

Nabaltec

Metal hydrates for PVC

APYRAL®

ACTILOX® B

ACTILOX® SM



Metal Hydrates for PVC

Product	D50* [µm]	BET [m ² /g]	Oil absorption [ml/100g]	Main application
APYRAL® – Ground				
APYRAL® 8	20	1.3	24	Wire and cable • Bedding compounds Construction • Roofing • Flooring
APYRAL® 15	14	1.7	27	
APYRAL® 16	21	1.8	17	
APYRAL® 24	13	2.5	19	
APYRAL® – Fine-precipitated				
APYRAL® 40CD	1.8	3.5	22	Wire and cable • Insulation compounds • Sheathing compounds Construction • Flexible profiles • PVC/NBR foams
APYRAL® 60CD	1.0	6	28	
APYRAL® 40T1	1.4	4.0	27	
ACTILOX® B – Boehmite				
ACTILOX® B30	2.2	3	28	Wire and cable • Insulation compounds • Sheathing compounds Construction • Flexible profiles • PVC/NBR foams
ACTILOX® B60	0.7	5	30	
ACTILOX® – Submicron co-flame retardants				
ACTILOX® 200SM	0.3	17	36	Wire and cable • Synergist / co-flame retardant Construction / wire and cable • Co-stabiliser for PVC

*Laser granulometer Microtrac S 3500

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.

Content

01 Flame retardancy of PVC

02 APYRAL[®] and ACTILOX[®] B

03 Cable compounds

Influence of plasticisers	10
Ignitability	11
Smoke release	12
Heat release, flame spread reduction	13
Physical properties	14
Water uptake	16

04 Compounding and processing

05 Improved thermal stability of plasticized PVC compounds

06 Plasticised PVC in construction, transport and other application fields

PVC / NBR foams	23
Conveyer belts	24
PVC plastisol	24

06 Rigid PVC

Flame retardancy of PVC

Polyvinyl chloride (PVC) was first polymerised in the year 1838, but a key breakthrough came in 1926 when Waldo Semon of B.F. Goodrich aimed to develop a synthetic replacement for the increasingly costly natural rubber. Instead he produced the first plasticised PVC by blending it with various additives to get a more flexible and easily processed material that soon achieved widespread commercial use.

Since then PVC has proven as one of the most important plastic material by being used since decades in rigid and plasticised applications like pipes, profiles, sheets, cables and plastisols.

57 wt.-% of PVC mass is made of chlorine. Its fuel content is therefore much less compared to other polymers. In addition the chlorine provides condensed phase and gas phase combustion resistance. Therefore PVC is considered as a polymer with a relatively high inherent flame retardancy. The following table gives a ranking by LOI (Limiting Oxygen Index according to ISO 4589) of most common standard polymers.

Material	LOI [% O ₂]
PE (Polyethylene)	17
PP (Polypropylene)	18
PS (Polystyrene)	18
PVC (Polyvinyl chloride)	43

However, when applying plasticisers to make PVC a flexible material, its intrinsic fire performance is sacrificed. Since many years it is common to suppress the ignitability of plasticised PVC by the addition of Antimony trioxide (Sb₂O₃ abbreviation: ATO). However, because of the gas phase reactions involved in the flame retardant mechanism of Sb₂O₃, such compounds generate large amounts of smoke and release corrosive gases when they catch fire.

Therefore and because of some other critical additives used in compound formulation, PVC received

a great degree of criticism in particular for its safety in terms of health and environmental hazards.

Mineral flame retardants based on metal hydrates are an environment friendly solution to this problem.

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

APYRAL [®]	Al(OH)₃
Chemical	Aluminium hydroxide
Mineral	Gibbsite
Common name	Aluminium trihydrate
Abbreviation	ATH
APYRAL [®] AOH ACTILOX [®] B	AlOOH
Chemical	Aluminium oxide hydroxide
Mineral	Boehmite
Common name	Aluminium monohydrate
Abbreviation	AOH

For general considerations regarding the working function of metal hydrates, please kindly refer to our brochure "Flame retardancy with metal hydrates".

PVC with low ignitability and flame spread in combination with low smoke generation can be achieved with so called Flame Retardant Low Smoke (FRLS) PVC compounds using metal hydrates as smoke suppressants. It is the purpose of this brochure to demonstrate that mineral flame retardants enable the development of environment friendly and sustainable PVC products imparting highest fire safety standards.

**APYRAL[®] and
ACTILOX[®] B**



APYRAL®

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

- ground
- fine precipitated

Fine precipitated **APYRAL®** products are broadly used to manufacture flame retardant insulation and especially sheathing compounds. Of highest importance for PVC wire and cable compounds is **APYRAL® 40CD**.

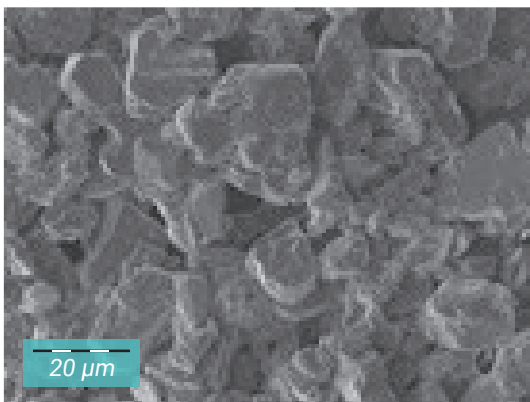
The ground grades **APYRAL® 8**, **APYRAL® 16** and **APYRAL® 24** find wide use in highly filled flame retardant bedding compounds, flooring and roofing applications. If high thermal stability of the

PVC compound needed, APYRALR 40T1 is recommended.

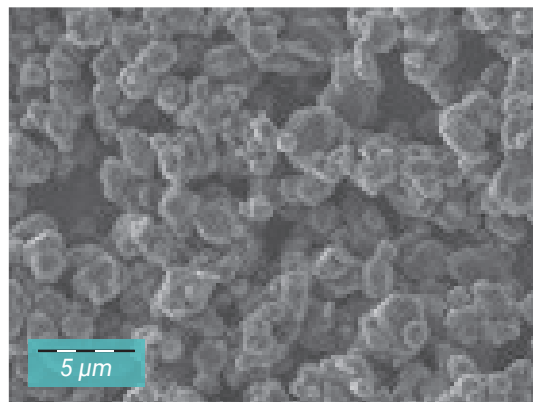
APYRAL® products have a very high chemical purity of at least 99.5 %. Due to its whiteness **APYRAL®** behaves neutral to the colouring of polymers.

Its Mohs hardness of 2.5 – 3 causes no tool abrasion problems even in highly filled molten masses. The high heat capacity c_p of 1.65 J/gK at 400K (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.



SEM image of **APYRAL® 24**



SEM image of **APYRAL® 40CD**

ACTILOX® B

Boehmite **ACTILOX® B** is produced by hydrothermal conversion of ATH.

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

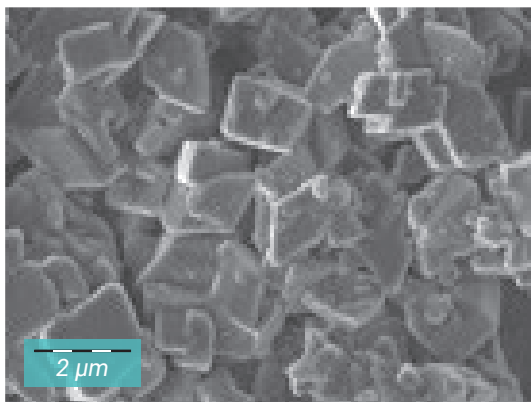
ACTILOX® B can easily be processed up to 340 °C. Additionally **ACTILOX® B** shows a very good chemical resistance, especially a very high acid resistance.

ACTILOX® B30 and **ACTILOX® B60** are characterised by an excellent processability. This is due to the unique cubic like morphol-

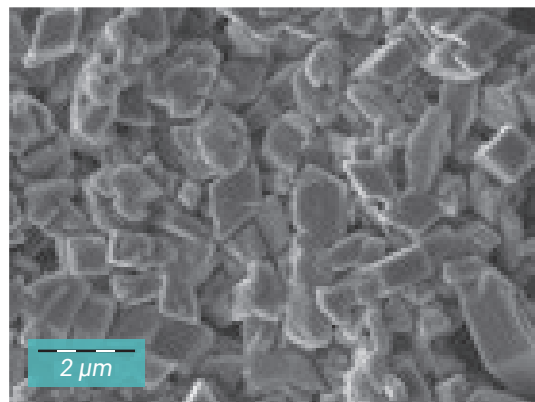
ogy of the boehmite crystals of these two **ACTILOX® B** grades. Therefore **ACTILOX® B30** and **ACTILOX® B60** are especially designated for fast extrudable compounds, whenever high line speeds and high extrusion temperatures are required.

Its high heat capacity ($c_p = 1.54 \text{ J/gK}$ at 500 K, 227 °C) combined with high temperature stability make **ACTILOX® B** ideal for use in electrically insulating heat sinks.

