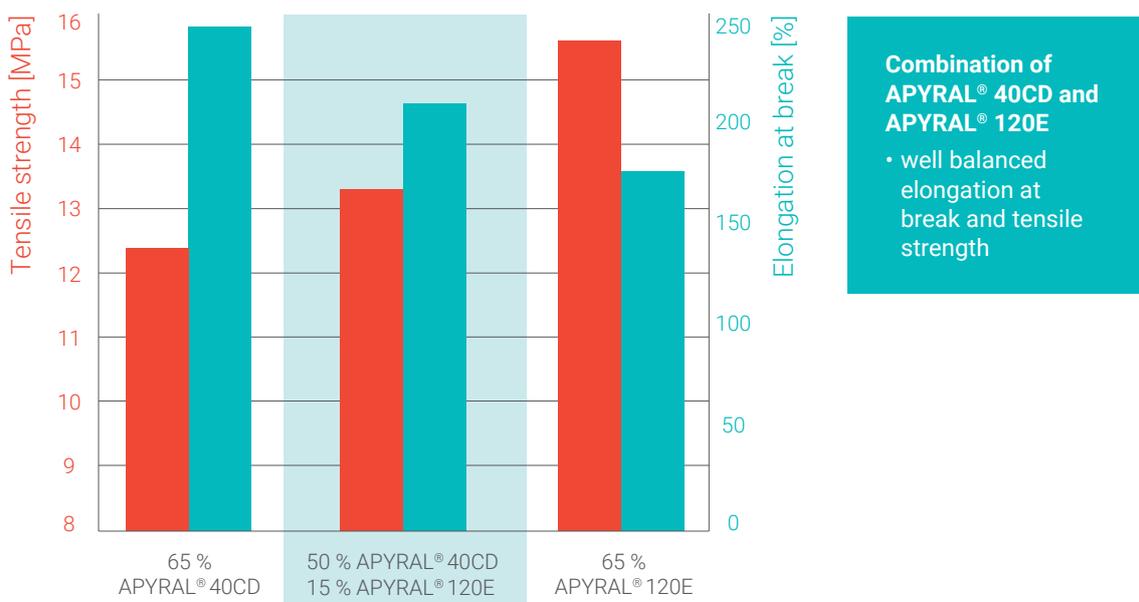
Adding this product can meet the increasing demands on the flame retardancy, good mechanical properties and processability requirements of compound systems.

To reach optimal tensile strength and flame retardancy of the compound, the metal hydrate used should consist of particles with the highest possible specific surface area. But to achieve an optimum in elongation at break and processability, the specif-

ic surface area of the filler particles should be as small as possible. The resulting challenge means that opposed properties have to be combined.

The following diagram shows this correlation for the mechanical data of EVA compounds (26 % vinylacetate) which contain 65 wt.-% of **APYRAL®** types with different specific surface areas. **APYRAL® 40CD** and **APYRAL® 120E** with a specific surface area of 11 m²/g and a combination of both products were melt-compounded into EVA.

The elongation at break and the tensile strength (TS) of the resulting compounds were determined. **APYRAL® 40CD** shows a very high level of elongation at break, **APYRAL® 120E** on the other hand side results in a high tensile strength value. The combination of 50 wt.-% **APYRAL® 40CD** with 15 wt.-% of **APYRAL® 120E** delivers a compound with a very good balance of both properties.

