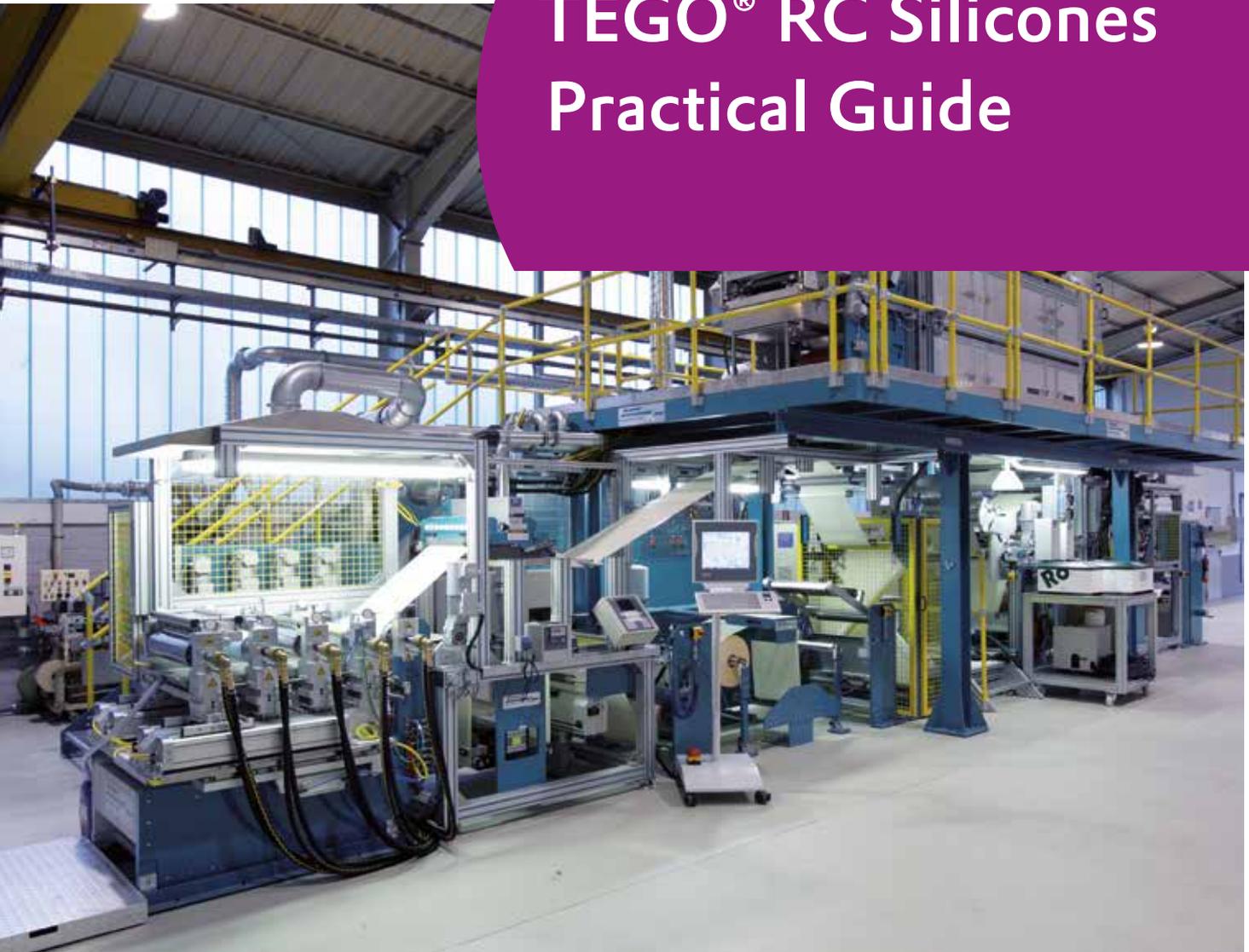


TEGO[®] RC Silicones Practical Guide



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Introduction

FINAT Educational Handbook (excerpt from section 4 "Silicones")

A key feature of all self-adhesive products is the permanently tacky adhesive. It is only possible to produce self-adhesive products and transport them to the point of application, if this tacky surface can be protected. In order that such protection is adequate, and also permits the easy separation of the self-adhesive product from the protective material, a coating of silicone is applied to the contact side of the backing or carrier material. Such a silicone release coating can be cured either by heat, electron beam (EB), or ultra violet (UV) radiation.

The technological development of silicone release coatings began with solvent-based, tin-catalyzed products. The evolving environmental consciousness soon resulted in the demand for solventless products. An attempt to move away from solvents led to silicone emulsions. Silicone emulsions never became a true substitute for solvent systems, as line speeds (water is a "slow" solvent) and corrosion risks were the limiting factors. Another step was development of true solventless 100% solid silicone which still require heavy metal catalysts (platinum or rhodium) and heat to cure the silicone release coating. All the above thermally curing silicone technologies have side effects:

- The high temperatures required for curing change the physical properties of the backing papers and significantly re-

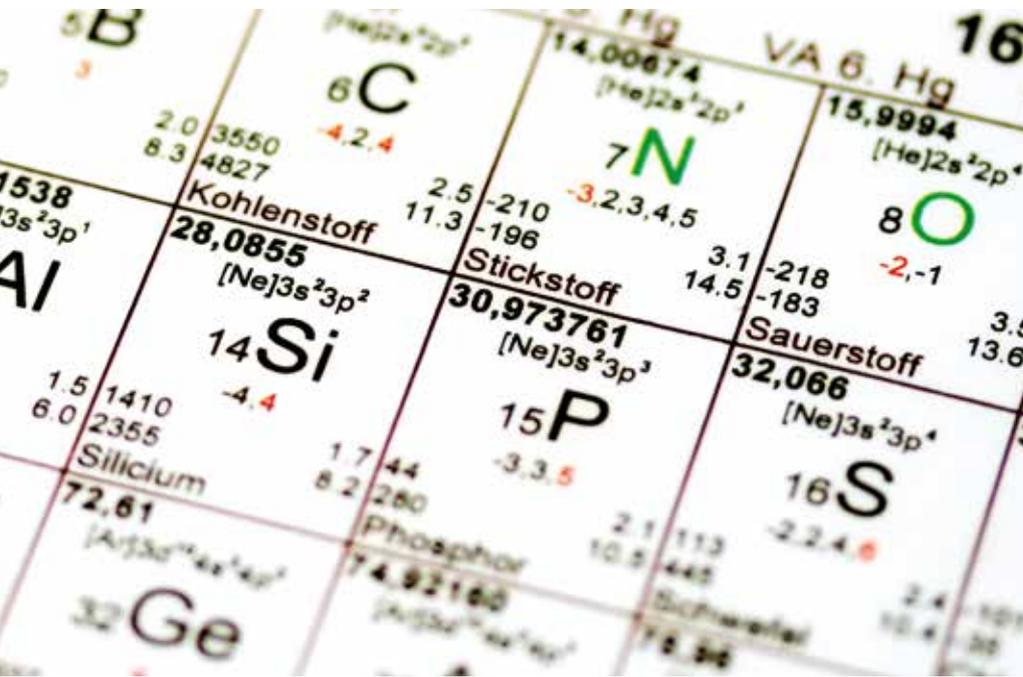
duce the natural moisture content of the paper. This loss of moisture has to be compensated by cost-intensive rehumidification (remoisturization) processes. Due to the heat exposure, the paper often shows poor lay flat behavior also known as curling.

