SILANES | ORGANOFUNCTIONAL

FOR POWERFUL CONNECTIONS
YOU’VE NEVER WORKED LIKE THIS WITH SILANES
WACKER organofunctional silanes, marketed under the trademark GENIOSIL®, include our established standard silanes as well as numerous specialty silanes that will be of use in a variety of new application areas.

We encounter industrial products containing organofunctional silanes every day, and without them, the world as we know it would be hard to imagine. As an important component of paints and varnishes, organofunctional silanes ensure that they adhere to a variety of substrates and last for many years. Adhesives containing silanes can replace rivets and bolts, while silane-modified fillers and glass fibers reinforce plastics, thus making them suitable for a huge range of applications.

WACKER has been actively researching and developing organofunctional silanes for decades. Chemically and technologically, they are closely related to silicones, which have always constituted one of our core businesses. Our experience in these fields, together with our expertise in organic fine chemicals, enables us to continuously develop innovative processes and products in these key technologies.

Today, WACKER produces a broad range of organofunctional silanes and markets them under the GENIOSIL® trademark. The product portfolio includes not only the established, standard products, but also novel molecules with unrivaled properties. These open up completely new fields of application.

This brochure provides an overview of the organofunctional silanes produced by WACKER. Special importance is given to the α-silanes.

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Organofunctional silanes act as molecular bridges between organic polymers and inorganic materials. These hybrid compounds combine the functionality of a reactive organic group and the inorganic functionality of an alkyl silicate in a single molecule. This special property means they can be used as molecular bridges between organic polymers and inorganic materials.

The choice of functional group $X$ will depend ultimately on the nature of the organic polymer. Commercially available silanes contain amino, epoxy and glycidoxy, mercapto and sulfido, isocyanate, methacryloxy and vinyl groups. How these functional groups bind to the organic polymer is described in detail on pages 6 and 7.

Bonding to the inorganic material is effected by way of the hydrolyzable functional groups –OR. The most common of these are the methoxy and ethoxy substituents. The silane’s alkoxy groups hydrolyze in the presence of moisture to form reactive silanols. These either react with themselves to form oligomeric siloxanes, or else react with the inorganic substrate. Since hydroxyl groups are present on almost all mineral and metallic surfaces, the silane will generally undergo a condensation reaction and bond covalently to the inorganic substrate.

The bridge-building property of organofunctional silanes is particularly important in three application fields:
- Adhesion promotion
- Surface modification
- Polymer crosslinking
MOLECULAR BRIDGES FORGE STABLE BONDS

Fig. 2: Adhesion promotion
Organofunctional silanes enhance adhesion of coatings, adhesives and sealants to a wide variety of substrates.

Fig. 3: Surface modification
Organofunctional silanes improve compatibility and enable inorganic fillers to bond chemically to organic resins.

Fig. 4: Crosslinking
Organofunctional silanes function as crosslinkers for organic polymers, e.g. in moisture-curing adhesives and sealants, in paints and varnishes and in thermoplastics.
The particular function and the nature of the organic polymer will determine the choice of silane as well as the way in which it bonds chemically to the polymer.

The mechanisms by which organofunctional silanes bond to organic polymers are as varied as the polymers themselves. The most important mechanisms are:
- Endcapping
- Radical grafting
- Copolymerization

All three methods produce silane-modified polymers. These polymers can then form stable siloxane bonds via the cross-linking of the remaining Si-alkoxy groups. Likewise, they can also bond to fillers or other inorganic surfaces.

Table 1 is a guide showing which silanes produce the best results in the various polymer systems.

**Fig. 5: Endcapping**

The mechanisms by which organofunctional silanes bond to organic polymers are as varied as the polymers themselves. The most important mechanisms are:
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Table 1 is a guide showing which silanes produce the best results in the various polymer systems.

**Fig. 6: Radical grafting**

In this case, the silane’s organofunctional group reacts with a terminal functional group of the precursor by forming a covalent bond (Fig. 5).