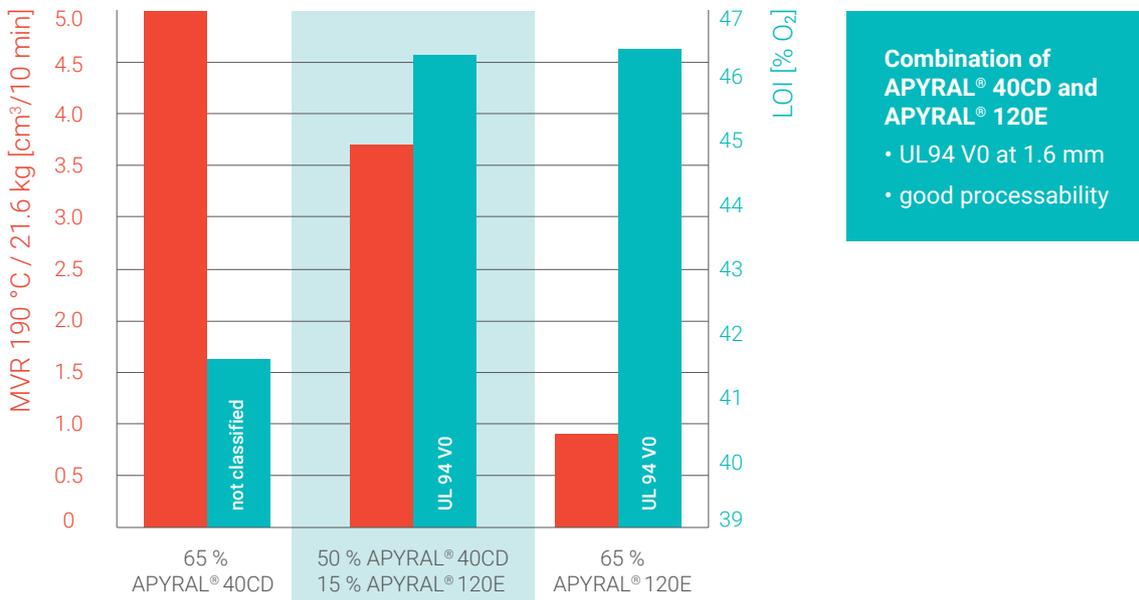


Elongation at break and tensile strength of EVA compounds with 65 wt.-% filling level.

While **APYRAL® 40CD** alone achieves no UL 94 V classification, both **APYRAL® 120E** and the combination of **APYRAL® 40CD** and **120E** reach a UL 94 V0 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-values, the UL94 V classification for specimen of 1.6 mm thickness and the melt volume rate (MVR) of EVA compounds filled with **APYRAL®** are displayed.*

Surface coated APYRAL® grades

Surface coated **APYRAL®** grades are used to improve phase compatibility of the inorganic filler (**APYRAL®** or **ACTILOX®**) in the organic polymer matrix. This gives an increased dispersion and homogenization during compounding process.

Most commonly organosilanes are used to modify the mineral surface. Given the various number of

functional coating agents, the designated interaction between the polymer matrix and the filler surface can be fitted specifically.

The compounding process will be simplified by covalent attachment of the functional groups adhered to the metal hydrate to the polymer backbone.

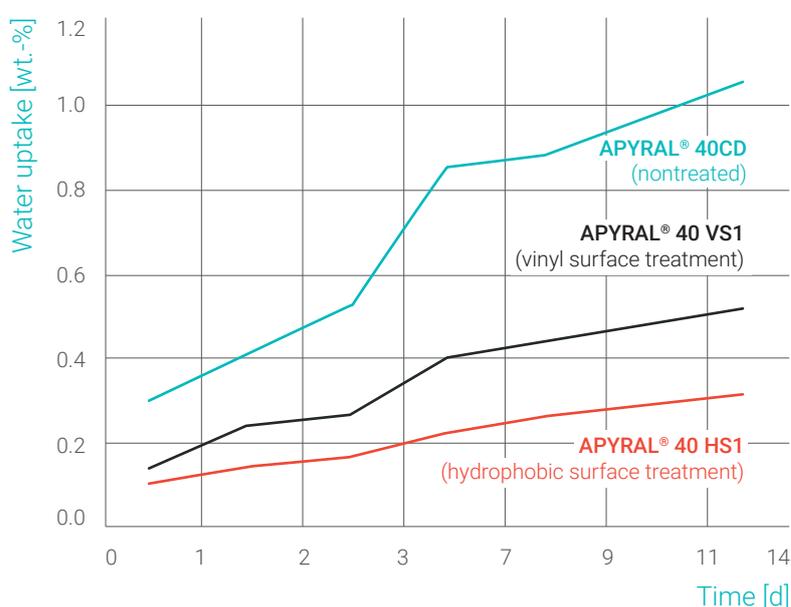
An amino-functionalized ATH can be attached to a PA or EVA matrix. A vinyl group can be grafted onto a polymer matrix induced by a radical initiator. Thermoplastic LDPE, crosslinkable PE (X-PE), natural, synthetic and particularly silicone rubbers are most common polymer matrices in this case.

