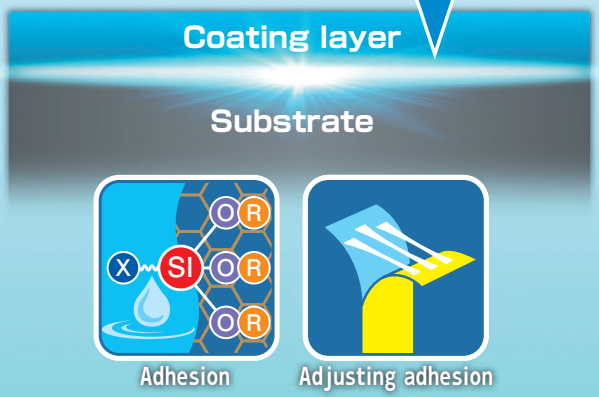
Coating Surface & Interface Modifying Additives

Shin-Etsu Silicone

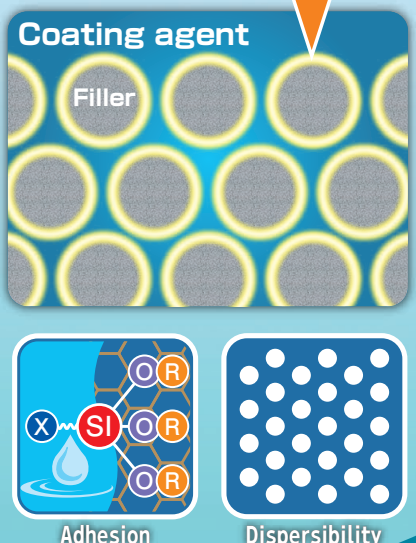
nano tech 2018 International Nanotechnology Exhibition & Conference

Surface Modifying Additives

Interface Modifying Additives (Coating layers × substrates)



Interface Modifying Additives (Coating agents × fillers)



Surface Modifying Additives

What is the KP Series?

■Features

1. Surface free energy: Low

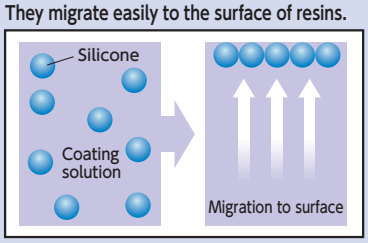
①Silicone moves easily to the surface to effectively improve the surface of resins and coatings.

2. The unique performance of dimethyl polysiloxane structure

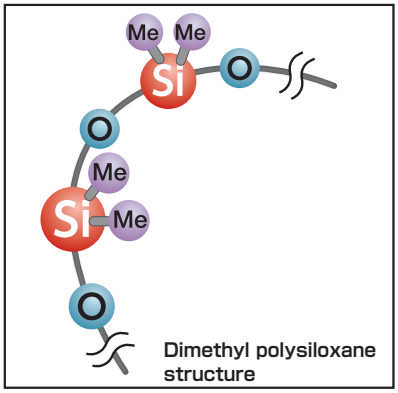
With their ②low surface tension, ③anti-fouling properties, and ④slickness, these products can be used to impart a variety of characteristics.

①Surface migration

They migrate easily to the surface of resins.



■Structure of KP Series



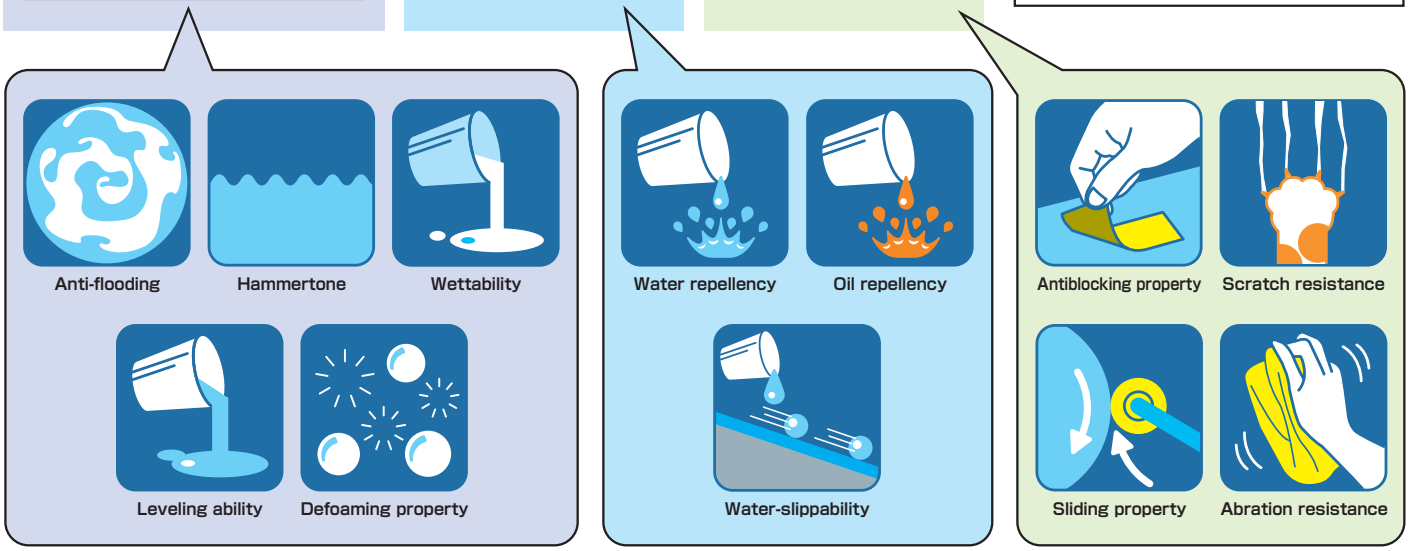
②Low surface tension

Silicone fluids have very low surface tension.

Types of liquid	Suface tension(25℃) mN/m
Dimethyl silicone KF-96	21
Toluene	28
Mineral oil	30
Glycerin	63
Water	72

③Anti-fouling property

④Slip property

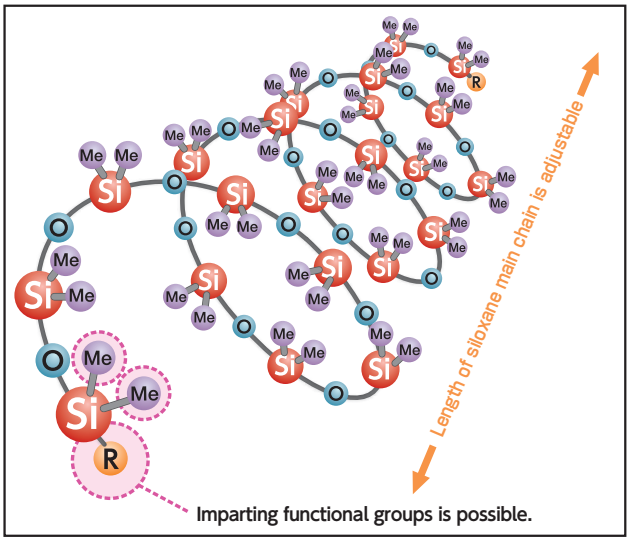


Customization

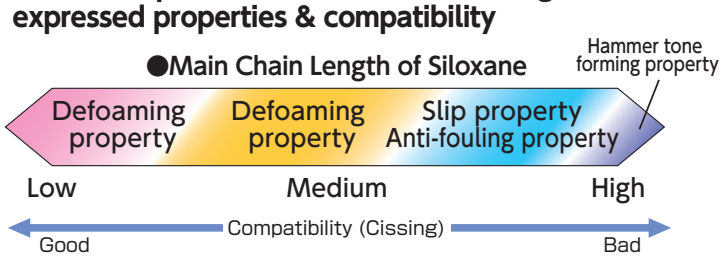
①The strength of the product's properties and its compatibility with particular resins can be altered by changing the length of the siloxane main chains.