- The high temperature needed to cure thermal silicones limits the use of plastic films, due to their heat sensitivity.
- The fact that all of the above systems require a catalyst for curing, results in the need for paper and film qualities to be free from any substances which could poison (i. e. inactivate, inhibit) the catalyst.

Solventless radiation curable silicones from Evonik are able to eliminate the disadvantages of thermal curing silicones described above.



Technical center in Essen, Germany (front cover), Hopewell USA, Shanghai, China



What are TEGO® RC Silicones?

The term RC Silicones stands for Radiation Curing Silicones. Release liners made with UV curable silicones are growing in popularity. For the last 25 years, Evonik has been an expert in UV curable silicone release systems for the PSA (pressure sensitive adhesive) market.

TEGO® RC Silicones are functional silicone polymers. The functional groups are firmly linked to the silicone backbone. The products are 100% polymeric materials and contain no solvents. UV curing requires the addition of a photoinitiator (PI). There are two UV curable silicone release systems on the market. Both are solventless and produce release coatings without the use of heat, but differ in their underlying chemistry. The first is based on silicone acrylate and cures via a free radical mechanism, whilst the other release system uses epoxy silicones and cures in the presence of a cationic photocatalyst. Evonik offers both free radical and cationic curing silicones.

This guide has been prepared as an aid for line supervisors and operators when using

RC Silicones from Evonik. It covers both our free radical curing silicone acrylates and the cationic curing epoxy silicones. Since the two systems partly show different properties and behavior, you will find one chapter with general information on UV Silicones and the technical details of the two systems in separate chapters.

Whether you are new to siliconizing or you are changing from conventional thermal silicones, this guide will provide you with general information. It is a good starting point to learn about the TEGO® RC Silicones and an excellent source of information for experienced manufacturers.

As always, please feel free to call your technical service teams in the Americas, Asia and Europe if you have any questions. The contact addresses can be found on the back side of this guide. Use this email address for best support: tego-rc@evonik.com

You will find all the regional sales representatives and other valuable information on our web page: www.evonik.com/tego-rc



UV Silicones in General

Storage and packaging

TEGO® RC Silicones are delivered in 200 kg (440 lb.) metal drums or 1000 kg (2200 lb.) UV proof plastic totes. The packaging will ensure that no sunlight reaches the material. Do not store these products in direct sunlight. Exposure to UV light will start the polymerization process. In addition, the packaging will absorb sunlight and heat the product above the recommended storage temperature. Shaded areas or artificial light are not a problem.

Stored at less than 30°C (86°F) and out of direct sunlight, the guaranteed shelf life of 12 months (exception TEGO® RC 1772 which has a shelf life of 6 months) can be extended. This is very dependant on the actual RC Silicone and requires in any case careful testing and qualification before use. When opening the drums or containers, prevent any dust or liquids, such as water or oil from getting into the silicone.

Mixing of TEGO® RC Silicones

It is best to keep silicones separated from all other coating materials. We recommend that the silicones are handled in a specially marked, separate area. Always wear personal protective gear: rubber gloves and eye goggles. In order to avoid spills of silicone on the floor, it is advisable to cover the floor with cardboard or paper. Note: spilled silicone will make any surface extremely slippery.

The silicones can be measured by their weight or volume. Variations of 1–2% silicone in a blend will not have a big influence on release properties. However, the photoinitiator or photocatalyst should be added more accurately, e. g. 2.0 +/- 0.2%.

To use RC Silicones, merely blend the appropriate amounts of RC Silicones needed for the desired release. Add the photoinitiator or photocatalyst as the last component to avoid having this small addition