In applications such as silicone rubber insulations, products with reduced wall thickness in combina-

tion with high resistance to electrical breakdown can be manufactured.

Usually, an increase of the mechanical properties e.g. tensile strength and Shore hardness are observed if a surface coated material is used.

When using a hydrophobic coating like vinyl and alkylsilane reduced water uptake in polymer compound can be achieved like shown in the diagram.



Reduced compound water uptake by hydrophobic surface coating of APYRAL®

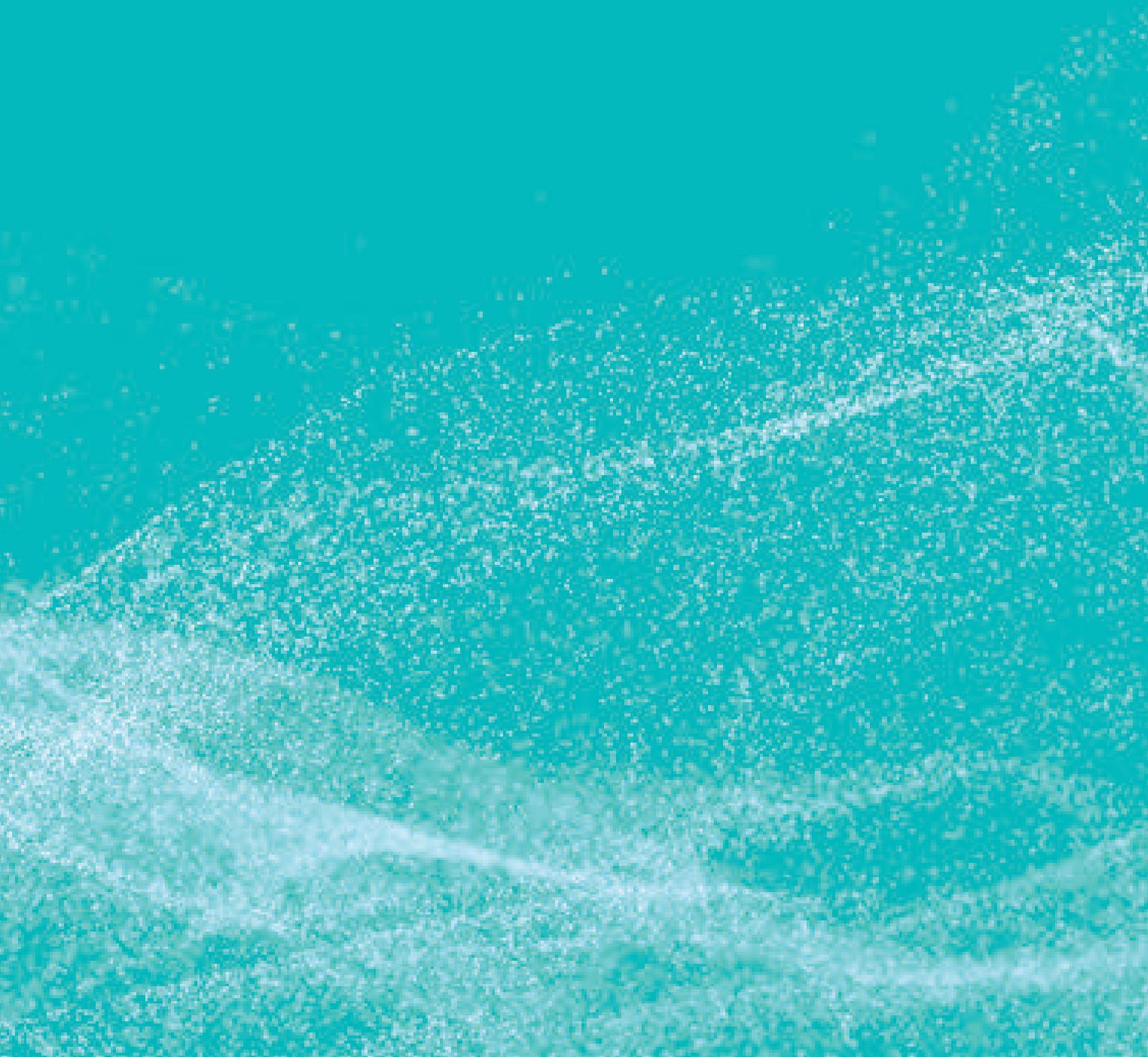
Water uptake of a PE/EVA compound (UL 00119) with 61.3 wt.-% ATH (waterbath 70 °C / 14 d).