②Compatibility and reactivity can be controlled by introducing various functional groups.

→Product can be customized to make it an ideal additive for the user's resin composition.



■Relationship between siloxane chain length and expressed properties & compatibility

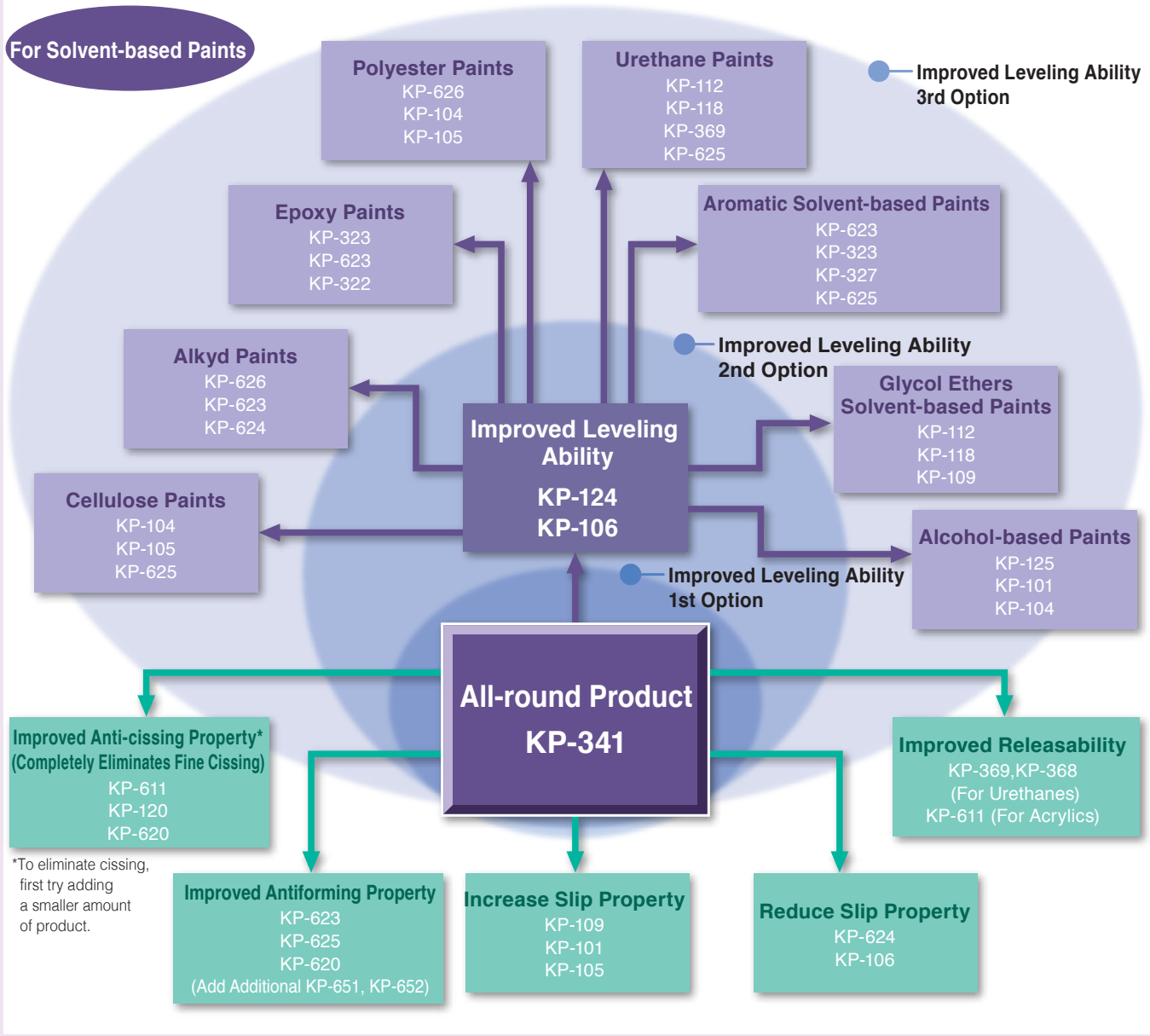


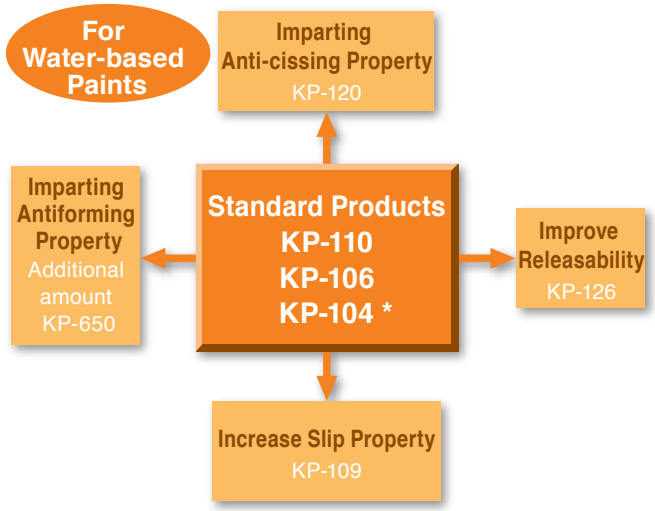
■Imparting functional groups and its effect

Polyether (compatibility, water solubility, recoatability), Polyol (hydrophilicity, high polarity) Acrylic (compatibility with acrylic resins), Fatty acid ester (compatibility with alkyd resins) Phenyl (compatibility, heat resistance), Fluorine (activity in presence of aromatic solvents) Alkyl (lipophilicity), Aralkyl (heat resistance, lipophilicity), Polyester (compatibility with polyester resins)

KP Series Selection Guide, Arranged by Purpose

*This map is offered as a guide for product selection.





* KP-104: Works especially well with water-soluble resins such as Polyvinyl Alcohol.

■Ranking of Slip Agents

KP Series, with radical curable groups

■Features

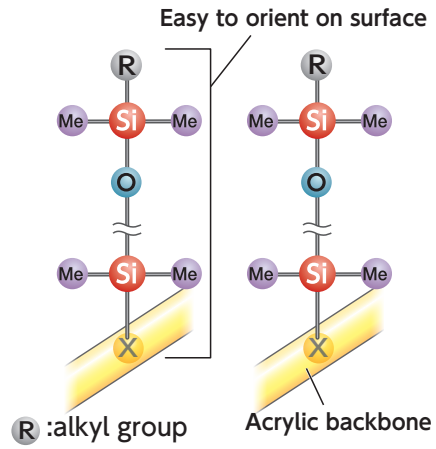
·Functional groups are meth(acrylate) groups.
·Can be added to films and radical-cure coating agents.
·Reactive and imparts long-lasting properties.