The choice of silane will depend on the polymer. The following combinations are common:
- Aminosilanes + NCO-terminated polyurethanes
- Aminosilanes + acrylic/methacrylic polymers
- Isocyanatosilanes + OH-polymers (polyethers, polyesters, polyurethanes)
- Amino- and epoxy/glycidoxy silanes + epoxy resin precursors
- Aminosilanes + phenol resin precursors
Radical Grafting

Unsaturated silanes, such as vinyl- or methacryloxysilanes, can be made to bond to polymer chains via a radical grafting reaction (Fig. 6):
- Vinylsilanes + polyolefines
- Methacryloxysilanes + polyolefines
- Methacryloxysilanes + unsaturated polyesters

Copolymerization

The polymerizable organofunctional groups of a silane (e.g., methacryloxy- or vinylsilane) are incorporated into the chain during production of the polymer (Fig. 7):
- Methacryloxysilanes + monomeric acrylates/methacrylates or styrene
- Vinilsilanes + monomeric acrylates/methacrylates or ethylene/vinyl acetate

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<th>Polymer</th>
<th>Organofunctional group on silane</th>
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<tr>
<td>Polyacrylate</td>
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<td>Butyl rubber, neoprene, phenolic resin, polyamide, polyether, epoxy resin, melanine</td>
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<td>EPR, EPDM, SBR</td>
<td>Vinyl, Mercapto</td>
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</tbody>
</table>

![Fig. 7: Copolymerization](image)

![Table 1: Compatibility of polymers and organofunctional silanes](table)
The nature of the “bridge construction” determines the application properties. α-Silanes, that is, silanes with a methylene bridge instead of the usual propylene spacer between the silicon atom and the functional group, are highly reactive and accordingly have huge application potential.

Most established organofunctional silanes are trialkoxysilanes with a propylene spacer between the silicon atom and the functional group X (Fig. 8). Dialkoxysilanes (Fig. 9) with a propylene spacer are also available but are of little practical importance compared with trialkoxy-silanes.

The most important functional groups X are the amino, glycidoxy, sulfur and methacryloxy groups. These standard silanes, of which the GENIOSIL® product portfolio includes a broad range, dominate today’s market.

WACKER’s strength is on specialty silanes. The name “specialty silane” implies first, a special choice of highly reactive organofunctional groups (e.g. isocyanate) and second, a relatively novel molecular structure, as emphasized by the methylene spacer or bridge that replaces the familiar propylene bridge.

These innovative silanes, referred to as α-silanes (Figs. 10 and 11), display new properties. Their most important property is without doubt the extremely high reactivity of the Si-alkoxy groups. This results from electron interaction of the functional group X with the Si atom, and it can only be observed in the presence of a spacer. The phenomenon is accordingly referred to as the “α effect.”

Since highly reactive silanes are needed for a wide variety of applications, WACKER has developed a specific portfolio of organofunctional α-silanes. Besides aminosilanes, it includes silanes with methacryloxy, isocyanate and carbamate functional groups.

In contrast to established standard silanes with a propylene spacer (γ-silanes), both trialkoxy (Fig. 10) and dialkoxy α-silanes (Fig. 11) are extremely important building blocks.

The next chapter explains the "α effect" and the huge potential offered by α-silanes.

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**The "α Effect"**

An electronegative donor such as nitrogen or oxygen that is in the α position relative to the silicon atom, i.e. is separated only by a methylene bridge, activates the alkoxy groups on the silicon atom. These groups demonstrate greater reactivity towards nucleophiles, as seen by their faster hydrolysis upon admission of water.

The "α effect" can be described very graphically using α-aminalkylsilanes as an example: back-bonding of the nitrogen's free electron pair to the silicon atom (Fig. 12) weakens the Si-O bonds. This back-bonding becomes stronger with increasing basicity of the nitrogen. The latter, in turn, can be varied selectively via the organic substituents on the nitrogen atom. The "α effect" can be determined quantitatively by investigating the hydrolysis kinetics of such silanes.

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**Practical Consequences of the "α Effect"**

The increased reactivity of the alkoxy groups in α-silanes not only opens up new fields of application for which standard γ-silanes are insufficiently reactive (e.g. for fast-curing adhesives), but also renders them interesting building blocks that offer many advantages in established fields of application.