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

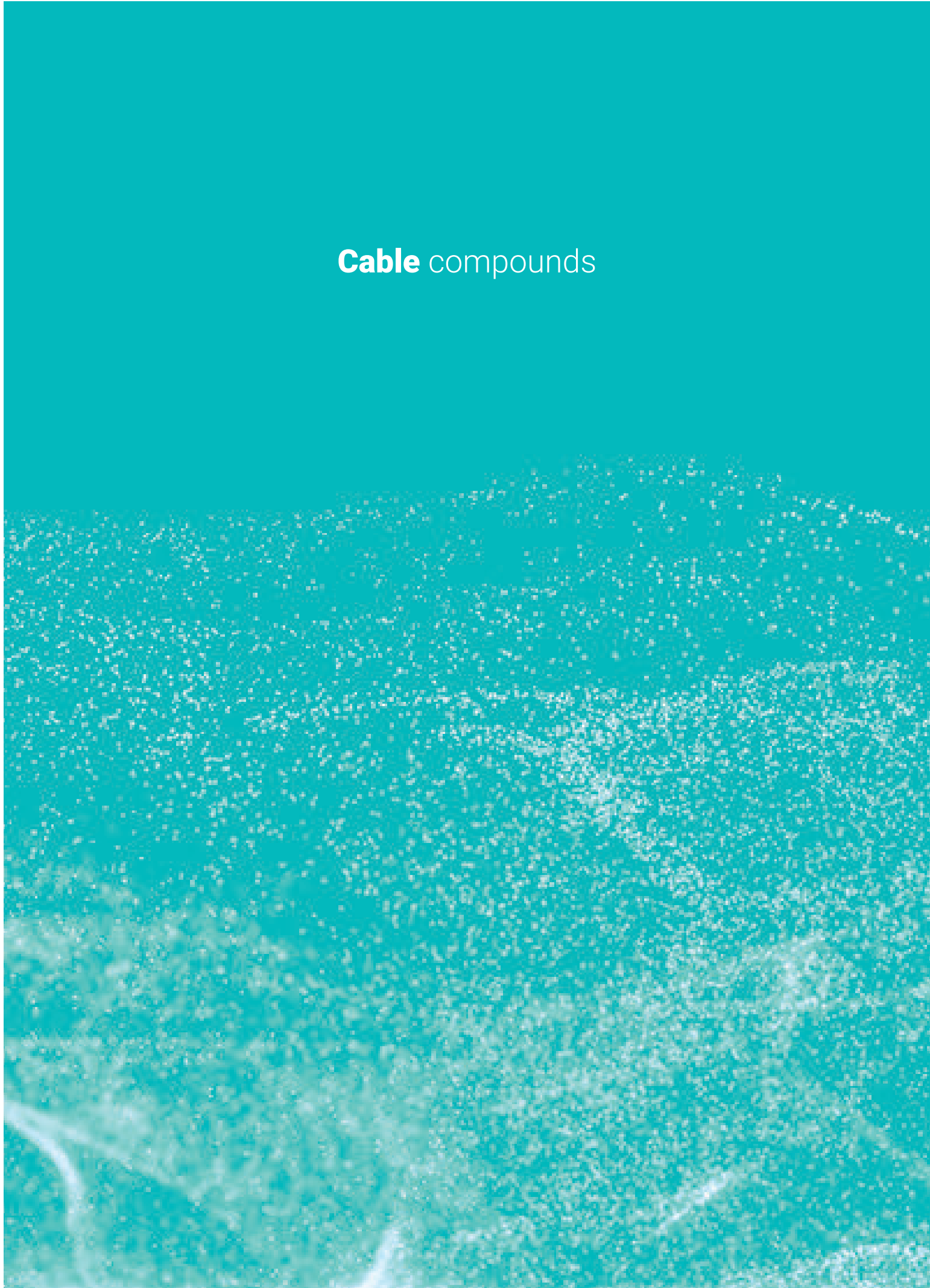


SEM image of **ACTILOX® B30**



SEM image of **ACTILOX® B60**

Cable compounds



Cable compounds

Preventive fire protection of cables is a topic of ever increasing importance. Fire retardants limit polymers, used for electrical insulation of conductors and mechanical protection of cable constructions, from ignition and flame spread.

Especially in buildings, spread of fire by cables is an important issue. Flame retardant cables can prevent a small cause, such as an electrical short circuit, from becoming a major fire catastrophe which can result in large material damages and even loss of life.

The requirements for fire safety of cables is defined by national and international standards. Fire resistance is sometimes part of standards for electrical and electronic applications, but with increasing importance it is also part of building codes like the CPD (Construction Products Directive) and new regulation CPR (Construction Products Regulation) in Europe.

The combustion behaviour in such standard tests is largely dependent on cable geometry and design. Due to this fact and because there are so many different standards existing it can't be the purpose of this brochure to give recommendations on how to fulfil such standards by defined PVC composition.

Traditionally the ignitability of a polymer compound used for insulating and sheathing was the most important criteria for material selection when it came to fire performance. This is documented in the following table giving most important North American and European wire and cable standards and trying to correlate these requirements with minimum LOI values needed for the polymer materials used in insulation and sheathing. This is a very rough indication and the exploitability of this table should not be overestimated.

Standards	LOI [% O ₂]
IEC 60332-1 ; CPR Class E ; UL VW1	> 26
IEC 60332-3 ; CPR Class D ; UL 1581	> 30
CPR Class C,B2 ; UL 1666	> 33
CPR Class B1 (S2) ; UL 910 (NFPA 262) CPR Class B1 (S1) ; NFPA 255 (259)	» 40

Nowadays other criteria like the amount of heat and smoke released in case of a fire become increasingly important. (For details regarding definitions and evaluation means in the field of flame retardancy we kindly request to refer to our general brochure "Flame retardancy with metal hydrates".)

PVC is the dominating material used in wire and cable manufacturing since many years and it is common to suppress the ignitability of plasticised PVC by the addition of Antimony trioxide (Sb₂O₃). But the sole use of Sb₂O₃ does not satisfy all fire retardant requirements. In addition there is ongoing research on health and safety issues connected to Sb₂O₃ exposure. Even though no general restriction by chemical laws exist, there is a considerable tendency in industry to reduce or even fully eliminate this mineral. The following pages give some examples on designing flame retardant PVC wire and cable compounds with reduced Sb₂O₃-content and compounds totally free of it.

Influence of plasticisers

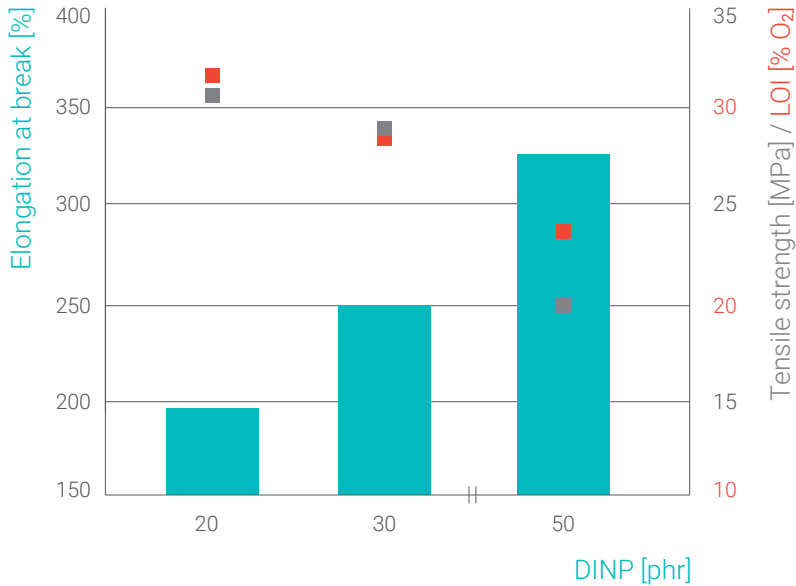


Figure 1: Mechanical properties and LOI of PVC (K = 70) compounds with different loadings of DINP.

To make PVC flexible enough for wire applications, plasticisers have to be added.

Plasticisers are highly burnable organic molecules used at high levels in plasticised PVC compounds. Among them, phthalates are the most important group for the use in PVC. These products have seen a lot of health and safety discussions over the recent years.

Developments for more environment friendly plasticiser solutions are ongoing. For wire and cable applications a change towards phthalate esters of longer chain alcohols is taking place. DOP or DEHP (Di-Octyl- or Di-ethyl-hexyl-phthalates), the

dominant plasticiser in PVC for many years, is more and more substituted by DINP (Di-isononyl-phthalate) and DIDP (Di-iso-dodecyl-phthalate).

The content of plasticiser has an important impact on physical and flame retardant properties of PVC as demonstrated in figure 1 above.

The green bars displays the importance of a high level of plasticiser for good elongation at break (E@B) values. Tensile values (TS, grey points) are going down with increasing amount of plasticiser. Especially the ignitability determined by LOI (limiting oxygen index) is deteriorated (red points). This is why PVC cable compounds need flame retardants.

Ignitability

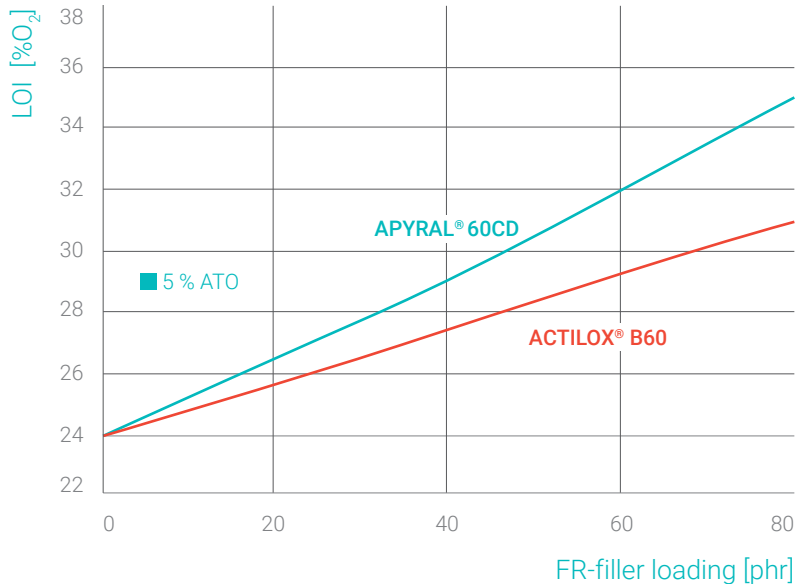


Figure 2: LOI vs. filler loading demonstrated on PVC-compounds ($K = 70$, 50 phr DINP) filled with **APYRAL® 60CD** and **ACTILOX® B60**. A compound with 5 phr Sb_2O_3 (ATO) is used as reference.

LOI is the most common and easiest way of assessing the ignitability of a polymer material. High LOI values are considered as a good indication for fire performance.