■3 product types and conceptual modification diagrams

Three types are available, depending on the position of the functional groups: end-functionalized, double end-functionalized, and side-chain-functionalized. Each type will impart different properties, so the user can select the right product for the intended purpose.

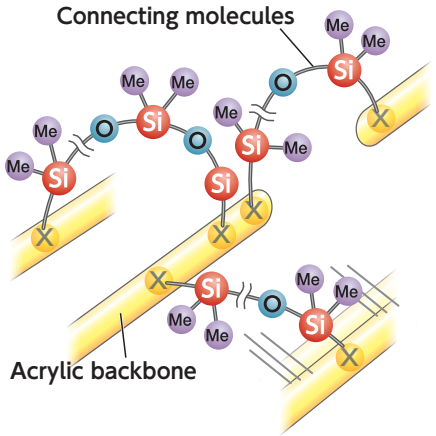
Single-end type

●Benefits
Very effective surface modification



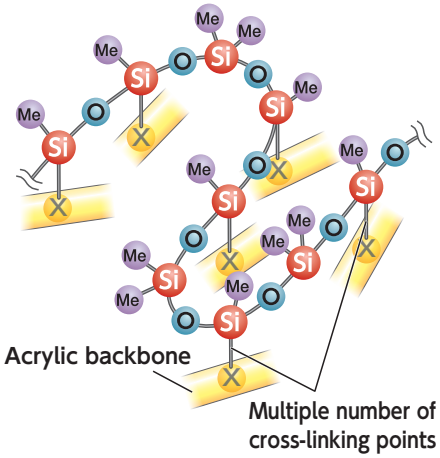
Dual-end type

●Benefits
Effective surface modification
Stress relief



Side chain type

●Benefits
Effective surface modification
Improves crosslinking density (hardness)



■Ranking of features and compatibility

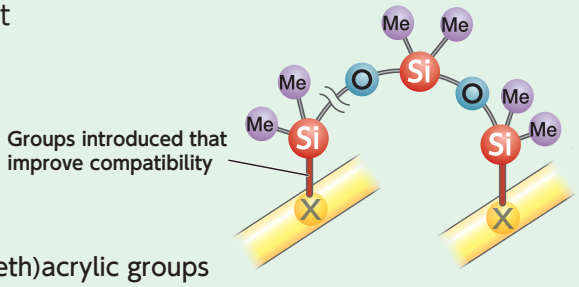
Long silicones (high molecular weight): Silicone characteristics imparted more effectively to the surface.
Short silicones (low molecular weight): Better miscibility with resins.

Product name	Single-end type	KP-422	KP-416				KP-418	
	Dual-end type	KP-410	KP-411	KP-412	KP-413	KP-423	KP-414	KP-415
	Side chain type							KP-420
Features of silicones (Molecular weight)		Low → High						
Compatibility		Good → Bad						

■Features of KP-423

Good compatibility despite the fact that the silicone chains are relatively long.
Benefits :
①Good reactivity, characteristics can be imparted effectively.
② Improved transparency.

■Conceptual modification diagrams



KY-1203: Fluorinated anti-fouling agent with radical curable groups

Water repellency | Oil repellency | Scratch resistance

■General Properties

Parameter \ Product name	KY-1203
Appearance	Transparent to pale yellow
Viscosity at 25°C mm²/s	1.2
Specific gravity at 25°C	0.88
Active ingredient wt%	20
Solvent	MEK、MIBK

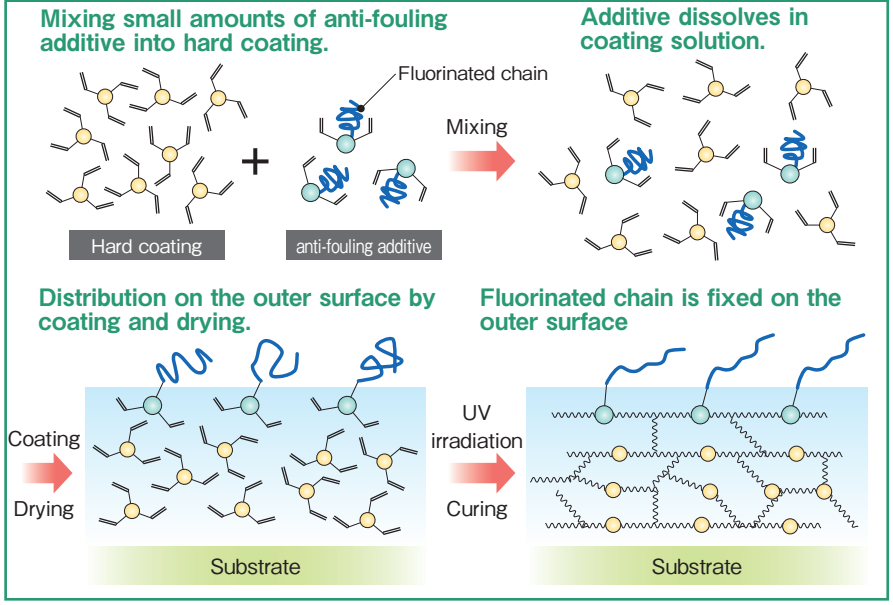
(Not specified values)

■Properties (Hard coated surface)

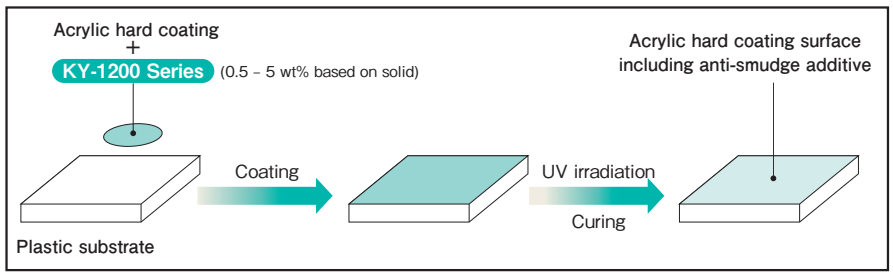
Parameter \ Product name	No addition	KY-1203 added
Water contact angle	59°	114°
Oleic acid contact angle	22°	73°
Oleic acid sliding angle	Not measurable	3°
Dynamic friction coefficient	0.6	0.1

Application example (1wt% addition based on solid)
Hard coating: EBECRYL 40 (made by DAICEL-ALLNEX Ltd.)..... 100 parts
Thinner: 2-propanol ..... 142 parts
Initiator: IRGACURE 184 (BASF Japan Ltd.) ..... 3 parts
Additive: KY-1203 ..... 5 parts
Substrate: Polycarbonate panel
Coating condition: Spin coating: 500 rpm × 10 sec + 3,000 rpm × 20 sec
Preliminary drying: 80 °C × 1 min
Curing apparatus: UV light (metal halide lamp) conveyor system
Irradiation condition: N_2 atmosphere, lamp capacity 80 W/cm, accumulated power 1,600 mJ/cm²

Function mechanism of anti-fouling additives



■Application process

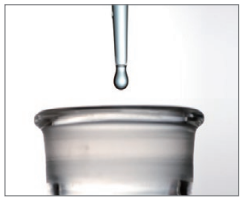


KP-983: Water repellency & slippability improver

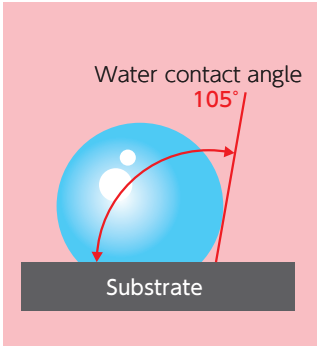
Water repellency | Water-slippability

■Features

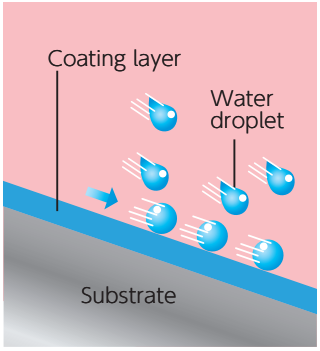
- KP-983 is a water repellency & water-slippability improver.
- Add 1-2% KP-983 to a coating agent to produce a coating with water-slippability.
- Active ingredient: 100% silicone



●Add 1-2% to coating agents.