For example, the "α effect" reduces the difference in reactivity between methoxysilyl and ethoxysilyl groups, thus making it possible to replace methoxysilane by ethoxysilane without impairing the application properties. This is particularly important in applications where the release of methanol, which has a higher order of toxicity than ethanol, is problematic.

Another important advantage of α-silanes is that the bifunctional compounds are also highly reactive, whereas dialkoxy silanes with a propylene spacer are not reactive enough for most applications. Especially in the field of flexible adhesives and sealants, as well as coatings, bifunctional crosslinker silanes have great potential since they permit selective adjustment of the crosslinking density.
Hydrolysis of Unsaturated Silanes

Unsaturated silanes — in particular vinyl- and methacryloxysilanes — can be incorporated via copolymerization or grafting reactions into polymer chains and are therefore important crosslinkers for organic polymers (p. 6/7). In the presence of water, the silane’s alkoxy groups hydrolyze to form silanol groups. Condensation reactions between these silanol groups produce a siloxane network.

The speed of hydrolysis is influenced strongly by ambient conditions, in particular pH and temperature, and also to a considerable extent by the nature of the silane. Fig. 13 shows the results of tests on the hydrolysis kinetics of three different methacryloxysilanes and a vinyl-silane. The tests were conducted in a water/acetone mixture at pH 4 at room temperature and demonstrate the speed at which the concentration of a monomeric silane decreases under such conditions.

The result is impressive: the new α-methacryloxytrimethoxysilane reacts 20 times faster than its γ counterpart. The latter is widely used in paints and varnishes as a crosslinker. Even the bifunctional α-methacryloxysilane hydrolyzes 14 times faster than the standard methacryloxysilane. Its reactivity thus corresponds approximately to that of vinyltrimethoxysilane, which is known for its high reactivity and therefore commonly used as a water scavenger in adhesives and sealants.

These experiments clearly demonstrate the impact of the “α effect” on the reactivity of silanes. The objective now is to capitalize on this effect in the form of corresponding product characteristics.

The “α Effect” in Adhesives and Sealants

The “α effect” is just as pronounced in adhesives and sealants as it is in the hydrolysis of methacryloxysilanes. If, for example, a polyether polyol is reacted with various alkoxy silanes bearing NCO functional groups, thus forming silane-terminated polymers (cf. p. 14: STP technology), the different reactivity of each of the silanes becomes readily evident when the polymers are used in standard sealant formulations and the skin-formation times are determined.
As Fig. 14 shows, skin-formation begins after just a few seconds when $\alpha$-trimethoxysilane is used, as compared to about 25 minutes in the case of $\gamma$-trimethoxysilane.

A comparison of the ethoxysilanes is even more impressive: $\alpha$-triethoxysilane induces curing in a few minutes whereas with $\gamma$-triethoxysilane no skin-formation can be observed even after 2 hours.

Another hugely important aspect of this application is that the dialkoxy $\alpha$-silane grades, even the ethoxy variants, likewise cure rapidly. Compared to this a corresponding $\gamma$-dimethoxy system would be far out of range (several hours) under the same conditions.

These examples demonstrate the great potential that di- and trialkoxy $\alpha$-silanes have as crosslinkers for moisture-curing adhesives and sealants and for post-curing coating systems. In addition, they are interesting as adhesion promoters, probably the most important field of application for organofunctional silanes in terms of quantities.
Silane crosslinking is a key technology for state-of-the-art adhesives and sealants. GENIOSIL® α-silanes offer unforeseen advantages.

Organofunctional silanes are essential components in almost all adhesives and sealants.

There are two basic methods of incorporating organofunctional silanes into adhesives and sealants: either the silanes are linked chemically to the base polymer by bonding the organofunctional groups with the reactive ends of the polymer (STP = Silane Terminated Polymers technology), or the silanes are mixed into the formulations in the form of additives.

The silanes fulfill various functions:
- They act as crosslinkers in moisture-curing reactive adhesives and sealants.
- They improve the physical and mechanical properties of filled and reinforced formulations.
- They enhance the resistance of the cured products to chemicals.
- They permit good adhesion to a variety of different substrates.
- They ensure good storage stability for formulations and prevent premature curing.
GENIOSIL® as Adhesion Promoter

In order to enhance adhesion, organo-functional silanes must bond chemically or physically to the organic polymer. Since there is a wide variety of adhesive and sealant systems, a large number of organofunctional silanes are needed. The most commonly used adhesion promoters are amino-, epoxy- and methacryloxy-silanes. How much of which silane will ultimately be required will depend on the specific formulations in question.