Functional coating agents on different APYRAL® grades.

| APYRAL® grade | Treatment / Functional | Polymers |
|---------------|---------------------------|--|
| AS1 | amino | EP, EVA, EVM, PA6, PA66, PBT, PUR, PVC |
| VS / VS1 | vinyl | EPDM, LSR, PE, PMA, PMMA, PP, PVC, SIR, SR, UP |
| H / HC / HS1 | hydrophobic | LSR, PUR, SM, SIR, TPU, PVC, PE, PP |
| P / PC | polar | EP, PUR, UP |
| T1 | PVC thermal stabilisation | PVC |
| On request | others | |

Aluminium oxide hydroxide

APYRAL[®] AOH / ACTILOX[®]



Aluminium oxide hydroxide APYRAL® AOH / ACTILOX® as co-flame retardant

The Boehmite (aluminium oxide hydroxide, AOH) grades produced by Nabaltec AG are sold under the trade names **APYRAL® AOH** and **ACTILOX®**.

The temperature stability of **APYRAL® AOH** and **ACTILOX®** is significantly increased compared to ATH. These products can be processed up to 340 °C.

At this moment, five Boehmite types are available, shown with their specific data in the table below. The special cubical shape of the Boehmite particles is shown in the SEM micrograph below. This particle shape explains the very good processability of **APYRAL® AOH** and **ACTILOX®** in polymer systems.

All products are highly pure, crystalline Boehmites with a very low residual content of aluminium hydroxide (Gibbsite).

Thus, very low electrolyte content can be guaranteed. In electrical insulating applications **APYRAL® AOH** can be used without any problem.

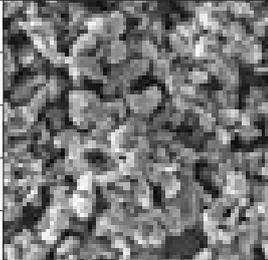
Advantages of Boehmite:

- high temperature stability
- high heat capacity ($c_p = 1.54 \text{ J/g}\cdot\text{K}$ at 500 K, 227 °C)
- good chemical resistance against aggressive media (accumulator acid)
- very fine powder
- good processability
- very low electrolyte level
- very high constant whiteness
- low Moh's hardness, comparable with other common fillers like CaCO_3

Compared to **APYRAL®**, Boehmite **APYRAL® AOH** and **ACTILOX®** contains less crystal bound water which is released at higher temperatures. Hence, the energy consumption is reduced, but combined with other flame retardants remarkable synergistic effects have been reported.

It is recommended that other halogen-free flame retardants, e.g. those based on phosphorous and/ or nitrogen are combined with the **APYRAL® AOH / ACTILOX®** to fulfill stringent flame retardancy classification like UL94 V0.