●Excellent water repellency



●Excellent water-slippability

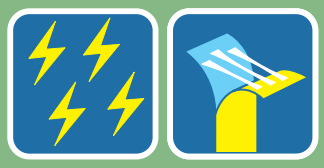
■Water repellency test

Adding to silicone oligomer type coating
【Adding amount】silicone oligomer type coating (KR-400) : KP-983 = 100 wt. part : 1 wt. part

Parameter	Before adding	After adding
Water contact angle*1 °	82	**103**
Water drop angle*2 °	51	**9**

*1 Water droplet volume 2μl *2 Water droplet volume 20μl

(Not specified values)



X-40-2450: Anti-static property improver

X-40-2450 is a silicone oligomer created through the silicone modification of an ionic liquid. When added in small amounts to resins, X-40-2450 migrates easily to the coating surface, improving its heat resistance, and provides long-lasting antistatic properties.

■Resulting Properties

- Excellent antistatic agent
- Imparting releasability

●Antistatic Properties Test Result

Parameter \ Product name		X-40-2450	Ionic liquid*4
Surface resistivity Ω	Initial	4×10^{10}	$>10^{13}$
	After water wiping test*1	1×10^{11}	$>10^{13}$
	After immersion test in water*2	3×10^{11}	$>10^{13}$
	After heating test*3	8×10^{11}	$>10^{13}$

(Not specified values)

- Mix ratio : Dipentaerythritol hexaacrylate / 2-Hydroxy-2-Methyl-1-Phenyl-Propane-1-one / Methyl ethyl ketone / X-40-2450 = 48.8 / 2.4 / 48.8 / 2.0
- Substrate : PET (Cosmo Shine A4300) made by TOYOBO CO., LTD.
- Cure conditions : 600mJ/cm² under a nitrogen atmosphere
- Film thickness : 5μm

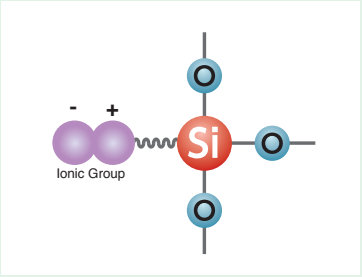
*1 After rubbing the cured specimen 50 times with wet absorbent cotton, wiped off remaining water and took the measurements.
*2 After submerging the cured specimen into water (25°C×5h), wiped off remaining water and took the measurements.
*3 Measured after heating the cured specimen (105°C×1 day).
*4 $(n\text{-}C_8H_{17})_3(CH_3)N^+(CF_3SO_2)_2N^-$

■General Properties

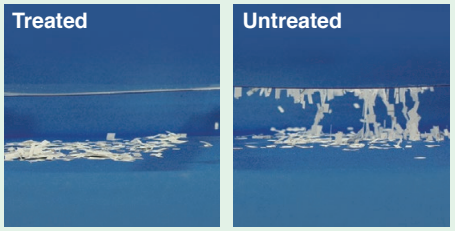
Parameter \ Product name	X-40-2450
Form of silicone	Siloxane
Appearance	Colorless transparent liquid
Non-volatile content %	55
Viscosity mm²/s	2.5
Specific gravity 25°C	0.97
Solvent	Methyl ethyl ketone
TSCA	Not Listed

(Not specified values)

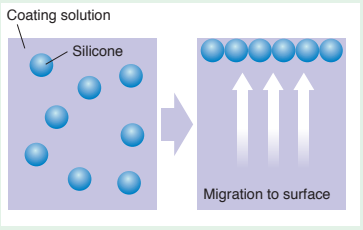
■Structural Model



■Comparison of Antistatic Properties



■Mechanism of Silicone Action



TMPS-E: Light-resistance improver

TMPS-E is a silane coupling agent that contains photostabilizing groups. TMPS-E neutralizes free radicals formed through exposure to light, thus protecting resins against degradation.

■Resulting Properties

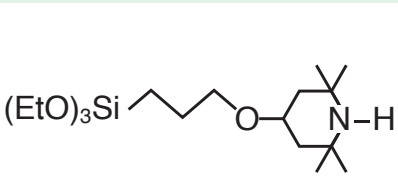
- Improves UV resistance
- Improves adhesion

■General Properties

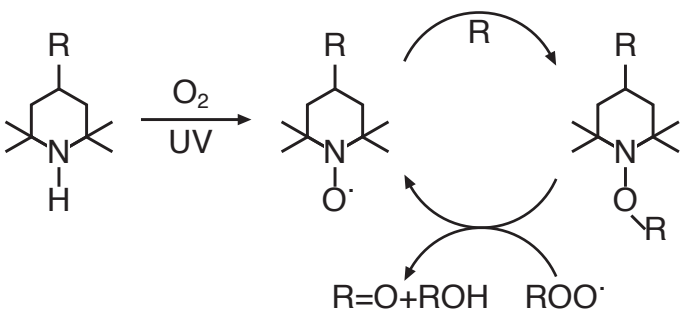
Parameter \ Product name	TMPS-E
Viscosity at 25°C mm²/s	8.0
Specific gravity at 25°C	0.95
Refractive index at 25°C	1.44
Active ingredient %	100
TSCA	Not Listed

(Not specified values)

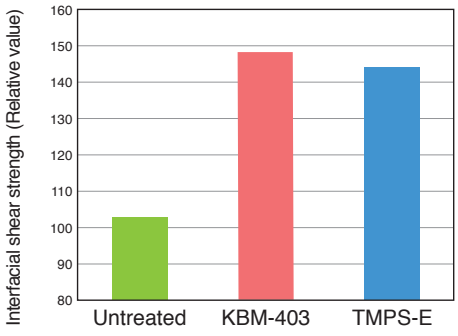
■Chemical Structure of TMPS-E



■Reaction Mechanism



■Glass Adhesion Test Data / Epoxy Resin Interface



KP-912, KP-913, KP-914: Hydrophilicity improvers

Hydrophilicity

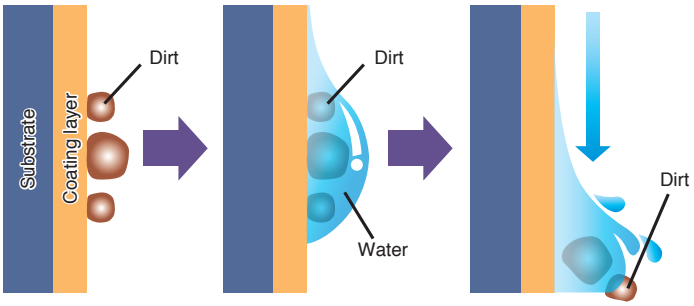
KP-912, KP-913 and KP-914 are silicone oligomers that contain alkoxysilyl groups. When mixed with water, alkoxysilyl groups hydrolyze to form hydrophilic silanols groups, properties which allow these products to function as antifouling agents in paints for construction materials. KP-913 shows its hydrophilic properties earlier.