GENIOSIL® as Water Scavenger

The most commonly encountered water scavenger in adhesive and sealant formulations is vinyltrimethoxysilane. Due to the electron interactions of the vinyl group, the Si-methoxy groups in this silane hydrolyze substantially faster than in saturated aliphatic alkylalkoxysilanes. Any moisture inherently present in the formulation is removed as the methoxy groups hydrolyze (methanol is split off) and the vinylsilane condenses. Only when the latter reaction is essentially complete will the remaining silane building blocks crosslink. The amount of silane added will depend on the water content of the formulation constituents; usually about one percent by weight is required.

For highly reactive adhesive and sealant systems, such as the α-silane-cross-linking polymers described in the next chapter, vinyltrimethoxysilane is only of limited suitability as a water scavenger. However, the carbamate- and methacryloxy-functional α-silanes are an excellent alternative for such systems.

GENIOSIL® as Crosslinker

Numerous polymers can be used in moisture-curable systems by incorporating silanes. Silane crosslinking is especially important for acrylates, polyethers, polyurethanes and polyesters as a means of selectively optimizing and customizing their properties. These include mechanical properties, such as tensile strength at break, elongation at break, tear strength and abrasion resistance but also storage stability, chemical resistance and weathering resistance. In connection with polyethers and polyurethanes, in particular, silane crosslinking has attracted much attention during the last few years, thereby opening up attractive fields of application for α-silanes. This technology is described in more detail in the next chapter.
There are two principal reasons for modifying polyurethanes with GENIOSIL®: first, to replace toxic and thus problematic isocyanate groups and, second, to improve such properties as adhesion, chemical resistance and storage stability.

Silane-terminated polyurethanes (subsequently referred to as STP-U) are usually produced by the reaction of NCO-containing prepolymers with aminosilanes (Fig. 15).

A disadvantage of STP-U sealants is the high viscosity of the polymers, which results from the urea units formed during silane endcapping reactions. Urea is known for its tendency to form hydrogen bridges resembling ladder rungs between the polymer strands. These high viscosities must be taken into account during the compounding and processing of such systems.

Viscosity can be kept under control if NCO-functional silanes are used instead of aminosilanes, and are reacted with hydroxyl-terminated polymers, such as polyethers, polyurethanes or polyesters (Fig. 16).

**STP Technology**

![Fig. 15: Synthesis of STP-U from NCO-prepolymers and aminosilanes](image1)

![Fig. 16: Synthesis of STP-E from polyethers and NCO-silanes](image2)
Processing is particularly easy when pure polyethers are used (subsequently referred to as STP-E), since the viscosity of these systems is significantly lower than that of STP-U. Special polyethers are needed to achieve mechanical properties comparable to polyurethanes.

Greater detail on WACKER’s silane terminated polymers is available in a separate brochure “Another step ahead with GENIOSIL® STP-E binders.”

The Unparalleled α-Silanes

The new, highly reactive GENIOSIL® α-silanes bring exceptional benefits for STP-E technology.

Adhesives and sealants terminated with these α-silanes cure completely and rapidly, and display outstanding mechanical properties. This is currently the only technology available where formulations are possible that do not require toxic tin catalysts.
## PRODUCT OVERVIEW GENIOSIL®
### ORGANOFUNCTIONAL SILANES