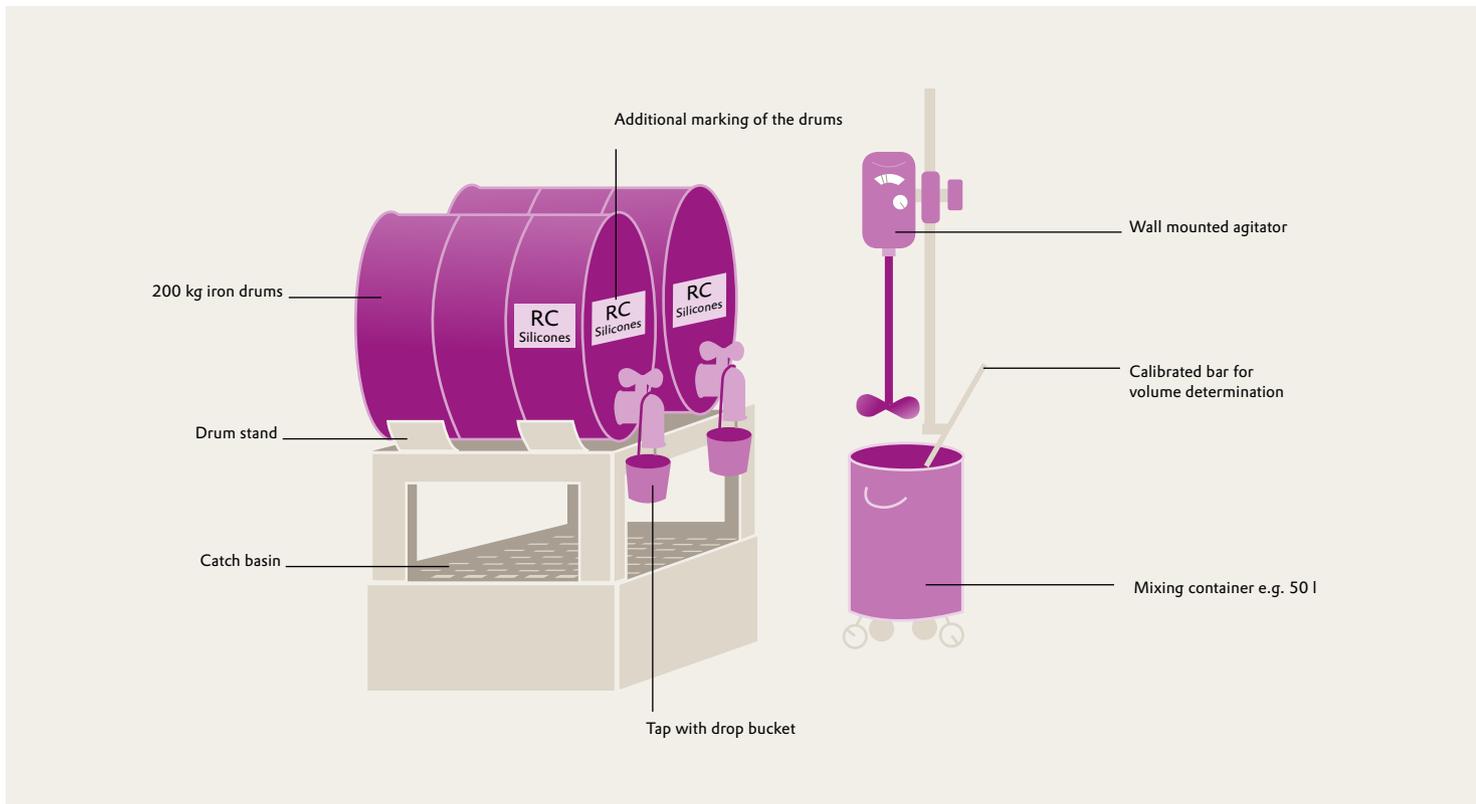
remaining on the bottom of your bucket and not mixed properly. Mix until the blend is homogeneous in color.

The recommended mixing equipment is depending on the daily volumes used. For smaller amounts of silicone up to 30 kgs, a drilling machine with a stirrer may be good enough. For higher volumes we would recommend more professional mixing equipment. However, there are no special requirements to blend TEGO® RC Silicones since they are easily mixable with each other.

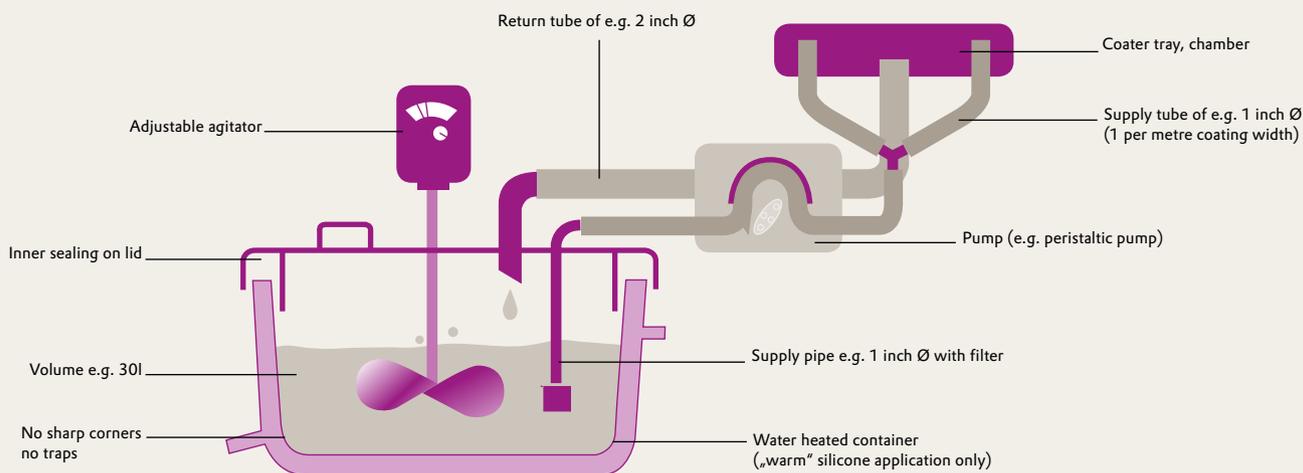
The different RC products have different colors. The color does not affect the quality of the RC product. With most recipes, the mixed silicones will turn turbid after blending. The turbidity of the silicone blend will disappear completely during the UV curing process. The silicone coating will become transparent.

The turbidity is due to the fact, that some of the RC Silicones are not soluble into each other. They are miscible, but will separate on standing. The time for separation is dependent on temperature, the silicone formulation and component ratio. If the mixture is left undisturbed, separation may occur within a few hours. Therefore, continuous stirring in the holding tanks at the process line is advisable. After longer stand-still or storage, separated silicone formulations can be easily remixed.

Also the one-component products TEGO® RC 1002 and RC 1772 will separate on standing, although this needs a much longer time. Therefore, these products should be stirred prior to use. The entire content of the drum must be stirred well before removing any material. To facilitate this, for these products extra headspace has been left in the drums. Tego® RC 702 does not need any stirring and can be used as is.



Example of equipment for medium range silicones consumption of 20 to 200 kg/day



Example of a holding tank for medium range silicone consumption of 20 to 200 kg/day

Silicones at the coater head

Mixed silicones should be transported to the coater head in a closed container or tank. We recommend that the holding tank is located next to the coater head or coating station and is equipped with a stirrer. This helps to keep the blend mixed during the production period. In order to get air bubbles out of the silicone blend, the RC Silicones should be circulated between the coater head and holding tank.