■General Properties

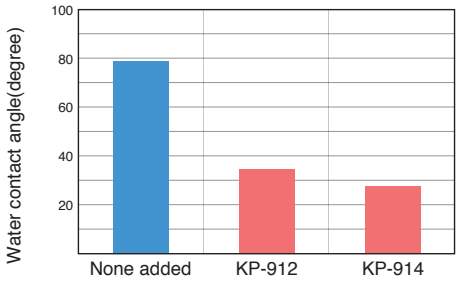
Parameter / Product name	Alkoxy Groups	Viscosity at 25°C mm²/s	Refractive index at 25°C	Alkoxy group content wt%	TSCA
KP-912	Methoxy / Ethoxy	12	1.414	50	Not Listed
KP-913	Methoxy	350	1.448	9.5	Not Listed
KP-914	Methoxy / Ethoxy	20	1.418	50	Not Listed

(Not specified values)

■Antifouling Mechanism



■Hydrophilicity of Coatings with Added Oligomers



Test conditions: Fluoropolymer paint with 5 phr of oligomer
Water contact angle measured
after immersion for 16 h in 2.5% sulfuric acid solution.

X-12-1214A: Anti-rust property improver

Anti-corrosion property

Adhesion

X-12-1214A contains a common corrosion inhibitor (benzotriazole) plus an alkoxysilyl group. As a result of improved adhesion to metals, X-12-1214A helps ensure long-lasting protection against corrosion.

■Resulting Properties

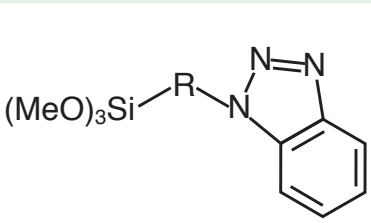
- Protects metal against corrosion (Especially for copper, silver and aluminum)
- Imparting metal adhesion

■General Properties

Parameter / Product name	X-12-1214A
Viscosity at 25°C mm²/s	170
Active ingredient %	100
TSCA	Not Listed

(Not specified values)

■Chemical Structure of X-12-1214A



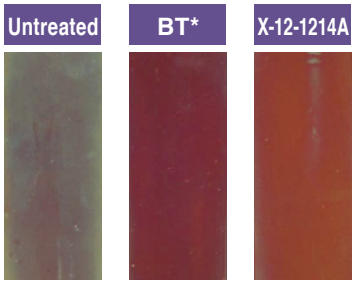
■Anti Rust Treatment on Copper plates

<Specimen preparation>

1. Copper plate is cleaned to remove sulfur and washed with water.
2. Plate is immersed in a 1 wt% solution of benzotriazole or a silane coupling agent for 5 min.
3. Drying

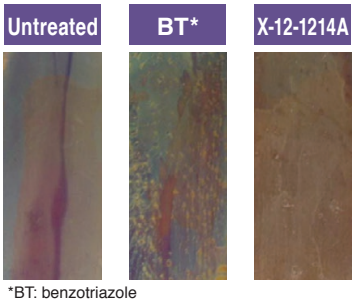
■Heat Resistance Test

1. Plate was left in a constant temperature chamber at 150°C for 5 hours.
2. Copper plate surface was observed.



■Sulfide Corrosion Test

1. Plates were immersed in a 100 ppm Na_2S aqueous solution for 5 min.
2. After drying, plate surface was observed.

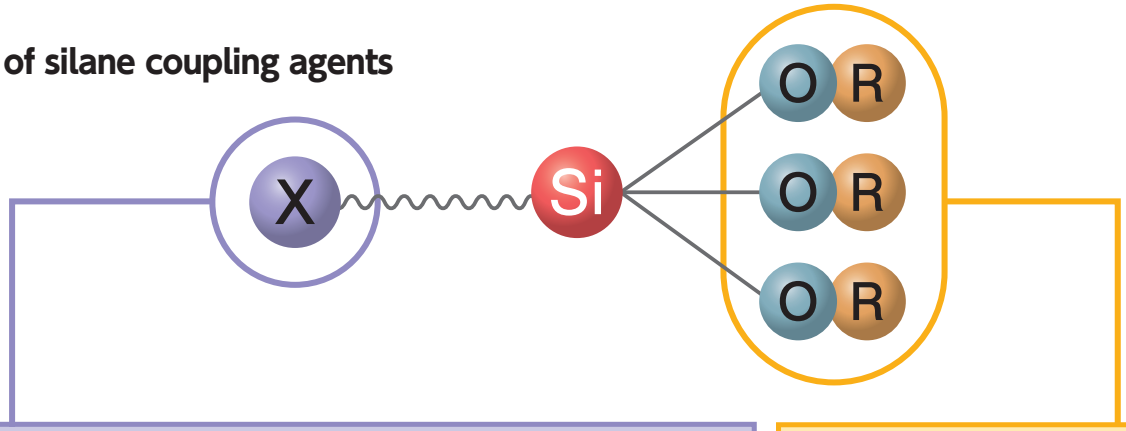


*BT: benzotriazole

What are silane coupling agents?

**In a silane coupling agent, the molecules are functionalized with groups that bond with organic materials and groups that bond with inorganic materials.
Silane coupling agents function to link organic and inorganic materials together.**

■Structure of silane coupling agents



X Reactive groups that form chemical bonds with organic materials such as synthetic resins

●Vinyl groups ●Epoxy groups ●Amino groups
●Methacryloxy groups ●Mercapto groups, other

■Reaction mechanism to organic materials

●Improving wettability
●Improving compatibility
●Forming chemical bonds with resins

RO Reactive groups that form chemical bonds with inorganic materials including glass, metals and inorganic fillers

●Methoxy groups ●Ethoxy groups, other

■Organic Functional Groups and Compatible Resins

Functional groups \ Resin	Thermoplastic resins										Thermosetting resins							Elastomer•Rubber									
	Polyethylene	Polypropylene	Polystyrene	Acrylic	PVC	Polycarbonate	Nylon	Urethane	PBT-PET	ABS	Melamine	Phenolic	Epoxy	Urethane	Polyimide	Diallyl phthalate	Unsaturated polyester	Furan	Polybutadiene rubber	Polyisoprene rubber	Sulfur-crosslinked EPDM	Peroxide Crosslinked EPDM	SBR	Nitrile rubber	Epichlorohydrin rubber	Neoprene rubber	Butyl rubber	Polysulfide	Urethane rubber
Vinyl	++	++														+	+				+	+							
Epoxy	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+					+	+	+		+	+	+
Styryl			+	+																									
Methacryloxy	++	++	++	+		+		+		++						+	++				+	++							
Acryloxy	+	+	+	+		+		+		++						+	++				+	++							
Amino	+	+	++	++	++	+	++	+	+	+	+	++	++	+	+			++			+	+		+		+	+	+	+
Ureide							++					+		+	+														
Mercapto	+	+	+		+			+		+		+	+	+					+	+	++	+	+	+	+	+		++	++
Isocyanate						+	+	++	+	+	+	+	+	++	+			+											+

++: Very effective + : Effective *Not all the functional groups are capable of coupling with the resins in question. This should be taken as a guide.