In wire and cable applications LOI values > 29 % oxygen are generally called flame resistant. However it must be stressed that LOI can only be used as an orientation or indication in material development.

Antimony trioxide (Sb_2O_3 , ATO) is a very effective flame retardant additive for PVC cable compounds. By adding only 5 phr ATO to a non-flame-retardant compound containing 50 phr DINP plasticiser, LOI can be increased from 24 % oxygen to nearly 29 % oxygen, keeping physical properties at very good level.

Metal hydrates on the other hand need significantly higher loadings to reach same level of LOI. This is demonstrated in the above diagram where LOI as a function of aluminium trihydrate and aluminium oxide hydrate (boehmite) loading is displayed. Both products have similar specific surface areas according to BET ($6m^2/g$).

As can be seen, LOI is mainly dependant on metal hydrate loading (FR filler). But the chemical type of metal hydrate has an influence too. The higher the loss on ignition of the metal hydrate the higher is the LOI at same loading.

Smoke release

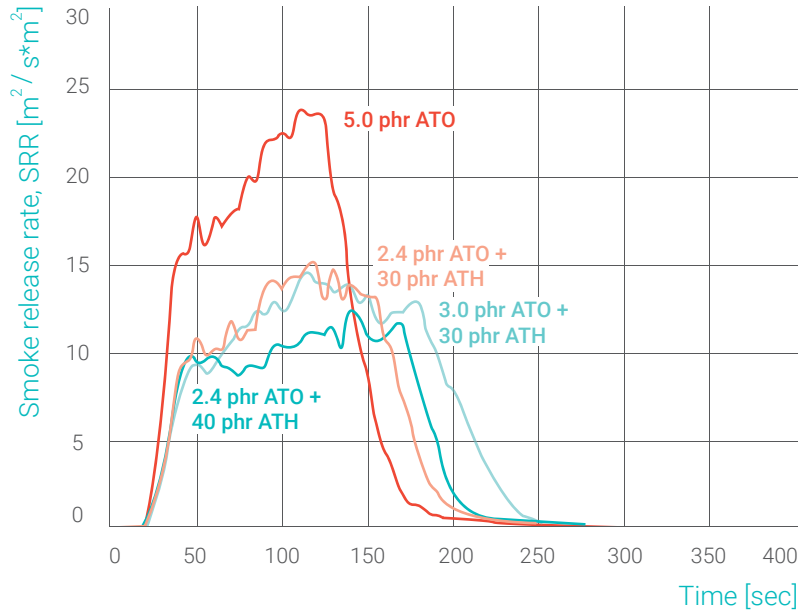


Figure 3: Smoke release rate (SRR) of PVC compounds (KV = 70, 50 phr DINP) combining ATO and **APYRAL® 40CD** (measured by Cone Calorimetry at 50 kW/m²).

Keeping smoke density as low as possible may mean the difference between life and death in the event of a fire. An escape route which remains visible for just a few more minutes can give many people the chance of saving their lives.

Due to its gas phase mechanism antimony trioxide produces black smoke when PVC is burning. By combining a reduced amount of ATO with ATH, smoke can significantly be reduced.

The diagram above (figure 3) demonstrates the effect of such combinations on smoke release rate (SRR) over time measured by Cone Calorimeter.

The results show that metal hydrates like **APYRAL®** can significantly reduce smoke generation. The aluminium oxide formed during ATH decomposition has a high internal surface where sooty particles, respectively polycyclic aromatic hydrocarbons are absorbed.

Compounds giving even less smoke can be designed when omitting antimony trioxide and using only metal hydrates as flame retardant. This effect is shown in the diagram on the next page (figure 4).

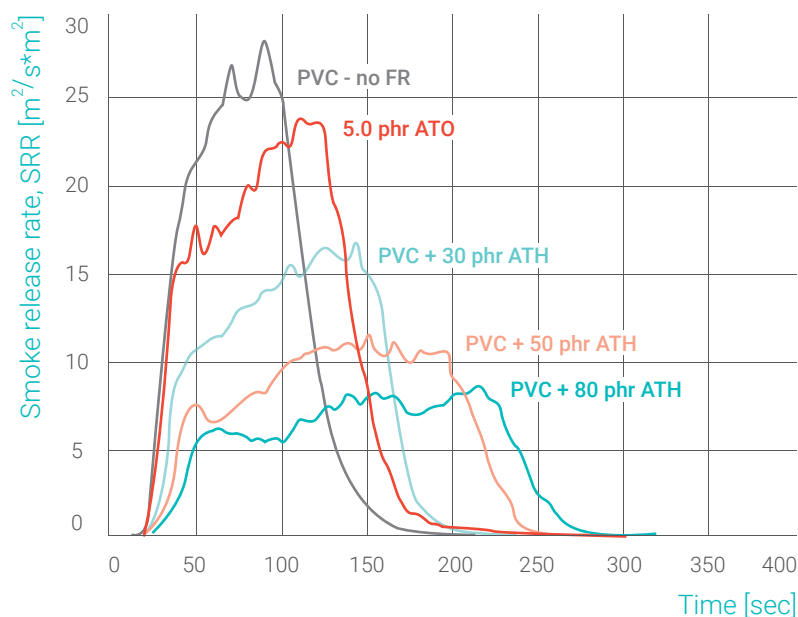


Figure 4: Smoke release rate (SRR) of PVC compounds ($K=70$, 50 phr DINP) containing different amounts of **APYRAL**[®] (measured by Cone Calorimetry at 50 kW/m²).

The diagram above demonstrates the effect of increasing **APYRAL**[®] load on smoke release rate (SRR) over time. Already by the addition of only 30 phr of **APYRAL**[®] smoke generation is delayed and also the peak of smoke release rate is reduced, giving human beings additional escape time. With increasing **APYRAL**[®] loading all smoke properties (total smoke, time to peak and peak of smoke release rate) are further improved.

Heat release, flame spread reduction

Heat release rate indicates a fire's intensity and allows a rough estimation whether a fire will grow and how fast. Materials which release a lot of heat will contribute more quickly to the growth of a fire and

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. See details in the following diagram.

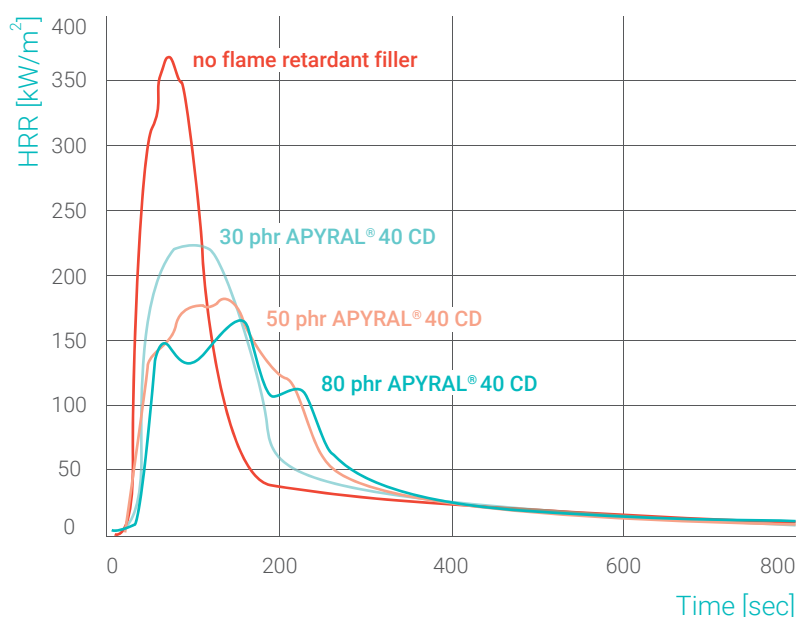


Figure 5: Heat Release Rate (HRR) over time of PVC compounds (K = 70, 50 phr DINP) containing different amounts of **APYRAL® 40CD** (measured by Cone Calorimetry at 50 kW/m²).

Metal hydrate flame retardants do not only reduce the fuel content of a polymer compound by simply diluting the burnable mass, but also significantly reduce the heat release as demonstrated for variable loadings of **APYRAL® 40CD** in the diagram above (figure 5).

The Peak of Heat Release (PHRR) is significantly reduced by the addition of metal hydrate **APYRAL®**. Time to ignition, time to PHRR, PHRR itself and total heat release are further reduced by increasing flame retardant filler loading.

Physical properties

Wire and cable insulations and sheathings need to pass certain minimum physical requirements. High loadings of mineral flame retardants deteriorate tensile strength and elongation at break.

Even when using high performance metal hydrate flame retardants like **APYRAL®** and **ACTILOX®**, physical properties suffer, but common standards can be achieved even up to very high loadings.

Using high loading levels of metal hydrate filler has an effect on physical properties (shown for aluminium hydroxide in the upper diagram 6 on page 15).

Elongation at break stays above 300% even up to 80 phr of **APYRAL® 40CD**. Deterioration of tensile strength is more significant and needs to be considered.

The bottom diagrams (figures 7, 8) on the next page indicate very well balanced physical properties for compounds filled with our boehmites even at 80 phr loading.

When using fine crystallised metal hydrate like ATH **APYRAL® 40CD** or boehmites **ACTILOX® B30** and **ACTILOX® B60**, very high loaded plasticised PVC compounds with excellent physical and flame retardant properties can be designed.