RC Silicone blends range in viscosity from 150 to 3000 mPa*s. Some silicone lines are not equipped to handle viscosities at the upper end of this range. High or inconsistent coat weights and poor coverage may result. Lowering the viscosity is possible by heating **free radical** curing silicone and coating head to 60°C (140°F), which will reduce the viscosity into the range of 150 to 1000 mPa*s. At 60°C (140°F) or below, there will be no risk of gelation on the coater head, but the blend will need to remain agitated in order to prevent separation.

To reduce the viscosity further, you can add special reactive UV curable diluents to the **free radical** silicones. These diluents must be of good silicone solubility to avoid fast separation. The addition of 5 to 10% dodecyl acrylate or multifunctional acrylates is possible. At this addition level, the release properties are influenced to a small but noticeable amount only. However the compatibility of these reactive diluents has to be tested thoroughly with the adhesives of the final application.

Cationic UV silicone formulations often show lower viscosities than comparable silicone acrylates. To avoid gelation on the coater head, they should not be heated to more than 30°C (85°F). Nevertheless it is advisable to control the temperature of the coating head in the range between 20 and 30°C (70 to 85°F). This ensures that the system is not heated up by friction during longer production runs and therefore enables constant coat weights and uniform coating quality.

Coating technologies

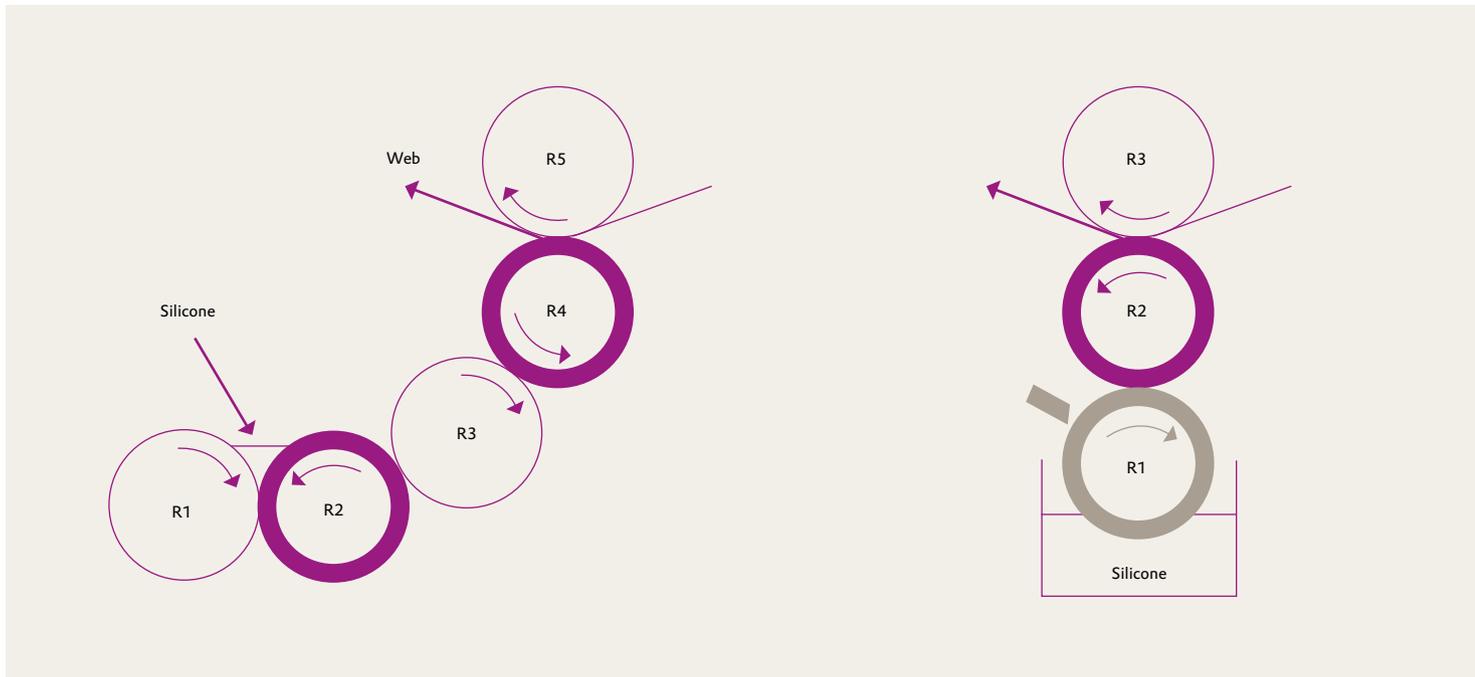
TEGO® RC Silicones can be applied with all current coating techniques such as 5-roll-coater, offset gravure and flexo.

5-roll-coater

For high speed coating and the best coating quality, TEGO® RC Silicones can be applied with a smooth multi roller coating head. Smooth filmic substrates can be siliconized with a silicone coat weight of 0.6 to 0.8 g/m². Rough substrates and paper may require higher silicone coat weights for full coverage.

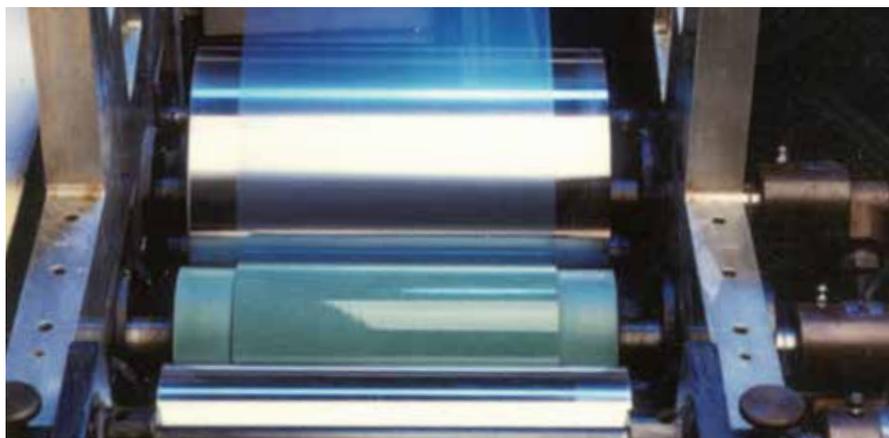
Offset gravure coater

Another common method to apply silicone is offset gravure coating. Smooth filmic substrates can be siliconized with a silicone coat weight of 0.8 to 1.2 g/m². Again, rough substrates and paper may need a slightly higher silicone coating weight for a sufficient surface coverage.