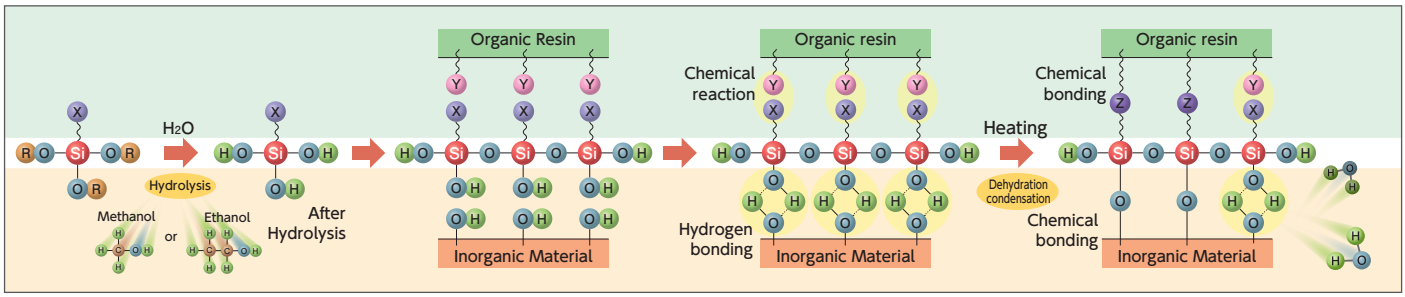
■Features of Hydrolyzable Silyl Groups

Methoxy type: Hydrolyzes rapidly.
Ethoxy type: Hydrolyzes slowly, and compositions will be highly stable even after addition. This type is more eco-friendly, because the product of hydrolysis is ethanol.
Dialkoxy type: Good stability after hydrolysis. Condensation products form straight-chain structures.
Trialkoxy type: High reactivity with high crosslinking density. Strong bonding with inorganic materials.

■Reactivity of silanol

Reactivity	Inorganic material
High	Glass, Silica, Alumina
	Talc, Clay, Mica, Aluminum, Iron
	Titanium oxide, Zinc oxide, Iron oxide
Low	Graphite, Carbon black Calcium carbonate

■Reaction Mechanism of Silane Coupling Agents

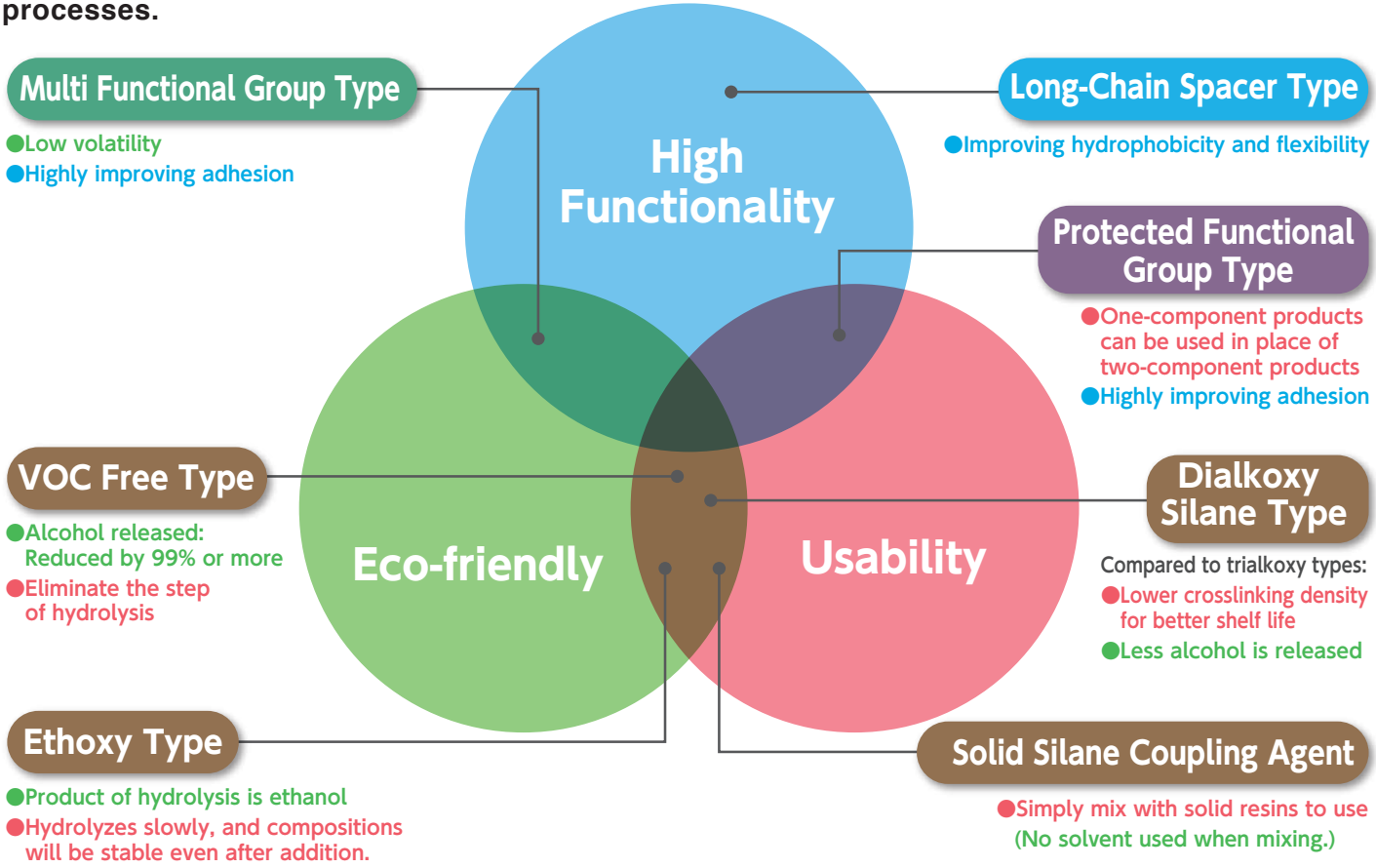


As for the product information in detail, please refer to the Silane Coupling Agents catalog.

Catalog download
https://www.shinetsusilicone-global.com/catalog/index.shtml

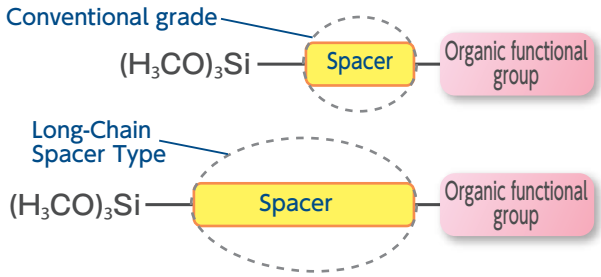
Development Concept of Shin-Etsu Silane Coupling Agents

Shin-Etsu Chemical is developing a range of new products with many special features. Our offerings include products that not only improve functionality but allow users to achieve greener product design, and are easier to use by virtue of allowing users to eliminate certain processes.