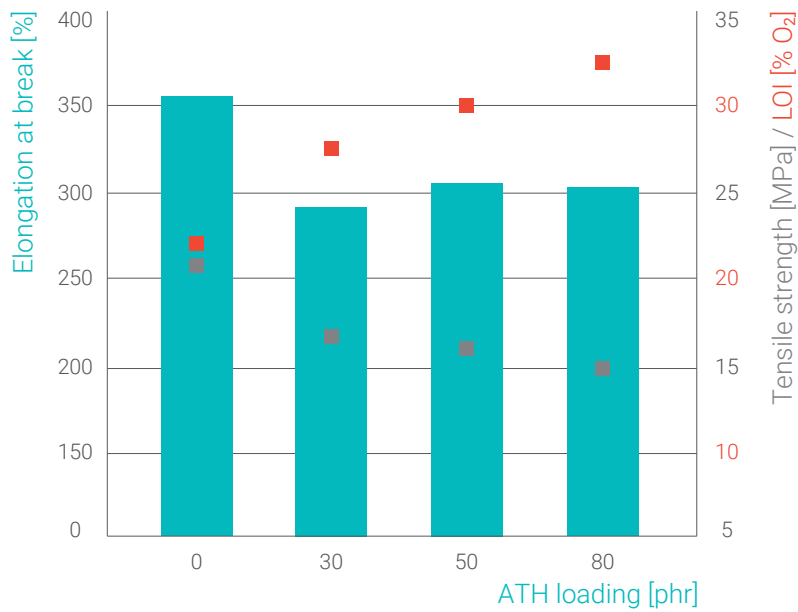


Figure 6: Mechanical properties and LOI of PVC compounds with different loadings of **APYRAL® 40CD**.

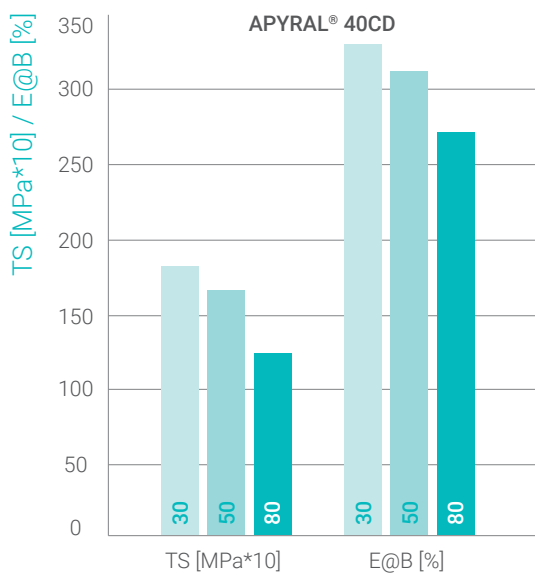


Figure 7: Tensile Strength (TS) and Elongation at Break (E@B) of PVC compounds filled with different loadings of **APYRAL® 40CD** (compounding on lab kneader chamber; specimens made of compression moulded plaques).

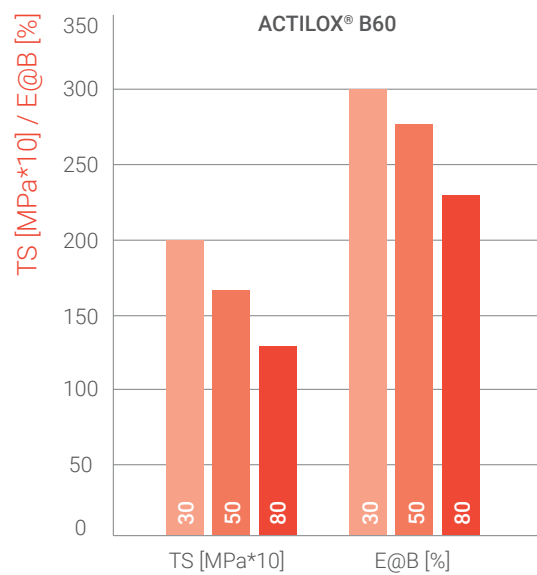


Figure 8: Tensile Strength (TS) and Elongation at Break (E@B) of PVC compounds filled with different loadings of **ACTILOX® B60** (compounding on lab kneader chamber; specimens made of compression moulded plaques).

Compounds using ATH - ATO combinations

A compromise between low flammability and physical properties is the combination of antimony trioxide and metal hydrate flame retardants.

Figure 9 shows that a combination of approximately 2 phr of ATO with 30 – 40 phr of **APYRAL® 40CD** results in an excellent balance of elongation, tensile and LOI.

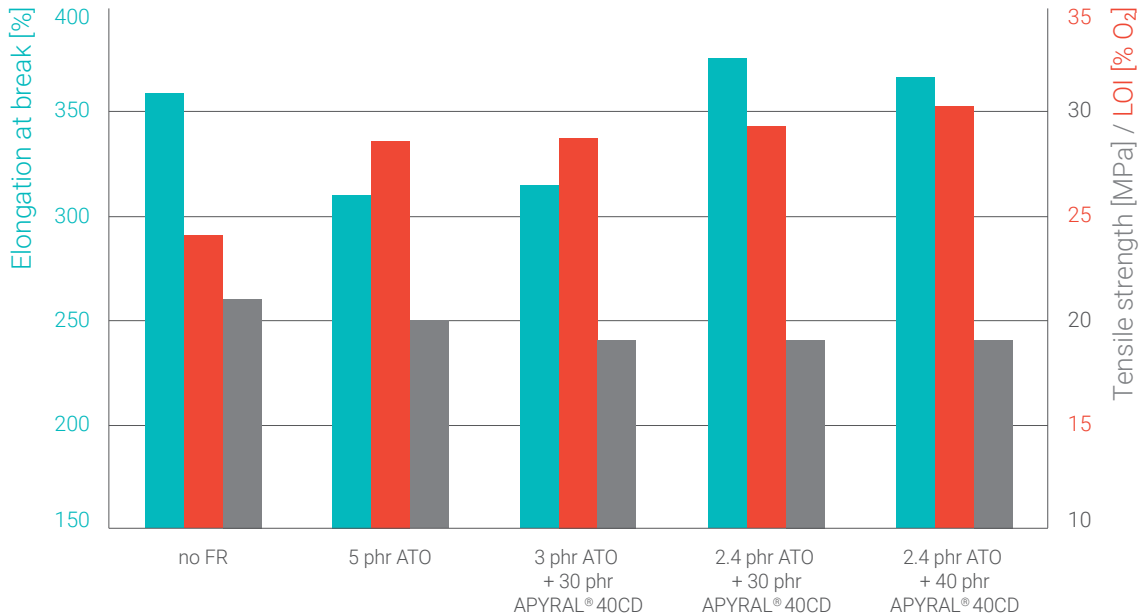


Figure 9: Mechanical properties and LOI of PVC compounds filled with ATO or combinations of **APYRAL® 40CD** and ATO.

Water uptake

Water immersion tests are an integral part of many wire and cable standards.

Mineral fillers have hydrophilic surfaces which support water uptake. But also phthalate plasticisers due to their low molecular weight and polarity of the ester groups have very significant influence on water uptake of PVC compounds.

Indeed, the plasticiser content is dominating water uptake behavior of plasticised PVC compounds. This is shown in diagram 10 on the following side. Water uptake at 70 °C on unfilled PVC

compounds with 20, 30 and 50 phr DINP is plotted versus test duration.

The same water immersion conditions have been chosen for tests on compounds filled with 80 phr **APYRAL®** and **ACTILOX® B** metal hydrate fillers. All compounds are based on Ca/Zn stabilised compounds with 50 phr DINP plasticiser content (figure 11).

It is striking that water uptake of these filled compounds is lower than compounds not containing any filler. This finding is not necessarily expected.

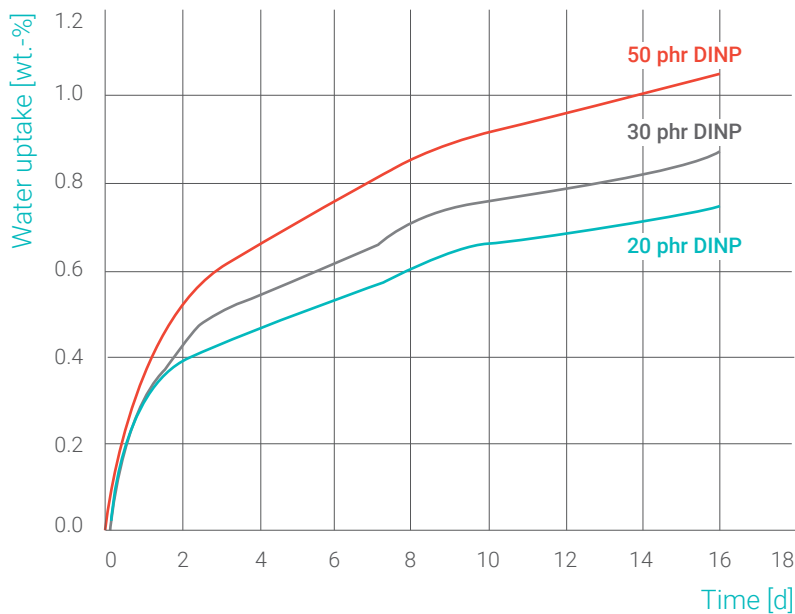


Figure 10: Influence of plasticiser content (DINP) on water uptake of PVC-compounds ($K = 70$, no filler).

Comparing boehmite (AOH) and ATH, the two compounds based on boehmite **ACTILOX® B** show the lowest water uptake.

This is presumably because boehmite (AOH) particle surfaces are covered with less hydrophilic hydroxyl groups (-OH) compared to ATH ($\text{Al}(\text{OH})_3$). The influence of the specific surface area on

water uptake is negligible. But the differences in BET surface for the investigated fillers was relatively small either.

Whenever water uptake of plasticised PVC compounds has to be improved it is recommended to use **ACTILOX® B** as flame retardant filler.