Specific data of Nabaltec's Boehmites.

| Product | D50 [µm] | BET [m ² /g] | Oil absorption [ml/100g] | Specific conductivity [µS/cm] | SEM |
|-----------------------|----------|-------------------------|--------------------------|-------------------------------|---|
| APYRAL® AOH 30 | 1.8 | 3 | 28 | 25 |  |
| APYRAL® AOH 60 | 0.9 | 6 | 30 | 65 | |
| ACTILOX® B30 | 2.3 | 3 | 25 | 35 | |
| ACTILOX® B60 | 1.2 | 5 | 28 | 80 | |
| ACTILOX® 200SM | 0.3 | 17 | 36 | 150 | |

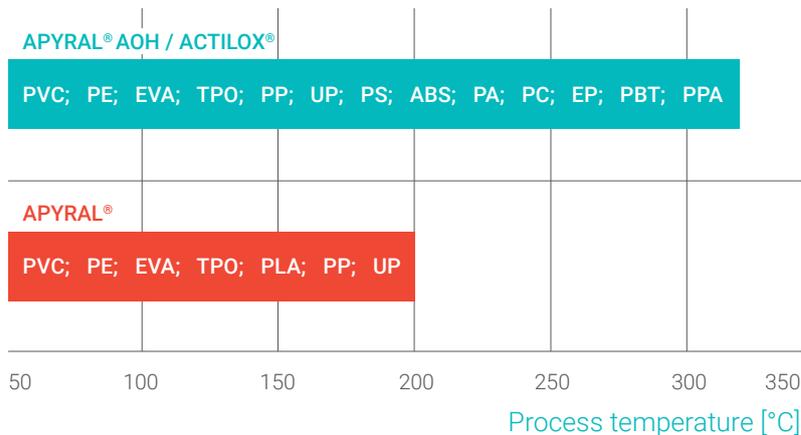
Processing

APYRAL® AOH and **ACTILOX®** can be processed by all common methods of polymer processing without any problems. In principle it is processed in the same way as **APYRAL®**. But its temperature stability allows an even increased range of accessible polymer matrices compared to **APYRAL®**. Further information about processing and equipment can be found in our brochure "metal hydrates for cables".

With mass temperatures of up to 340 °C, **APYRAL® AOH / ACTILOX®** can be used in PP, PS, PA6, PA66, PBT, PET and other technical polymer types.

In the latter polycondensates (PA, PBT, PET) stronger alkaline fillers, such as magnesium hydroxide, can cause the partial hydrolytic decomposition of the polymer chain.

Due to its low alkalinity and residual moisture **APYRAL® AOH / ACTILOX®** has clear advantages here, but should be combined with other flame retardants in order to fulfill high flame retardancy requirements.



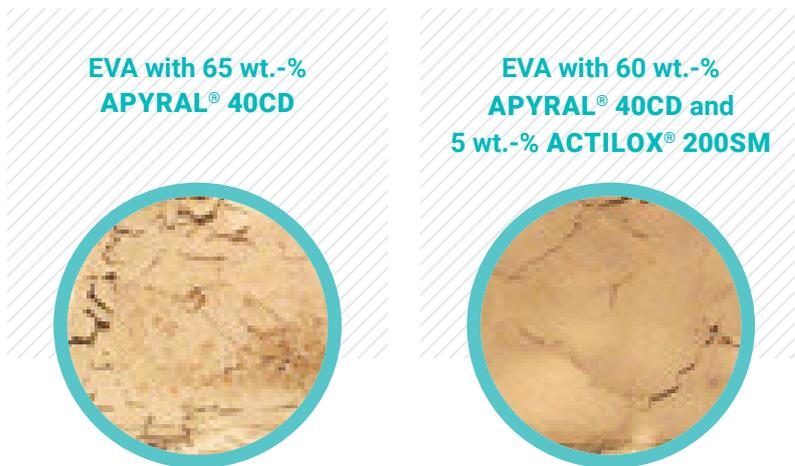
APYRAL® AOH / ACTILOX®
processable up to 340 °C

Process temperatures wherein the metal hydrates can withstand and typical polymers in which these fillers can be used.