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## Product Overview

### Non-Functional Silanes

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<td>Silan M1-Trimethoxy Methylalkoxy</td>
<td>Methyltrimethoxysilane</td>
<td>(CH₃O)₃SiCH₃</td>
<td>136.2</td>
<td>0.95</td>
<td>11</td>
<td>102</td>
<td>[1185-55-3]</td>
<td></td>
</tr>
<tr>
<td>Silan M1-Triethoxy Methylalkoxy</td>
<td>Methyltriethoxysilane</td>
<td>(C₂H₅O)₃SiCH₃</td>
<td>178.3</td>
<td>0.89</td>
<td>33</td>
<td>143</td>
<td>[2031-67-6]</td>
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</tr>
<tr>
<td>Silan M2-Dimethoxy Methylalkoxy</td>
<td>Dimethylmethoxysilane</td>
<td>(CH₃O)₂Si(CH₃)₂</td>
<td>120.2</td>
<td>0.86</td>
<td>-11</td>
<td>78</td>
<td>[1112-39-6]</td>
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<tr>
<td>Silan M2-Diethoxy Methylalkoxy</td>
<td>Dimethyldiethoxysilane</td>
<td>(C₂H₅O)₂Si(CH₂)₂</td>
<td>148.3</td>
<td>0.83</td>
<td>13</td>
<td>113</td>
<td>[78-62-6]</td>
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<tr>
<td>Silan M3-Ethoxy Methylalkoxy</td>
<td>Trimethylethoxysilane</td>
<td>(C₂H₅O)₂Si(CH₂)₃</td>
<td>118.3</td>
<td>0.75</td>
<td>&lt;-20</td>
<td>76</td>
<td>[1825-62-3]</td>
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</tr>
<tr>
<td>Silan IO-Trimethoxy Alkylalkoxy</td>
<td>Isooctyltrimethoxysilane</td>
<td>(CH₃O)₃SiCH₂CH(CH₃)₂CH₂CH₂CH(CH₃)₂</td>
<td>234.4</td>
<td>0.90</td>
<td>52</td>
<td>90/13 hPa</td>
<td>[34396-03-7]</td>
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<tr>
<td>Silan IO-Triethoxy Alkylalkoxy</td>
<td>Isooctyltriethoxysilane</td>
<td>(C₂H₅O)₃Si(CH₂)₃</td>
<td>276.5</td>
<td>0.88</td>
<td>&gt;40</td>
<td>236</td>
<td>[35435-21-3]</td>
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<tr>
<td>Silan 25013 VP Alkylalkoxy</td>
<td>Hexadecyltrimethoxysilane</td>
<td>(CH₃O)₃SiCH₂CH(CH₂)₁₅H₃₃</td>
<td>346.6</td>
<td>0.89</td>
<td>122</td>
<td>159/2 hPa</td>
<td>[16415-12-6]</td>
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<tr>
<td>Silan CHM-Dimethoxy Alkylalkoxy</td>
<td>(Cyclohexyl)methyl-dimethoxysilane</td>
<td>(CH₃O)₂SiCH₂CH(CH₂)₅CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂CH₂</td>
<td>188.3</td>
<td>0.94</td>
<td>76</td>
<td>198</td>
<td>[17865-32-6]</td>
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</tr>
<tr>
<td>Silan CP2-Dimethoxy Alkylalkoxy</td>
<td>Dicyclopentyldimethoxysilane</td>
<td>(CH₃O)₂SiCH₂CH₂CH₂CH₂</td>
<td>228.4</td>
<td>0.98</td>
<td>&gt;100</td>
<td>257</td>
<td>[126990-35-0]</td>
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<tr>
<td>Silan P-Triethoxy Phenyl</td>
<td>Phenyltriethoxysilane</td>
<td>(C₂H₅O)₂SiSi(CH₃)₂</td>
<td>240.4</td>
<td>0.99</td>
<td>&gt;40</td>
<td>235</td>
<td>[780-69-8]</td>
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<tr>
<td>Crosslinker ES 23 Acetoxy</td>
<td>Triacetoxyethysilane</td>
<td>OOCCH₃</td>
<td>234.3</td>
<td>1.14</td>
<td>&gt; 100</td>
<td>110/13 hPa</td>
<td>17689-77-9</td>
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</tr>
<tr>
<td>Crosslinker ET 13 Acetoxy</td>
<td>1,2-Bis(triethoxysilyl) ethan</td>
<td>H₂C₂H₄OCOCH₃</td>
<td>354.6</td>
<td>0.96</td>
<td>&gt; 100</td>
<td>118/13 hPa</td>
<td>16068-37-4</td>
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</table>
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All figures are based on fiscal 2012.
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