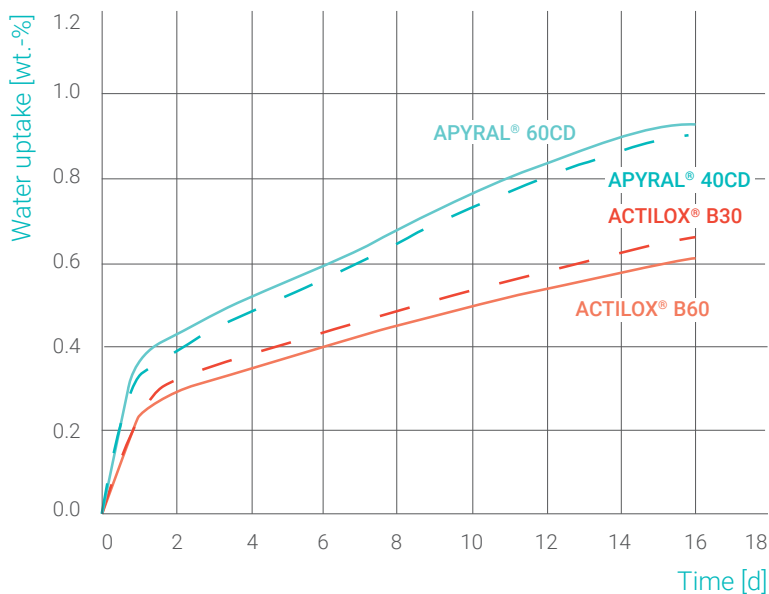


Figure 11: Influence of the type of mineral filler on water uptake of PVC-compounds ($K = 70$, 50 phr DINP, 80 phr filler).

An aerial photograph of a lush green field, possibly a golf course or a park, with a prominent white path or fairway cutting through the grass. The image is partially obscured by a dark teal gradient at the bottom.

Compounding and **processing**

Compounding and processing

The term compounding is used differently for PVC than for other thermoplastic polymers. PVC resin is delivered as a powder and the blending with other additives to a dry-blend powder is considered as the compounding step during production. Only a restricted number of PVC formulations are melt compounded into granules before being processed into shape by extrusion, injection moulding, calendering and other processes. The majority of PVC is melt processed starting from a dry-blend powder.

Dry-blending or powder mixing refers to the addition and distribution of ingredients without melting the PVC. High speed mixers are commonly used, which allow for a quick heat up of the powder blend by friction. This heat up is needed to get the PVC above its glass transition temperature (T_g , 82 °C). For the fast diffusion of stabilisers and plasticisers into the PVC and to melt waxes and other ingredients so they do not segregate by later handling. Last but not least the heat helps to reduce porosity and to create a higher apparent bulk density, which is important for the subsequent processing.

For rigid PVC the addition of metal hydrates, also because loadings are comparably low (see chapter "Rigid PVC"), is noncritical and similar to the handling of calcium carbonate and pigments like titanium dioxide.

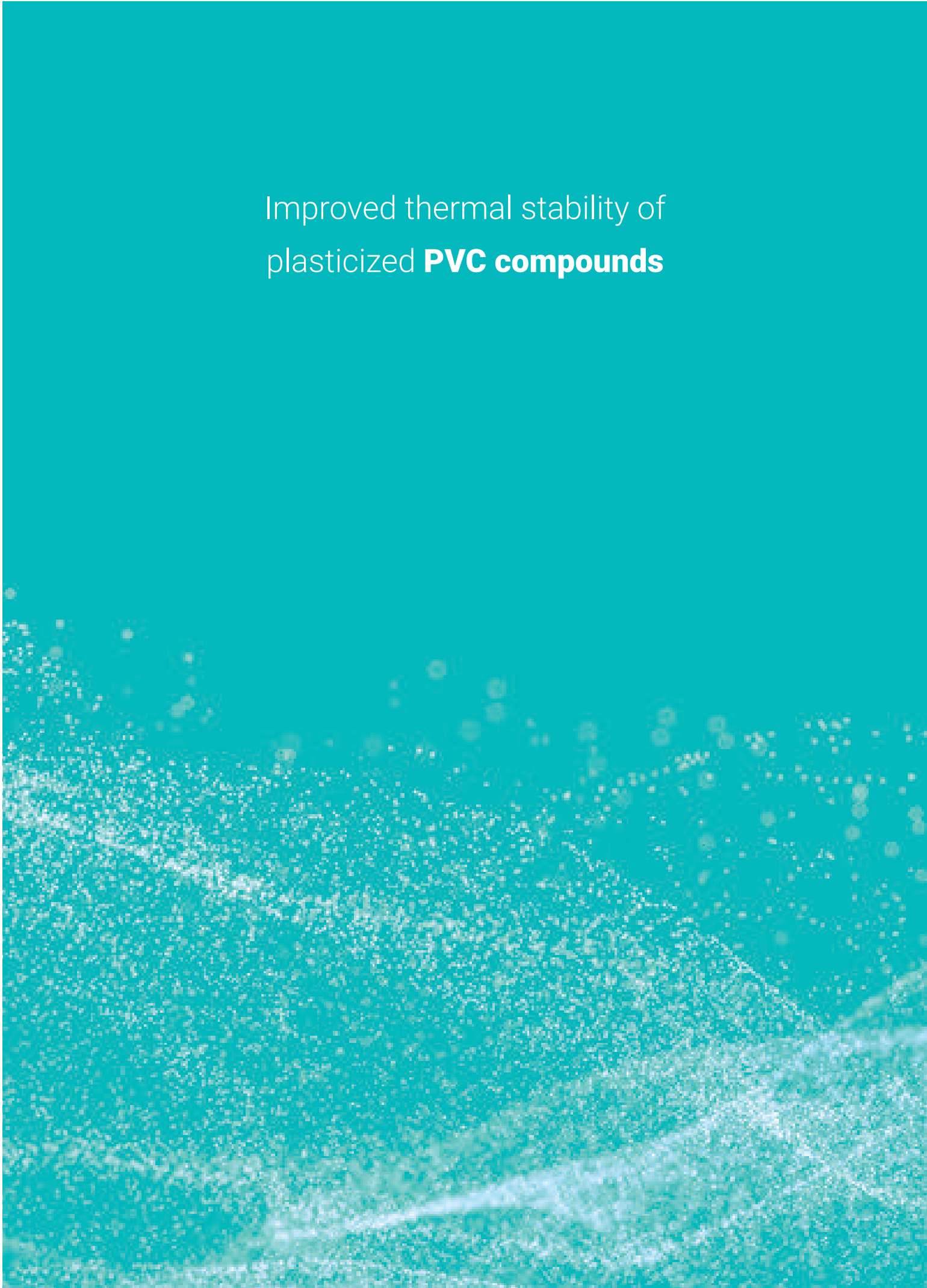
In plasticised PVC compounds it is important to add the mineral flame retardants after the stabilisers and plasticiser have been thoroughly diffused into the PVC. **APYRAL® 40CD** can be added in a second step together with other fillers, most preferably the dry-blending temperature for adding the metal hydrate should be in the range of 90 – 110 °C.

It is preferred to discharge the dry-blend into a low speed cooling mixer equipped with a vacuum degassing. Together with the correct sequence of component addition the cooling mixer will avoid potential agglomerates.



Schematic drawing of a high speed mixer used for PVC dry-blend preparation ("PVC compounding")

Improved thermal stability of
plasticized **PVC compounds**



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 (HCl) 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.

Product advantages

- Good conveyance and dosing
- Very fine particles
- Excellent mechanical properties
- Improved thermal stability in PVC
- Non-reactive surface treatment

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. 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 HCl (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 plasticized PVC compound. The stability time with **APYRAL® 40T1** is almost three times higher compared to the compound where **APYRAL® 40CD** was used.

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 8553 KA-ST/3)		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