ACTILOX® 200SM as heatshield

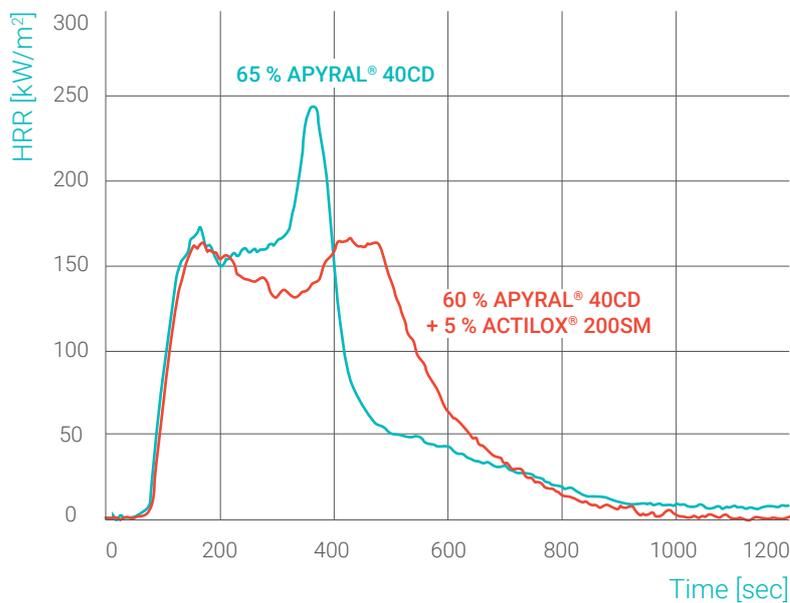
Due to the decomposition process of the metal hydrates filled compounds, an ash forms which acts as a protective shield against heat in the fire and furthermore retards the transport of volatile and flammable decomposition products to the

flame zone. As can be seen on p. 26, the residue of the Cone Calorimeter test specimen shows a very stable ash residue. Especially, the addition of **ACTILOX® 200SM** leads to a smooth ash, hardly showing any cracks.



ACTILOX® as synergist

- closed, stable and finer ash formation
- tighter surface structure
- higher diffusion barrier



ACTILOX® as synergist

- remarkable decrease of PHRR

Heat Release Rate (HRR) and Total Heat Released (THR) of EVA compounds filled with 65 wt.-% of metal hydrates.

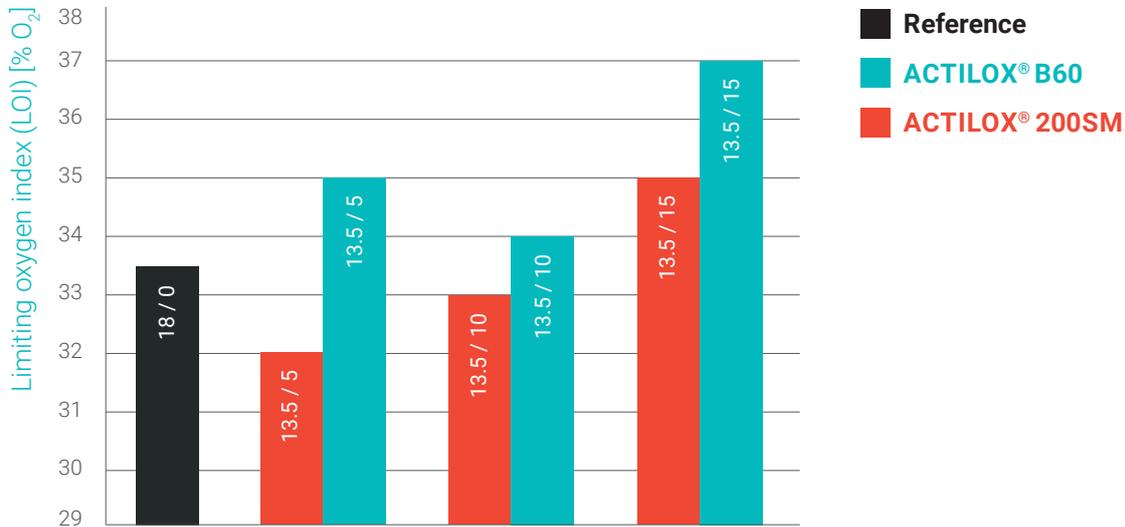
The synergistic effect of **ACTILOX® 200SM** can also be demonstrated by Cone Calorimeter measurements.