5-roll-coater

Offset gravure coater



Coater head setting for good coverage

For best coating quality, the rubber application roller should have a good balance between footprint (pressure to the web) and differential speed. On paper, a low differential speed (101 – 102% of line speed) and high footprint (depending on diameter and rubber hardness) is preferred. For filmic substrates, the nip footprint should be lower while the differential speed should be higher.



Silicone coating with smooth application roller



Silicone coating with worn application roller

Rubber rollers

The most commonly used roller materials are

- EPDM Ethylene-Propylene-Diene Rubber
- PUR Polyurethane Rubber

The preferred hardness of the applicator for silicone coating both paper and film substrates is Shore A 55 – 75. Softer rollers have a better recovery compared to harder rollers. The preferred hardness of the dosage roll of 5-roll-coater heads is Shore A 75 – 80. The cleaning solvents must be compatible with the rubber roller material you use. If rubber rollers are cleaned with unsuitable solvents, the solvent will swell the rubber. Repeated swelling and shrinking can reduce the diameter and flexibility

by extracting plasticizers and fillers. The silicone can migrate into the rubber and become sticky, which decreases the life span of the roller and harm the coating quality. We recommend isopropanol for cleaning. For PUR, however, you should consult with the manufacturer, which solvents are recommended.

The surface smoothness of the rubber does have a huge influence on coating quality. These microscope pictures (50 times magnification) show silicone coatings made with Shore A 60 applicator EPDM rollers: Left a coating made with a very smooth roller, right a coating employing a roller with a worn surface.



Silicones and printing operations

If you keep the work place clean and the silicones confined, RC Silicones can be used in the same production area, even on the same production machinery as printing inks or overprint varnishes. It is important

to avoid silicone contamination of the printing stations or the printing inks. Silicone contamination can cause print failures, such as pinholes or orange peel effects.

Silicone coverage and release properties

A high quality silicone coating requires good coverage of the substrate. Uncoated areas, pinholes, and exposed paper fibers will increase the release value and give poor release stability over time. In general, we can expect similar release values on all substrates when coating the same release blend.

Air bubbles and foam in the silicone can have an influence on the silicone coverage. This occurs most often with offset gravure coaters, but also happens with 5-roll coaters. We recommend circulating the silicones between the holding tank and coater. This allows the silicones to deaerate. With open pan or 5-roll coaters, it may be useful to discharge these air bubbles by means of a moving bar. This will avoid the formation of areas with high foam/air bubbles, which may transfer to the web and cause stripes in the machine direction.

Prerequisites for consistent release values

- very good silicone coverage
- no interfering additives or migratory ("blooming") components in the substrate
- good silicone anchorage
- same surface roughness of the substrates

Corona treatment

In-line corona treatment just before siliconizing is always recommended when using **free radical** RC Silicones. It can also help to improve the anchorage of **cationic** curing silicones. Corona treatment forms hydroxyl, carboxyl, and free radical groups, which are important for anchorage of the RC Silicones. Corona treatment also influences the spreading behavior of the silicones. High surface tension is not a guarantee for good silicone anchorage, rather it is the presence of reactive molecules. As they disappear quickly, in-line corona treatment is recommended even on high level pre-treated substrates. With most types of

film, in-line corona treatment is recommended for good silicone anchorage. Some PET films generate very high surface tension, and may need only a little or no in-line treatment. On clear films, a good in-line corona treatment will help to coat a transparent silicone coating without blemishes. Flame treatment or extremely high corona treatment of a plastic film can cause low molecular weight polymers to be formed on the film surface, which can cause anchorage problems.

With rough substrates, such as some clay coated papers, in-line treatment

may not be necessary. When using a pre-treated substrate for single side siliconizing, please make sure to in-line treat and siliconize on the pre-treated side. If the non-treated side is silicone coated and wound onto a roll, the silicone coated side of the substrate and the pre-treated side (backside) will be in contact. This contact will influence the release properties and may result in undesirable release values. This is a time-dependant effect and may take months to become noticeable.

Corona treatment when double-side release coating

When coating the second pass of a double-sided release coating, it is important to make sure that the in-line corona treatment is not affecting the first silicone coating. Backside corona treatment can happen with some corona treaters. It is generally caused by a thin layer of air trapped between the treater roll and the film. Even a small amount of backside co-

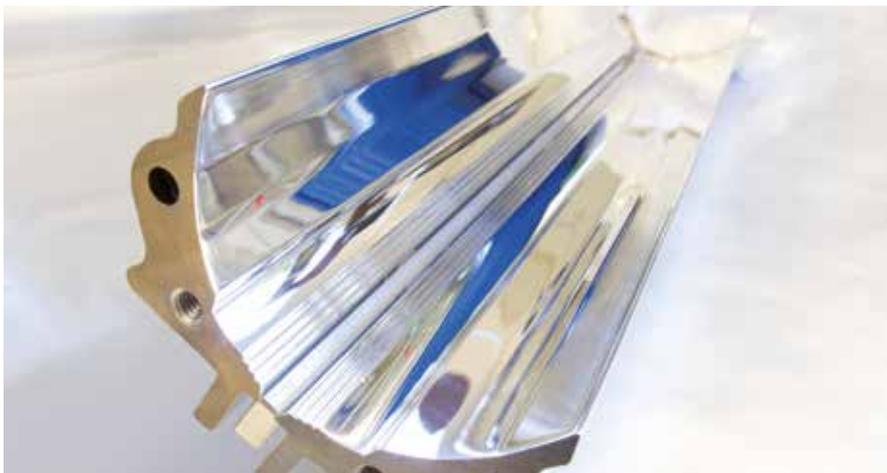
rona treatment can increase the release value of the first pass silicone coating. Usually, back side treatment does not happen uniformly across the web, thus results in stripes or spots.

To avoid backside corona treatment, use a corona treater with a lay-on roll, a tight wrap angle, and a clean, smooth treater

roll. This will help minimize the amount of air being trapped behind the substrate.

When a double side silicone coating is made with differential release, it is advisable to run the tight release side first.





UV power demand and cure speed

Speed of cure is one of the essential key points of our RC technology. Due to the cold curing there is little heat stress to the substrate. Contrary to the thermal silicone systems, the maximum cure speed does neither depend on the type or the gauge of the substrate to be coated nor on the silicone coat weight.