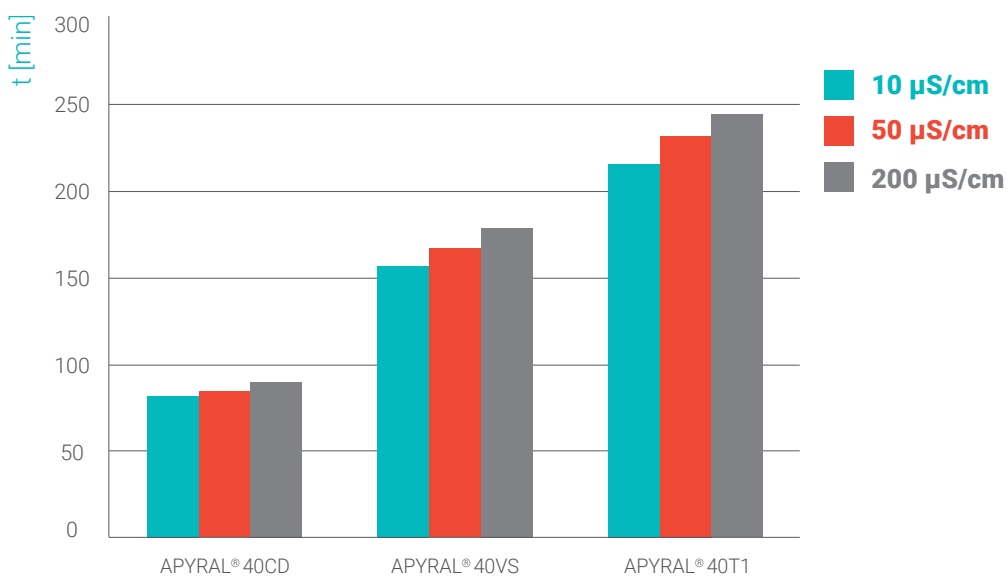
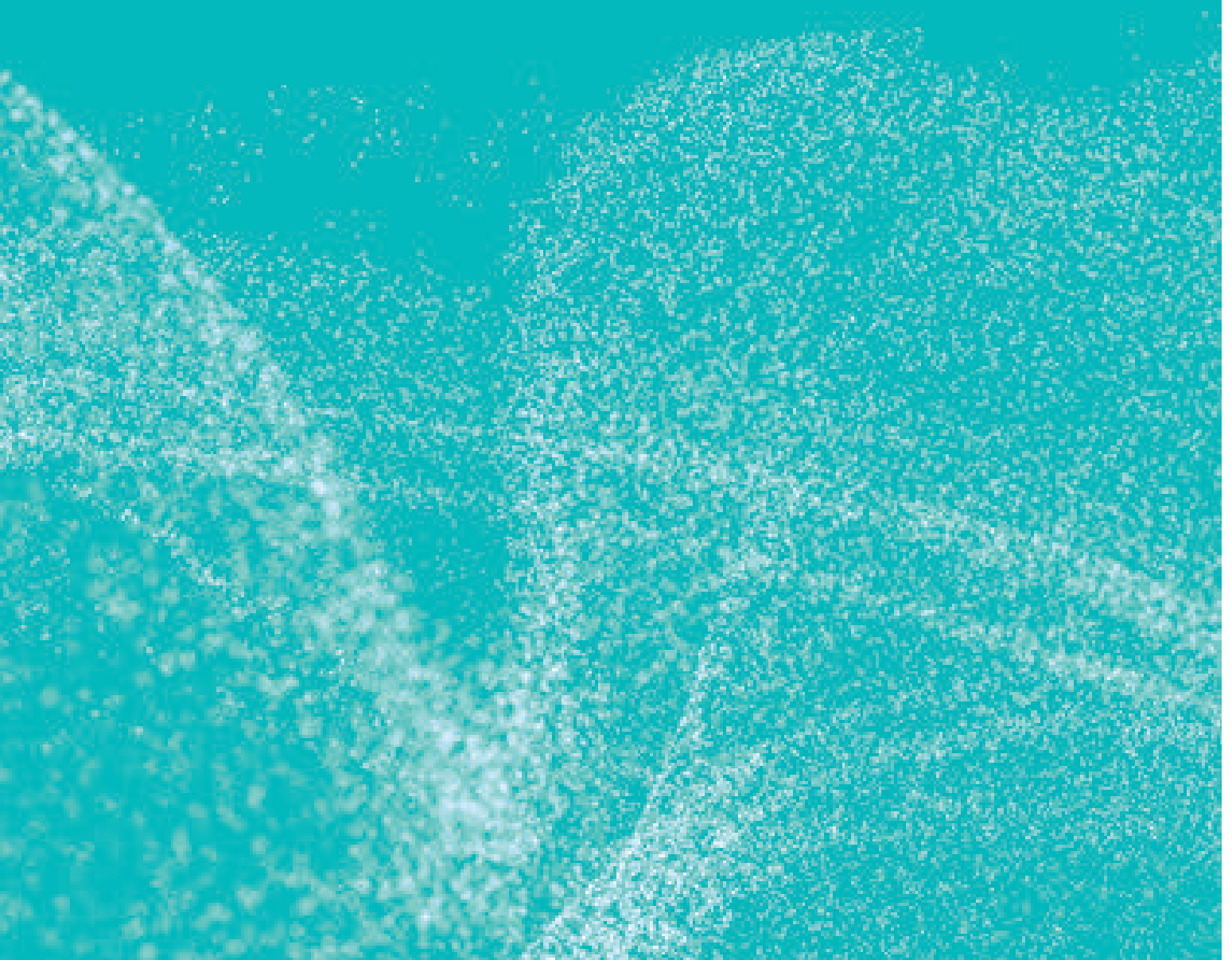


Figure 12: Stability measurement via dehydrochlorination (DHC) test of PVC compounds filled with 80 phr of various **APYRAL®** grades.

Plasticised PVC in construction, transport and other application fields



Plasticised PVC in construction, transport and other application fields

Wire and cable is of course the most important market by volume for flame retardants. But there

are other end applications using plasticised PVC fulfilling flame retardant requirements.

PVC / NBR foams

A very important application for **APYRAL**[®] flame retardants is thermal insulation foams made of PVC/NBR (NBR = Nitrile Butadiene Rubber) blends. These elastomeric materials have to fulfil severe fire resistant requirements, especially when applied in linear product, e.g. for insulating of heating and plumbing pipe work in multistorey buildings.

A proper cell structure in combination with a low foam density is a must for thermal insulation foams. Consequently very fine metal hydrate flame retardant fillers are required to enable closed cell foam formation. The total loading is restricted by the foam density requirement.

APYRAL[®] is indispensable for reduced smoke release, but to fulfil requirements of construction products tested according to EN 13823 class B and C or ASTM E 84 class B, a mix of flame retardants has to be applied. Brominated flame retardants (Br-FR) like Deca-bromo-diphenyl-oxide (Deca) or Ethane-1, 2-bis-(penta-bromo-phenyl) and antimony trioxide are used. Additionally chlorinated (Cl)-paraffins and phosphate esters used as plasticisers also contribute to reduced fire spread properties.

Current development work in industry focuses on the reduction or even elimination of brominated flame retardants. Because of ongoing discussion of the health and safety status of Cl-paraffin, formulators also should consider substitution of this plasticiser in future.

Nabaltec's **APYRAL**[®] **CD** technology offers significant processing benefits over standard fine

precipitated ATH grades when considering formulations with low or zero level of Cl-paraffin. This is because of the low oil absorption values of **APYRAL**[®] **CD** products, making reformulation to lower plasticiser levels easier.

Typical formulation of a flame retardant PVC/NBR elastomeric foam.

Component	[phr]
Polymers	
NBR PVC, K=57 or	45
PVC-co-VAc	50
BR (Butyl Rubber)	5
Flame Retardants	
Sb ₂ O ₃	10
Br-FR (Deca)	30
APYRAL [®] 60CD	100
FR-Plasticisers	
Cl-Paraffin	35
Phosphate Ester	20
Others	
Carbon Black	6
Blowing Agent	30
Antiozonant	3
Vapour Barrier Wax	5
ESBO (Epoxidised Soy Bean Oil)	1.5
Stearic Acid	1
Polyethylenglycole	5
Activators	7

Conveyer belts

Conveyer belts, especially when used in coal mines, have to fulfil flame retardant requirements. This is why PVC is often preferred over more flammable elastomers.

Combinations of flame retardants and phosphate plasticisers instead of phthalates are very often used to further improve flame retardancy of the PVC compound.

Starting formulation for conveyer belts.

Component	[phr]
PVC (K = 57)	100
Ca/Zn or Ba/Zn stabiliser	3
Phosphate plasticiser	50
APYRAL® 40CD	50
CaCO ₃	10
Sb ₂ O ₃	4

PVC Plastisol

PVC plastisol can be found in many applications like construction and transportation. The requirements of ecofriendly flame retardants like ATH for flooring and roofing as well as tarpaulins for trucks and artificial leather are increasing.

For flooring and roofing applications products like **APYRAL® 16** and **APYRAL® 24** are recommended. These products have not only an attractive price, they also meet the required conditions for the application. They show suitable high whiteness, good dispersion and low oil absorption.

Fine precipitated ATH grades like **APYRAL® 40CD** and **APYRAL® 60CD** have often been used for coated fabrics and artificial leather because of the surface finish requirements and foaming property of end products and proper fabric wetting by the PVC plastisol needed during manufacturing.

For higher flame retardancy requirements more phosphate at the cost of phthalate plasticisers are recommended. The diagrams on the next page display the flame retardant property (LOI) and viscosity shifting of ATH filled PVC plastisol with a formulation used for a compact layer.

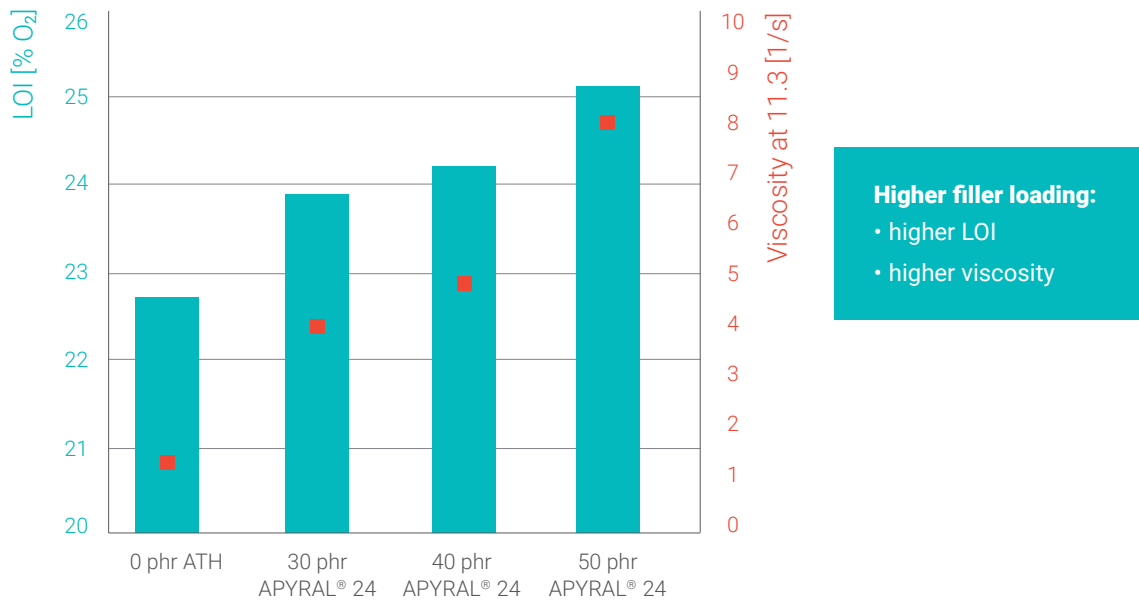
As expected, LOI and viscosity increase with increased ATH loading. With higher fineness of the

Basic formulation for coated fabrics. APYRAL® loading and plasticiser composition dependant on specific performance requirements.

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

filler material, more plasticiser will be absorbed and the viscosity of the plastisol will increase. Nevertheless, a loading of 50 phr **APYRAL® 24** still shows good flow property, which is necessary in plastisol application (figure 13).