The blend of **ACTILOX® 200SM** leads to a remarkable decrease of the peak heat release rate (PHRR).

The above diagram compares heat release over time for EVA compounds filled with 65 wt.-% **APYRAL® 40CD** and a blend of 60 wt.-% **APYRAL® 40CD** and 5 wt.-% **ACTILOX® 200SM**, respectively.

Furthermore **APYRAL® AOH / ACTILOX®** in combination with metal phosphinates can give formulations which significantly exceed the flame retardancy of the individual single product (see following chapter). Additional information can be found in our brochure "Metal hydrates for technical thermoplastics".

ACTILOX® B as synergist in E&E applications



LOI-comparison of glass fibre reinforced (22.5 wt.-%) PA 6/66 compounds:

- Reference: 18 wt.-% DEPAL
- 13.5 wt.-% DEPAL in combination with 5, 10 and 15 wt.-% of **ACTILOX® 200SM**
- 13.5 wt.-% DEPAL in combination with 5, 10 and 15 wt.-% of **ACTILOX® B60**

All specimen pass UL94 V0 test (0.8 mm material strength).

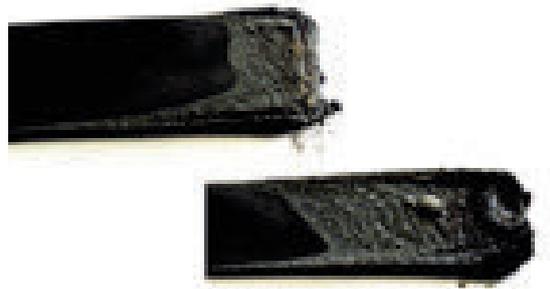
Compounds with excellent flame retardant properties can be designed by using a combination of our fine precipitated **ACTILOX® B** grades with metal phosphinates.

The highly temperature stable **ACTILOX® B** has been developed to be used as a co-flame retardant for thermal demanding polymer types such as polyamide (PA) or poly(butylene)terephthalate (PBT).

In particular, a commercially attractive alternative to currently established single product solutions can be developed. Especially for glass fibre reinforced halogen-free flame retardant PA and PBT compounds a combination of **ACTILOX®** with a metal phosphinates like aluminium-di-ethyl-phosphinate (DEPAL) is promising.

Here the combination of 5 wt.-% **ACTILOX® 200SM** with 13.5 wt.-% DEPAL in glass fibre reinforced PA 6/66 achieves a UL 94 V0 classification at 0.8 mm. The UL 94 V-specimens displayed below

show solidified and ceramified surfaces after first (top) and second (bottom) flame application respectively.



Specimen of glass fibre reinforced PA 6 (GF: 22.5 wt.-%) filled with DEPAL (13.5 wt.-%) and **ACTILOX® 200SM** (5 wt.-%) passes UL94 V0 at 0.8 mm.

The high specific surface area of the applied **ACTILOX® B** types influences the flame retardant system positively. Additional information can be found in our brochure "Metal hydrates for technical thermoplastics"

Service

for our customers

Technical service development / production

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

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

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

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

Laboratory services

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

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

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

We will gladly inform you about our capabilities.