UV lamps are available with outputs of 80 - 240 W/cm depending on the working width. Standard medium pressure mercury lamps are widely used in the UV printing industry. These Hg-lamps have strong emission bands at 250 - 300 nm, which fits best into the absorption spectrum of the commonly used photoinitiators and photocatalysts.

The cure speed of **free radical** curing silicones is very fast. Their UV power demand for a complete cure is very low. One bank of 120 W/cm (300 W/in) lamps can be sufficient to cure at 200 m/min (660 ft/min). Some of our silicone acrylates cure even much faster. As the UV bulbs and reflectors have a reduced UV output over time, it is recommended to overdose a little, e. g. to 160 W/cm (400 W/in) at 200 m/min. The properties of the release coating will not be affected by a higher UV output applied during the curing process.

The reaction speed of **cationic curing** silicones is mainly influenced by the substrate used and by the silicone formulation and therefore can show strong variations. Therefore, cationic curing silicones often re-

quire more UV power than free radical curing silicones. One bank of UV arc lamps with 120 W/cm should be enough to run line speeds up to 100 m/min.

The shape of the reflector has a major influence on cure speed. Trials have shown, that a diffused UV light reaches a higher cure speed compared to focused UV light. Perhaps due to this, we have seen a higher cure speed of both UV silicone systems with standard arc lamps compared to microwave powered lamps. The latter always have a focused reflector system. Best results for free radical curing have been seen with standard medium pressure mercury lamps (so called H-lamps, not doped). For cationic curing, Gallium doped mercury bulbs, so called D-bulbs, may be of some advantage.

Suitability tests

Before using any new silicone formulation, it is recommended to check whether the final product meets all given requirements.

After coating and curing, it is necessary to check whether the cured silicone has the desired performance. This includes

- coverage of the silicone coating on the substrate

- full cure of the silicone coating
- silicone anchorage
- compatibility of the release coating against the adhesives via release ageing tests at low and high temperatures

Post-irradiation may cause a property change in the final product. If the final product is subject to

- electron beam or Gamma irradiation for e. g. sterilization purposes or
- a secondary UV exposure e. g. when curing UV printing inks on label stock with a clear face stock,

the influence on the final product should be tested.



To change between different RC blends

If you use different mixtures of RC Silicones of one and the same system (cationic respectively free radical), they will not influence each other in curing properties, but might have an influence on release properties. If you change from a blend of easy release to a blend of tight release, it will be necessary to clean the

coater head carefully. Small amounts of an easy release blend can reduce the release of a tight release blend considerably.

If you want to produce low and very high release values on the same production line, we recommend having a separate stirrer, holding tank, and piping for each

blend. The coating order for production should be to coat tight release blends first, followed by lower release blends. However, if you are not sure, clean the coater head very carefully in order to avoid any production failures.

Using TEGO® RC Silicones and other type of silicones on the same machine

Thermal, cationic and free radical silicone systems are not compatible with each other; thus contamination of one of the other systems will not cure within the main silicone system. Any residuals of different silicone types left on the coater head, in the holding tank, or in the piping, will remain uncured in the final silicone coating. This

will cause release and subsequent adhesion values to decrease. Therefore, strictly avoid cross-contamination of different silicone systems as the curing mechanisms are not compatible with each other.

When changing between silicone systems, it is important to clean all parts very care-

fully. There may be silicones, especially in the piping and mixing container, which are not easy to be removed completely. Therefore, we recommend having separate silicone mixing devices available for each silicone system you use.

Health and safety

Most of the TEGO® RC Silicones are non-sensitizing; however they are eye irritants. In comparison to acrylic UV printing inks or overprinting varnishes, TEGO® RC Silicones have very moderate toxicological properties, due to their high molecular weight. Nevertheless, we strongly recommend to wear protective gloves and eye goggles. Avoid contamination of skin, clothes, tools or machinery parts with RC Silicones.

During the coating process, TEGO® RC Silicones mist far less than other silicones.

The formation of aerosols (misting) begins at considerably higher coating speeds, typically above 400 m/min (1200 ft/min). If you run high line speeds or aerosols are observed, installation of an appropriate ventilation unit or encapsulation is recommended. Aerosols not only interfere with other coating processes, but they are harmful if inhaled.

For details in health and safety, please refer to our material safety data sheets.

Food contact

There is a certificate from the German ISEGA institute available covering certain formulations of either the free radical RC Silicones corresponding to directive 2002/72/EC. These certificates are for TEGO® RC release coatings that are produced as per our recommendations with regard to application, inerting and cure.

Most of the TEGO® RC Silicone acrylates

are in conformity with the FDA regulations 21 C.F.R. 175.105 („Adhesives“) and C.F.R. 175.125 („Pressure sensitive adhesives“) and have Food Contact Notification FCN 041 or 369.

For details, please contact your technical service team.

Disposal of TEGO® RC Silicones

Residuals of RC Silicones are a chemical waste and need to be disposed in accordance with local, state, and federal regulations. Treat the silicones as chemi-

cal waste, the same as any solvents. If you have any questions about disposing of RC Silicones, please call your local technical service team.

Free Radical

Free radical curing silicones, products & formulations

The free radical polymerization of the acrylate groups is much faster compared to the cationic polymerization. Therefore, full cure is reached more quickly with silicone acrylates. UV curable silicone acrylates are very robust and cure is unaffected by impurities of the substrates. This allows the use of many substrates for silicizing, especially paper substrates. There is no post-curing and no photoinitiator poisoning. The silicone coating is ready

for conversion, in-line adhesive coating or quality testing immediately after curing.

Silicone acrylates need an inert atmosphere (nitrogen) when cured because the presence of oxygen will lead to the termination of the polymerization. Some technical effort is required to meet this necessity for the acrylate system. However, inerted UV units are state of the art and can be built by a number of manufacturers.



TEGO® RC Silicone Acrylates - the basic silicone types

We offer a range of silicones that can be blended to meet the requirements of the intended application:

TEGO® RC Silicone Acrylates

- RC 711 is used for tight releases and to improve anchorage to the substrate. The RC 711-concentration in every free radical formulation should not be lower than 30%.
- RC 902 is used for easy release, and is typically used in a 70/30 blend with RC 711.
- RC 715 is used mainly in applications where controlled release values are required. It is typically used in a 70/30 blend with RC 711.
- RC 709 and RC 719 are controlled release additives (CRA), used with RC 711 and either RC 902 or RC 715 to obtain intermediate release values.
- RC 922 is a premium release additive for adhesives and other sticky materials with low cohesion like bitumen and mastic. In combination with RC 902, it can help to reduce the release level for other applications like label stock.