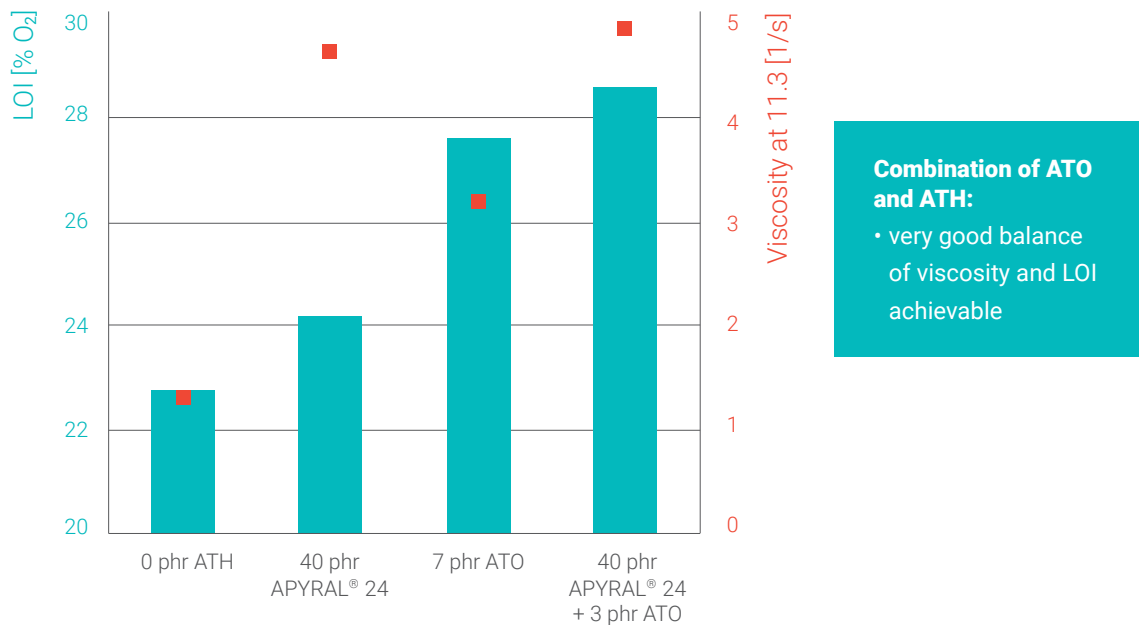
The results shown in the lower diagram (figure 14) on the following page indicates that also an addition of 40 phr **APYRAL® 24** enables a reduction of more than 50 % of Sb₂O₃ (ATO) provided that an acceptable viscosity, Yellow Index, mechanical properties and similar LOI are sufficient.



Higher filler loading:

- higher LOI
- higher viscosity

Figure 13: LOI (on cured specimen) and viscosity of ATH filled plastisol (before curing) (carried out in accordance with the basic formulation on page 24 CaCO₃ but without Sb₂O₃).

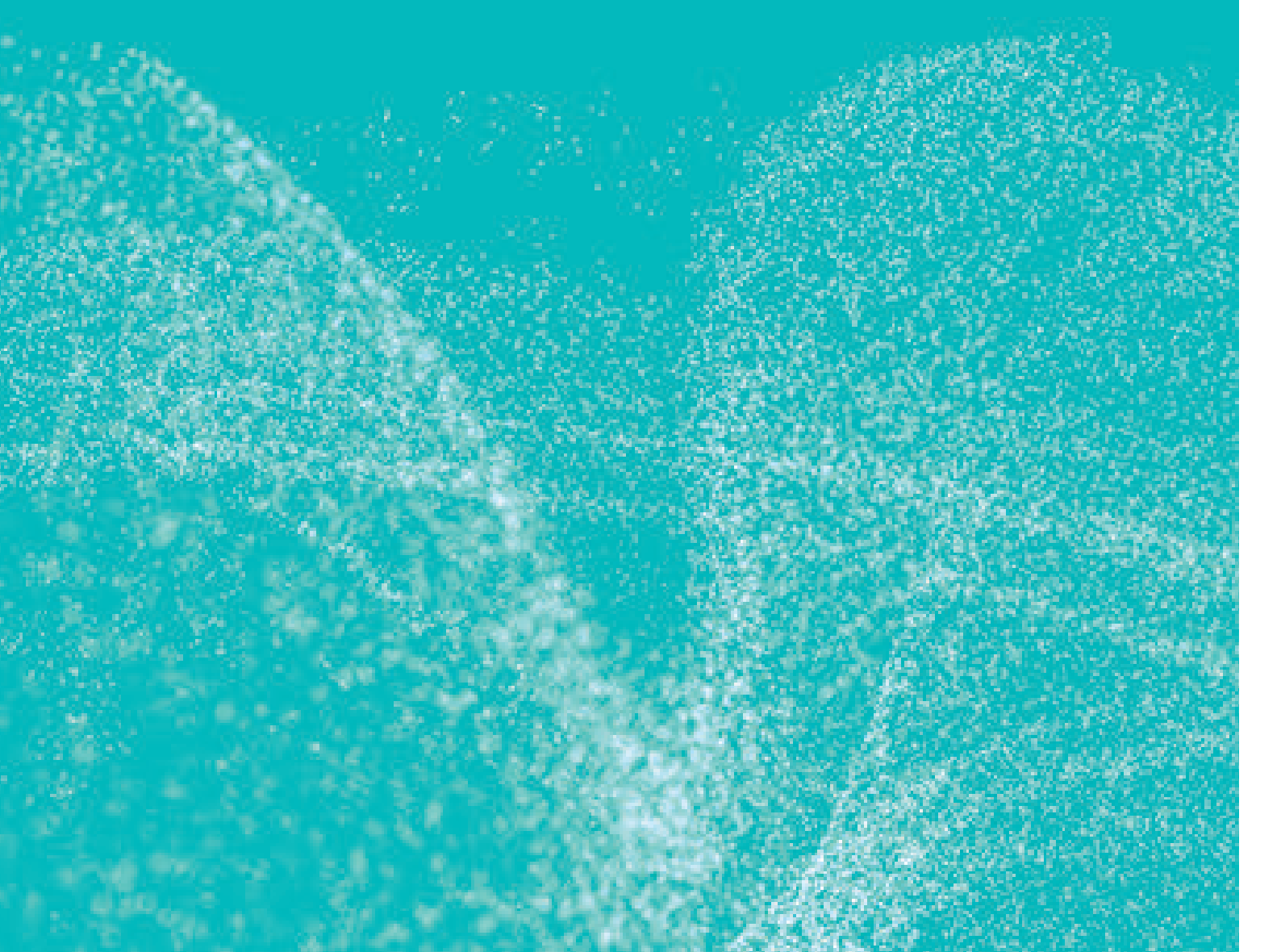


Combination of ATO and ATH:

- very good balance of viscosity and LOI achievable

Figure 14: LOI (on cured specimen) and viscosity of filled plastisol (before curing) filled with ATH and ATO (carried out according to the basic formulation on page 24).

Rigid PVC



Rigid PVC

Due to its intrinsic fire retardant properties rigid PVC does not require any flame retardant for most applications. Nonetheless rigid sheets and profiles of low thickness need additional flame retardants to fulfil most stringent requirements in construction applications according to DIN 4102, B1 or Epiradiateur M1.

Antimony trioxide (Sb_2O_3) can be used but a more favourable and sustainable solution in respect to environmental aspects is the use of ATH **APYRAL®** or boehmite **ACTILOX®** (AOH).

The below diagram displays LOI of rigid PVC compounds at 15, 20 and 25 phr of **APYRAL® 40CD** or **ACTILOX® B30**, loading. Specimens of 2 mm thickness have been prepared with a lab single screw extruder (figure 15).

Unexpected and in contrary to plasticised PVC compounds the LOI of boehmite **ACTILOX® B30** containing compounds is higher than of ATH **APYRAL® 40CD** filled compounds. The surface finish of **ACTILOX® B30** filled compound is very smooth and gelation is very easy without the addition of any lubricants or process aids.

It is believed that the unique cubic like morphology of the **ACTILOX® B** boehmite crystals in

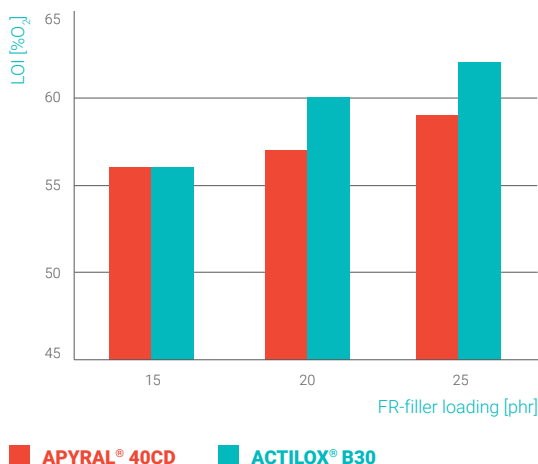


Figure 15: Influence of filler type and filler loading on LOI of rigid PVC compounds (K = 65, 2.5 phr Ca/Zn stabiliser).

combination with their high temperature stability is responsible for its good processability.

At the same extrusion conditions, the compound output of 20 phr **ACTILOX®** filled formulations was 10 % higher compared to **APYRAL® 40CD** and also compared to calcium carbonate ($CaCO_3$) at same loading.

The $CaCO_3$ filled compound was run to compare the colour of these very simple rigid PVC formulations. It was found that **ACTILOX® B**, in addition to its excellent flame retardant properties gives very white extrudates. The following diagram (figure 16), compares the Yellowness Index (YI) of boehmite filled compounds with $CaCO_3$ (a European window profile quality) filled compounds.

No pigment was used to discriminate the intrinsic colour of the FR-filler. In contrary to the very white **ACTILOX® B30**, extrudates made with **APYRAL® 40CD** showed yellowish to brownish colour.

Due to its excellent processability, its good flame retardancy according to LOI and its high whiteness it is recommended to use **ACTILOX® B30** as flame retardant filler for rigid PVC sheets or profiles.