Nabaltec

product portfolio

ACTILOX®

Boehmite, as flame retardant filler and catalyst carrier

APYRAL® AOH

Boehmite, as flame retardant and functional filler

APYRAL®

Aluminium hydroxides, as flame retardant and functional filler

GRANALOX®

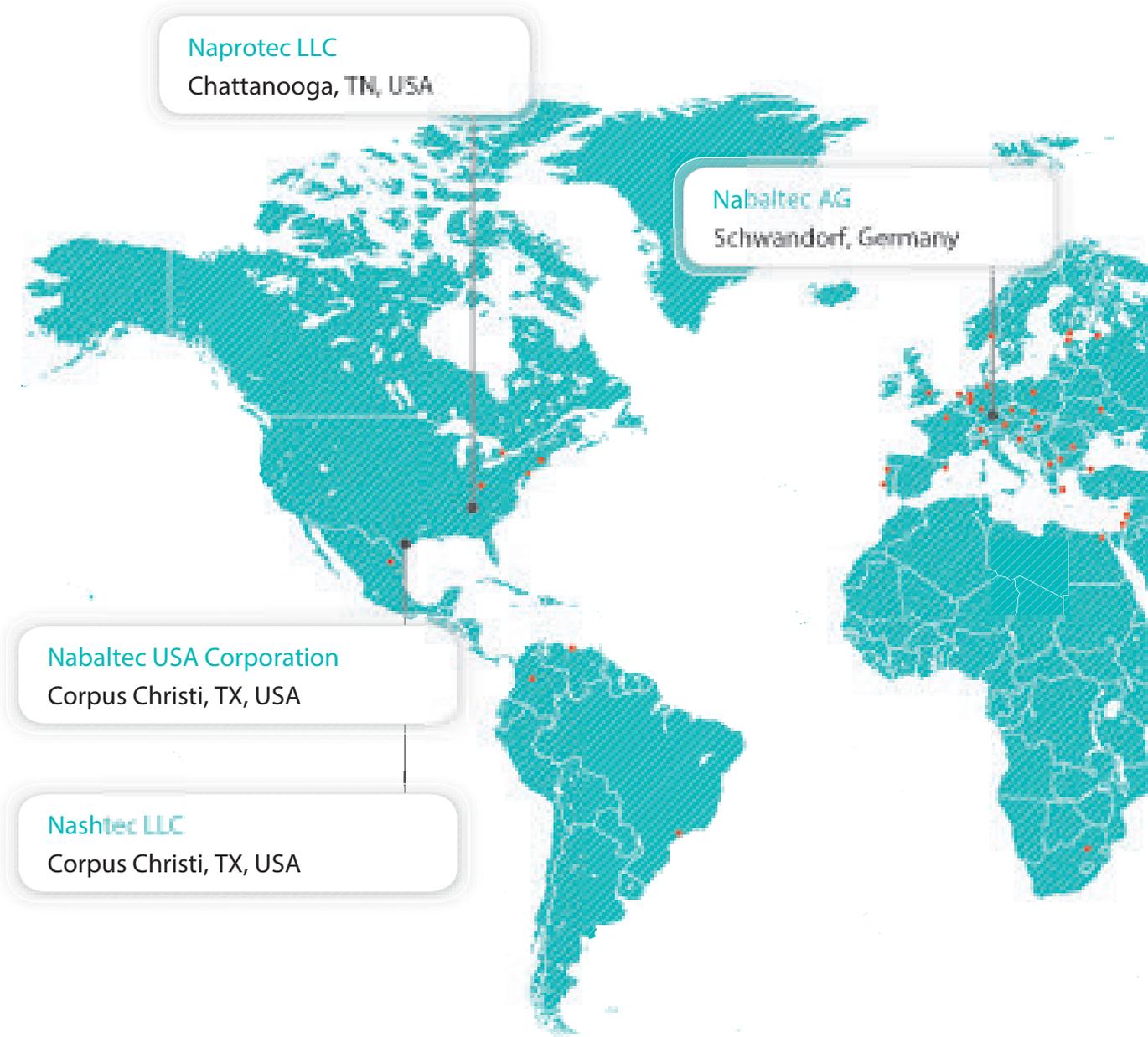
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● Locations ● Agencies



Nabaltec (Shanghai) Trading Co., Ltd.
Shanghai, China

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

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.

11 / 2022