For UV curing, these silicones need the addition of a suitable photoinitiator.

Besides these individual component silicones, there are some one-component ready to use products that contain both the photoinitiator and anchorage component RC 711.

1-Component TEGO® RC Silicones

- RC 1002 allows siliconizing of slightly porous papers, using low coat weights. This special product counterbalance the absorption effect of alternative (not specially made for silicone coating) papers.
- RC 1772 contains a high amount of large size silica particles. This product was developed to produce matte coatings or to reduce the coefficient of friction of silicone coatings. It can be used as is or blended with other RC silicone formulations.
- RC 702 is especially designed for in-line siliconizing on narrow-web printing machines. It provides easy release for label applications.

For detailed information regarding our product range, please have a look in the Product Data Sheets or contact your local technical service team.

Our recommended photoinitiator

We recommend α -hydroxy-ketones as photoinitiators. Our TEGO® Photoinitiator A18 was specially developed for use in free radical RC Silicones. This photoinitiator is a low viscosity liquid, which blends easily with the RC Silicones. The recommended addition level is one to two parts per 100 parts of silicones.

This photoinitiator can run at high line speeds, has excellent through cure and perfect release stability with our RC Silicones. The UV curing process with this photoinitiator produces almost no volatile by-products that can cloud the quartz plate of the UV unit. This allows the UV equipment to stay clean for a much longer period of time. We recommend cleaning of the quartz plates when they show significant yellowing.

Please note, that other photoinitiators require more frequent cleaning of the UV unit due to outgassing. Also, the cure speed may be reduced when using other photoinitiators.

Inerting

Inerting is the exclusion of oxygen from the surface of the substrate and the area under the UV source. In practical terms, it means introducing nitrogen into a specially designed curing chamber.

Silicone acrylate polymerization is initiated by the formation of free radicals, which are formed by UV irradiation of the photoinitiator. Free radicals are chemical species that have a free electron but no charge. They exist everywhere, including in human bodies. They react quickly with other free radicals, acrylates, and oxygen. In fact, they react so quickly with oxygen that they would react with oxygen molecules rather than the acrylate groups of the TEGO® RC Silicone acrylates, preventing the silicones from curing. Therefore, it is imperative to ensure as much as possible the absence of air-borne oxygen in the curing chamber for the reaction in order to achieve the required release characteristics. The proportion of oxygen remaining on the surface of the substrate as well as at any place in the inerted chamber should not exceed 50 ppm.

Inerting

- Quality of nitrogen < 10 ppm oxygen content
- Residual oxygen content in curing chamber < 50 ppm
- Nitrogen consumption dependent on web width, line speed and substrate

The inert gas recommended for the process is nitrogen as supplied for general technical applications, with a purity level of 99.996 vol. % (quality 4.6 or 5.0). Residues are hydrocarbons, oxygen, argon and other noble gases. For the purging process, the residual oxygen should not exceed 10 ppm. The quality of nitrogen should be discussed well ahead of time with the supplier. An economical way to purchase nitrogen is a liquid nitrogen tank with an evaporator. Both, tank and vaporizer normally are rented on a monthly basis from the nitrogen supplier. The nitrogen tank will be refilled without interrupting production. Nitrogen tanks

and evaporators are available in different sizes and will be adapted to your needs and requirements.

Nitrogen is extremely safe to use. It makes up approximately 80% of the air we breathe (the remaining 20% is oxygen and other gases), and nitrogen is non-toxic. By using nitrogen as the inerting gas, special exhaust systems and safety precautions are not needed. However care must be taken to ensure that outside air is circulated in the working environment. Nitrogen gas is easily available worldwide and inexpensive.

Carbon dioxide (CO₂) has been proposed as an inert gas as well. Unlike nitrogen, CO₂ is heavier than air and would concentrate on the production area floor. Thus, CO₂ is not a safe inert gas and is not recommended for this application.





Nitrogen consumption

Every substrate has a surface boundary layer of air with an oxygen content of approximately 20 vol. %. If the substrate passes through a purged reaction chamber, the oxygen will be lost by diffusion. However, since this diffusion process requires too much time, efficient production would be seriously hampered. It is, therefore, necessary to accelerate the removal of the boundary layer by the use of suitable nitrogen jets or nozzles.

The chamber under the UV lamps is specially engineered to remove the boundary layer of air that is carried along by the moving web. This is accomplished by using a nitrogen "knife" at the front of the chamber. The "knife" provides a laminar flow of nitrogen, which effectively removes the boundary layer, like a real knife peeling an apple. The exit side of the chamber is designed to allow the substrate to pass from the chamber and limit the amount of nitrogen used. Good sealing of the chamber is essential to minimize the consumption of nitrogen and

prevent air (oxygen) from getting inside the chamber.

The preconditions for effective inerting and minimum inert gas consumption are as follows:

- low reaction chamber volume and good reaction chamber seals (gaskets)
- properly sealed quartz plates
- effective barrier nozzle at the entry side
- uniform nitrogen distribution in the chamber
- carefully conceived oxygen monitoring
- good exit-side resistance against nitrogen outflow
- certain interlocks and further options

The overall nitrogen consumption depends on the width of the line and the number of UV lamps, thus the length of the UV unit. In addition, the line speed influences the nitrogen consumption to a great extent. In a UV line with a working width of 1600 mm, a well designed inerting unit will work efficiently on smooth

film substrates with the following quantities of nitrogen:

- at 100 m/min approx. 45 - 60 m³/h,
- at 200 m/min approx. 60 - 80 m³/h
- at 300 m/min approx. 75 - 100 m³/h.

On rougher substrates, replacing the air boundary layer is more challenging than on smooth surfaces. In such cases, the nitrogen consumption for the inerting of paper substrates is expected to be approx. 20 - 50% higher.