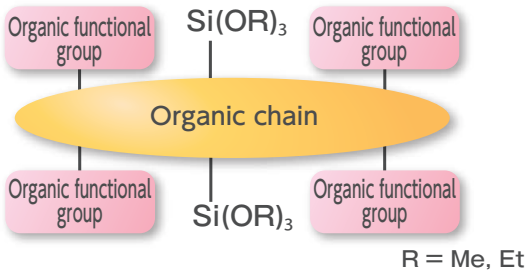


■Model of Chemical Structure

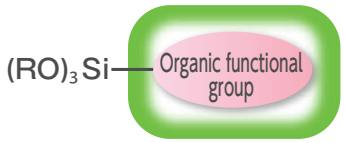
Long-Chain Spacer Type



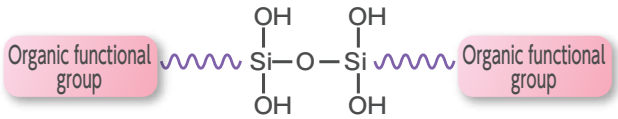
Multi Functional Group Type



Protected Functional Group Type

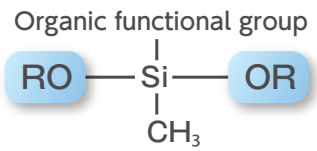


VOC Free Type



Remaining amount of alcohol is up to 1 wt %.

Dialkoxy Silane Type



Ethoxy Type

Dispersibility Improver

Surface Modifiers for Pigments & Fillers

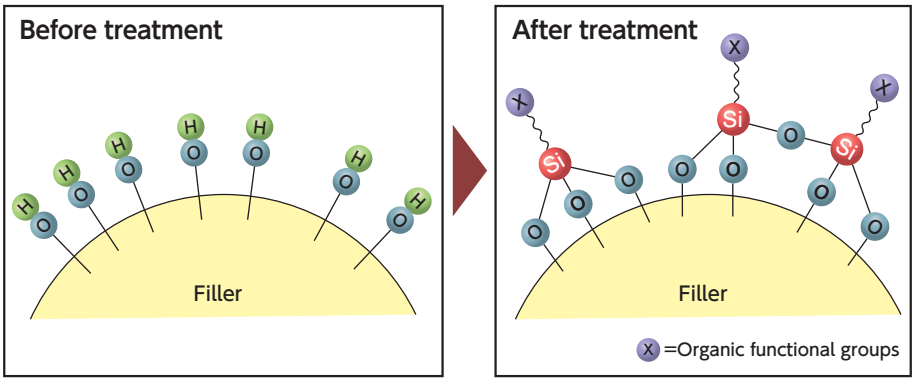
Alkoxy Silanes & Silane Coupling Agents

Silanes and silane coupling agents can be used as surface treatments for pigments and fillers to improve their compatibility with resins and improve adhesion.

■Product List

Product category	Functional group	Product name	Chemical structure	Features	TSCA
Silane coupling agents	Alkenyl	KBM-1003	$(MeO)_3Si$	Vinyl silane, standard product	Listed
		KBM-1083	$(MeO)_3Si$	Long-chain spacer type of KBM-1003	Listed
	Epoxy	KBM-403	$(MeO)_3Si$	Epoxy silane, standard product	Listed
		KBM-4803	$(MeO)_3Si$	Long-chain spacer type of KBM-403	Not Listed
	Methacrylic	KBM-503	$(MeO)_3Si$	Methacrylic silane, standard product	Listed
		KBM-5803	$(MeO)_3Si$	Long-chain spacer type of KBM-503	Not Listed
	Amine	KBM-603	$(MeO)_3Si$	Diamino silane	Listed
		KBE-903	$(EtO)_3Si$	Monoamino silane	Listed
		KBM-6803	$(MeO)_3Si$	Long-chain spacer type of KBM-603	Not Listed
Alkoxy silanes	Alkyl	KBE-3063	$(EtO)_3Si$	Long chain alkyl (C6), ethoxy type	Listed
		KBM-3063	$(MeO)_3Si$	Long chain alkyl (C6), methoxy type	Listed
		KBE-3083	$(EtO)_3Si$	Long chain alkyl (C8), ethoxy type	Listed
		KBM-3103C	$(MeO)_3Si$	Long chain alkyl (C10), methoxy type	Listed
	Fluoroalkyl	KBM-7103	$(MeO)_3Si$	Fluorinated silane. Water repellency, oil repellency	Listed

■Model of Filler Surface Treatment



■Dispersibility of treated silicas

Long-chain spacer silane coupling agents improve the dispersibility of fillers, and compositions will be more transparent.

Formulation : Silane treated silica 10 wt% / Multifunctional acrylic compounds 90 wt%

■Types of Inorganic Materials and Reactivity of Silanol

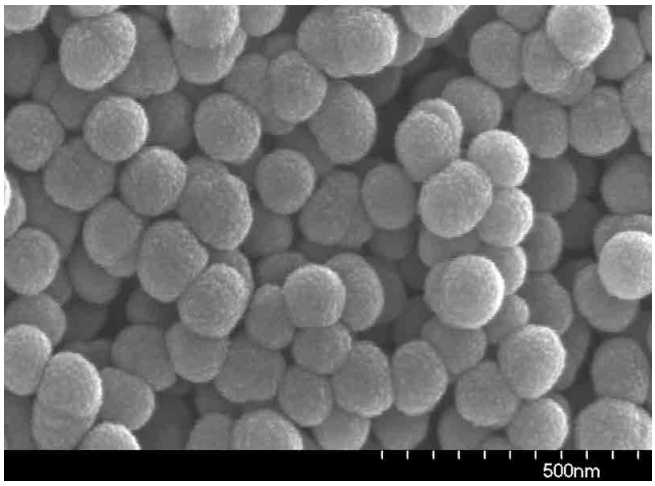
Alkoxy groups hydrolyze to form silanols, which hydrogen-bond to hydroxyls on the surface of inorganic substrates. Typically, Silane coupling agents react more easily with inorganic materials having larger number of active hydroxyl groups on their surfaces.

Number of Hydroxyl Group on the Surface	Large → Small			
Reactivity	High → Low			
Inorganic material	Glass Silica Alumina	Talc Clay Mica Aluminum Iron	Titanium oxide Zinc oxide Iron oxide	Graphite Carbon black Calcium carbonate

Surface Modifiers for Pigments & Fillers

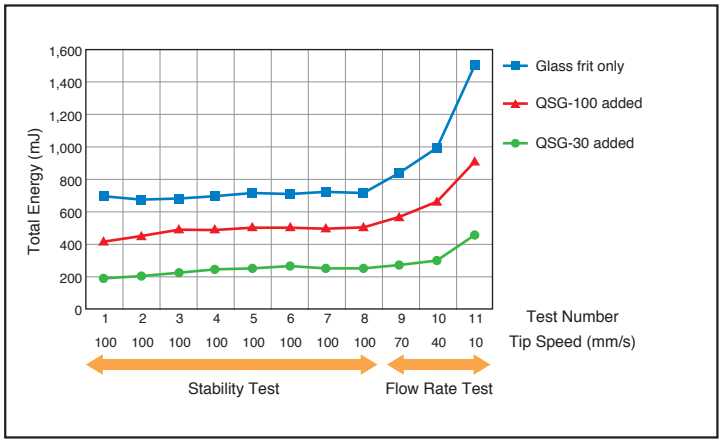
Spherical Silica Fine Particles

Spherical Silica Fine Particles are extremely small and have a narrow particle size distribution. Particle surfaces have been treated to be extra hydrophobic. The particles thus have excellent dispersibility, water repellency, lubricity, flow properties, and can be added to other powders, in a dry process, to improve those powder's performance. Spherical Silica Fine Particles can be used with organic pigments and fillers as well as inorganic ones.