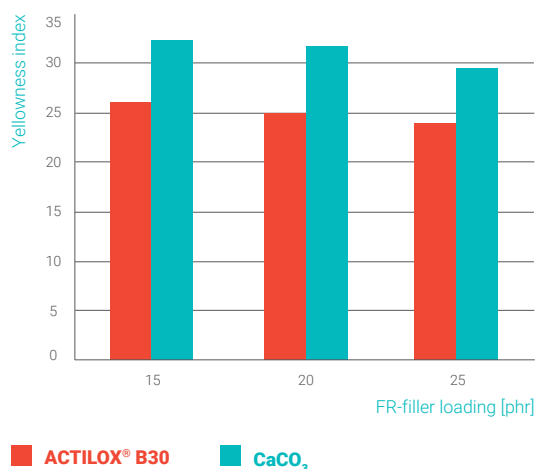


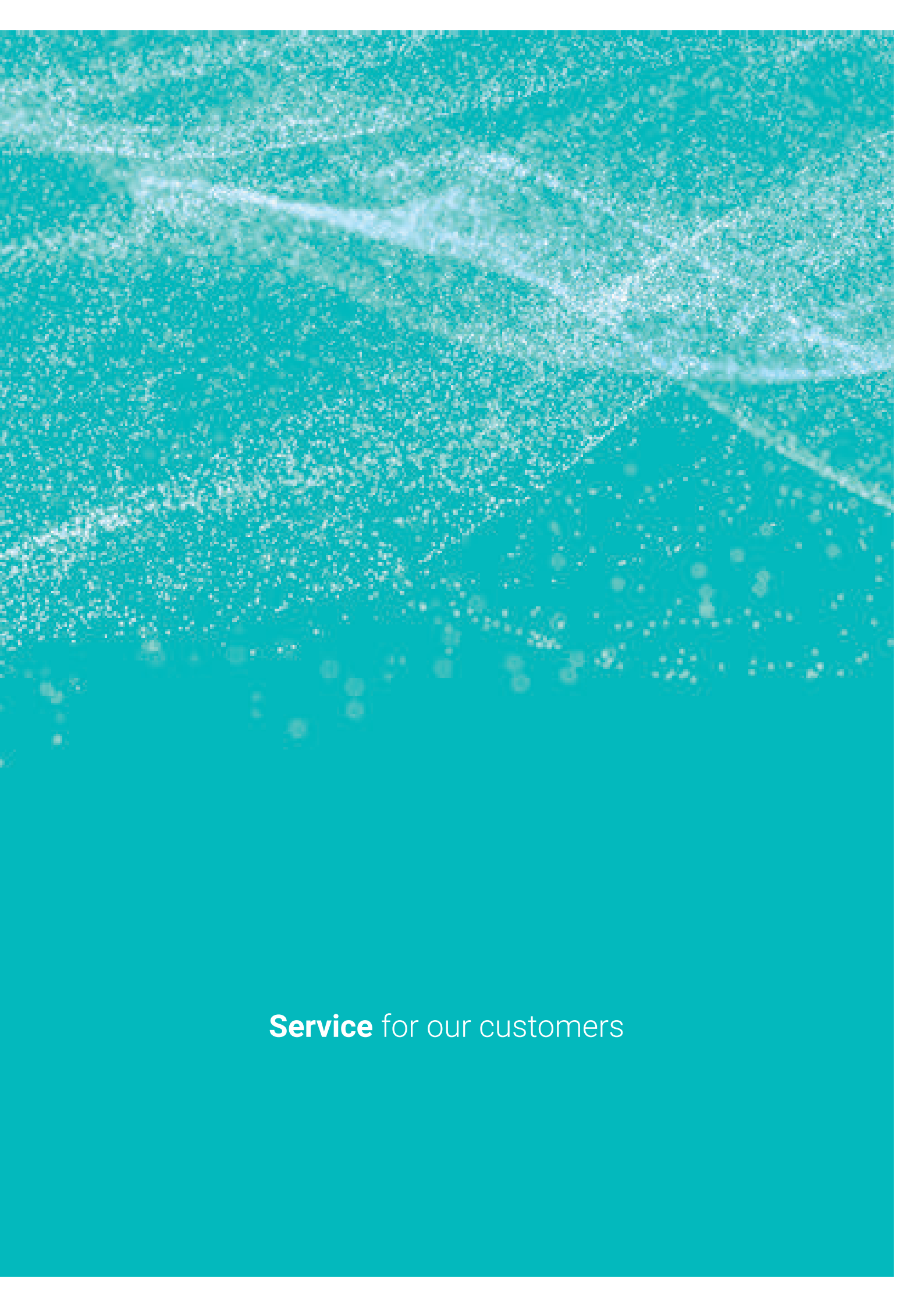
Figure 16: Influence of filler type and loading level on Yellowness Index of rigid PVC compounds (K = 65, 2.5 phr Ca/Zn stabiliser).

Annex



Annex

Abbreviation	Meaning
AOH	Aluminium oxide hydroxide, aluminium monohydrate, boehmite
ATH	Aluminium trihydrate, aluminium hydroxide, gibbsite
ATO	Antimony trioxide, Sb ₂ O ₃
BET	Specific surface area according to Brunauer, Emmett, Teller
BR	Butyl rubber
Br-FR	Brominated flame retardants
c _p	Thermal / heat capacity
CPD	Construction products directive
CPR	Construction products regulation
d	Day
D50	Median particle diameter/size
DHC	Dehydrochlorination
DEHP	Di-ethyl-hexyl-phthalates
DIDP	Di-iso-dodecyl-phthalate
DINP	Di-isononylphthalate
DOP	Di-octyl-hexyl-phthalates
E@B	Elongation at break
ESBO	Epoxidised soy bean oil
FR	Flame retardant
FRLS	Flame retardant low smoke
HCl	Hydrogen chloride
HRR	Heat release rate
K	Viscosity index for PVC
LOI	Limiting oxygen index
MVR	Melt volume rate
NBR	Nitrile butadiene rubber
PE	Polyethylene
phr	Parts per hundred of resin
PHRR	Peak of heat release
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl chloride
SEM	Scanning electron microscope
SRR	Smoke release rate
T _g	Glass transition temperature
TS	Tensile strength
YI	Yellowness index



Service for our customers

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®

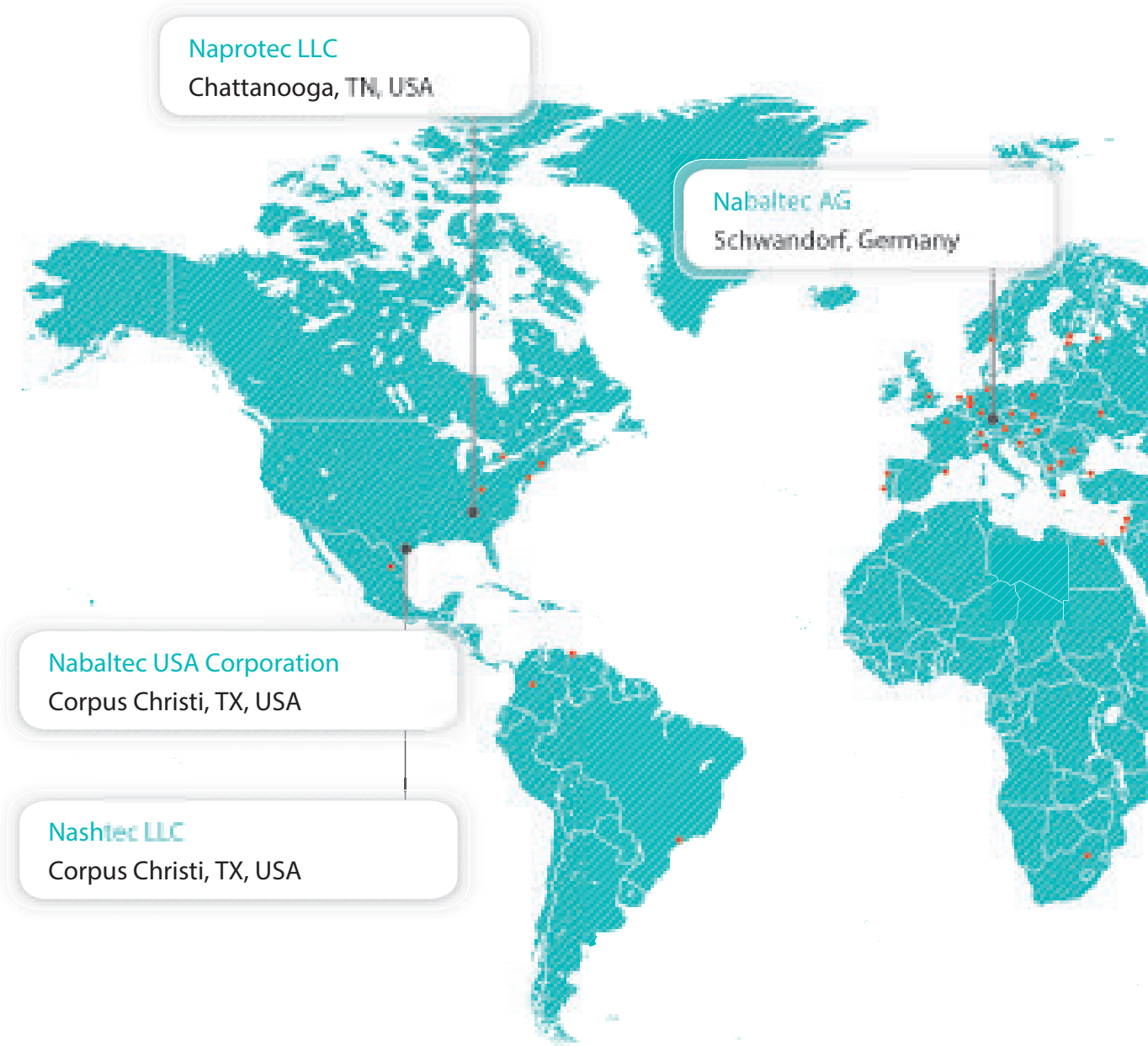
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All data listed in this brochure are reference values and subject to production tolerance. These values are exclusive to the product description and no guarantee is placed on the properties. It remains the responsibility of the users to test the suitability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

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