●QSG-100

■Improved Flowability of Glass Frits



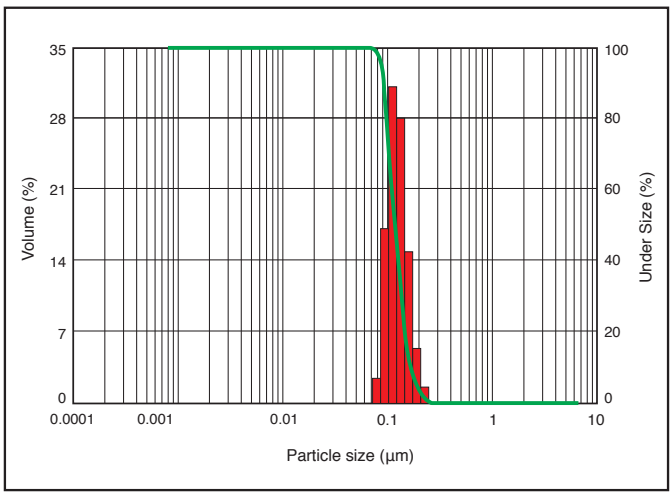
■Product List

Parameter / Grade		QSG-10	QSG-30	QSG-100	QSG-170
Appearance		White powder			
Shape		Spherical			
Average particles size	nm*	10	30	110	170
Bulk density	g/cm³	0.46	0.46	0.44	0.44
True specific gravity		1.8	1.8	1.8	1.8
Specific serface area	m²/g	172	143	25	16
Hydrophobicity, Methanol wettability	%	67	67	67	67
Production method		Sol-Gel			
TSCA		Listed			

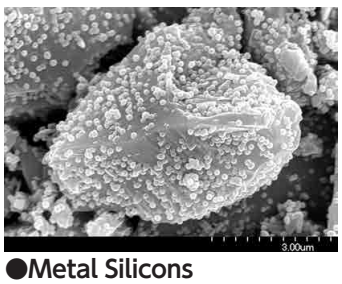
∗ The average particles size measured by dynamic light scattering(Laser Doppler)

(Not specified values)

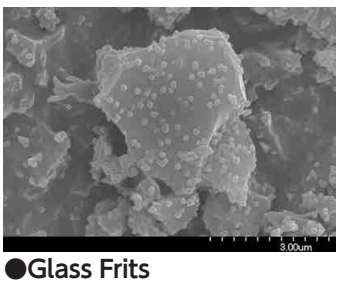
■Particle Size Distribution of QSG-100



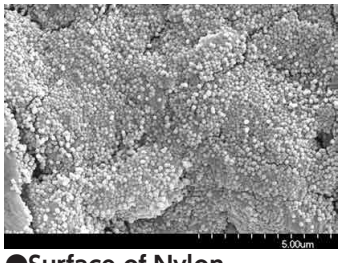
■Adhesion on various surfaces by QSG-100



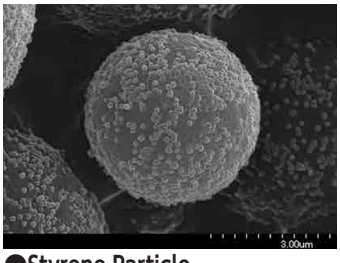
●Metal Silicons



●Glass Frits



●Surface of Nylon



●Styrene Particle

Shin-Etsu Chemical Co., Ltd.

Silicone Division

6-1, Ohtemachi 2-chome, Chiyoda-ku Tokyo, Japan

Sales and Marketing Department I

Phone : +81-(0)3-3246-5132 Fax : +81-(0)3-3246-5361

Sales and Marketing Department II

Phone : +81-(0)3-3246-5131 Fax : +81-(0)3-3246-5361

Shin-Etsu Silicones of America, Inc.
1150 Damar Drive, Akron, OH 44305, U.S.A.
Phone : +1-330-630-9860 Fax : +1-330-630-9855

Shin-Etsu do Brasil Representação de Produtos Químicos Ltda.
Rua Coronel Oscar Porto, 736 11º Andar - 114/115
Paraíso São Paulo - SP Brasil CEP: 04003-003
Phone : +55-11-3939-0690 Fax : +55-11-3052-3904

Shin-Etsu Silicones Europe B. V.
Bolderweg 32, 1332 AV, Almere, The Netherlands
Phone : +31-(0)36-5493170 Fax : +31-(0)36-5326459
Products & Servises : Fluid products

Germany Branch
Rheingaustrasse 190-196, 65203 Wiesbaden, Germany
Phone : +49-(0)611-962-5366 Fax : +49-(0)611-962-9266
Products & Servises : Elastomer products

Shin-Etsu Silicone Taiwan Co., Ltd.
Hung Kuo Bldg. 11F-D, No. 167, Tun Hua N. Rd.,
Taipei, 10549 Taiwan, R.O.C.
Phone : +886-(0)2-2715-0055 Fax : +886-(0)2-2715-0066

Shin-Etsu Silicone Korea Co., Ltd.
GT Tower 15F, 411, Seocho-daero, Seocho-gu,
Seoul 137-856, Korea
Phone : +82-(0)2-590-2500 Fax : +82-(0)2-590-2501

Shin-Etsu Singapore Pte. Ltd.
4 Shenton Way, #10-03/06, SGX CentreII, Singapore 068807
Phone : +65-6743-7277 Fax : +65-6743-7477

Shin-Etsu Silicones India Pvt. Ltd.
Flat No.712, 7th Floor, 24 Ashoka Estate, Barakhamba Road
New Delhi 110001, India
Phone : +91-11-43623081 Fax : +91-11-43623084

Shin-Etsu Silicones (Thailand) Ltd.
7th Floor, Harindhorn Tower, 54 North Sathorn Road,
Bangkok 10500, Thailand
Phone : +66-(0)2-632-2941 Fax : +66-(0)2-632-2945

Shin-Etsu Silicone International Trading (Shanghai) Co., Ltd.
29F Junyao International Plaza, No.789,
Zhao Jia Bang Road, Shanghai 200032, China
Phone : +86-(0)21-6443-5550 Fax : +86-(0)21-6443-5868

Guangzhou Branch
B-2409, 2410, Shine Plaza, 9 Linhexi Road,
Tianhe, Guangzhou, Guangdong 510610, China
Phone : +86-(0)20-3831-0212 Fax : +86-(0)20-3831-0207

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http://www.shinetsusilicone-global.com/

This is an edited version of the product data released on Feb. 2018.
This catalog was published for nano tech 2018