Continuous gauging of the residual oxygen content in the reaction chamber is advised for production safety reasons. The oxygen meter can be installed as a passive device or integrated into the control system for the coating line. Depending on the oxygen concentration in the curing chamber it is thus possible to adjust line speed, nitrogen consumption etc. accordingly. Often, the oxygen level reading is linked in the UV control system and stops production when the reading gets too high.



Siliconizing of plastic films

The UV radiation necessary to cure RC Silicones will not increase the web temperature during production significantly. Typically a temperature increase of only 5°C (10°F) is seen, depending on the material, thickness and the colour of the film. A broad range of plastic films down to 3 µm PET (polyethylene terephthalate) or 15 µm LDPE (low density polyethylene) can be coated. When stopping the line, very thin filmic substrates may get hot due to heat released from the quartz glass windows. This may require the web to run at very low speed through the line until the window is cooled down.

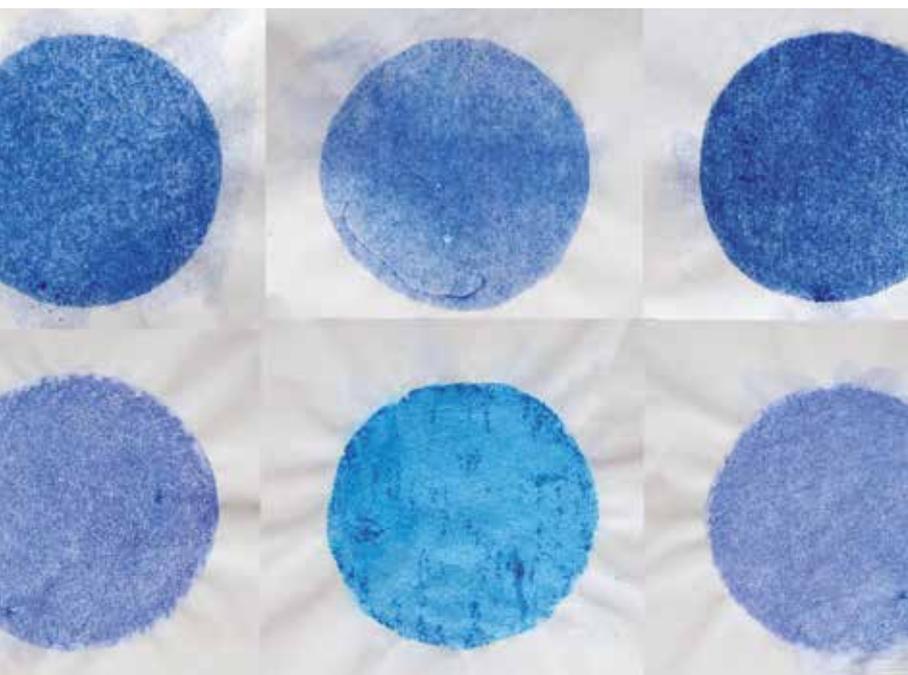
Free radical RC Silicones will cure on all types of substrates. However, there are additives in some plastic films that may in-

fluence the silicone anchorage to the film, e. g. plasticizer in PVC (polyvinylchloride) or slip additives and anti-stats in PP (polypropylene) or PE (polyethylene) films. Silicone anchorage (rub-off) with RC Silicones usually is not dependant on time. If the anchorage is good right after coating and curing of the silicones, the rub-off will not change. A bad rub-off usually stays bad. There are only few exceptions from this rule: In some rare occasions (mainly on PET) the rub-off may improve within the first 24 hours after coating. On the other hand, on soft PVC, the plasticizers might be migratable and cause initially good anchorage to get worse over time.

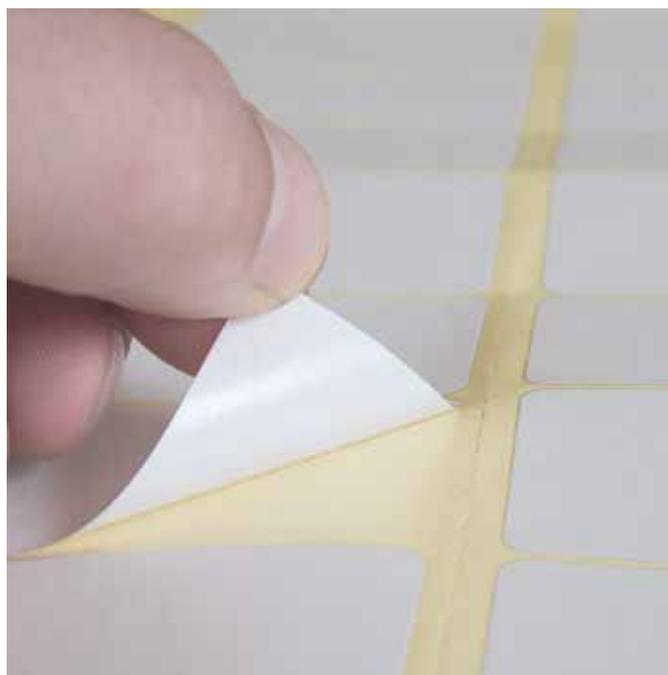
Some migratory (“blooming”) components of the film can travel through the

silicone coating and reduce the release force or give false subsequent adhesion values. Silicone coated soft PVC therefore often exhibits low subsequent adhesion values, as low as 55%, although the silicone coating is fully cured.

If anchorage failure is observed on a particular type of film substrate, a special anchorage additive to the silicone may help. With the addition of an additive, good silicone anchorage can be achieved even on most PVC substrates. Please contact your Evonik technical service team for further details.



Dye stain test of different paper substrates for hygiene applications.



Siliconizing of paper substrates

Siliconizing on paper with free radical curing silicones from Evonik has some special advantages over thermally cured silicones. The silicone acrylates will cure on all kinds of paper, even papers containing additives and/or impurities. Because there is no poisoning of the radical curing mechanism, silicone coating can also be accomplished on thermal sensitive and writing papers. The use of recycled paper grades is possible as well.

Since RC Silicones cure at ambient temperature, there is no heat stress on the paper. The paper moisture will not be reduced during the silicone curing process,

and there is no paper shrinkage. The paper fibers will remain unchanged and retain moisture better than any thermally cured, re-moisturized release liner. The lay-flat behavior of sheet labels, e. g. when used in photocopiers or laser printers, will be excellent. You also may be able to increase speed when applying water based acrylics onto a RC siliconized paper without risking curling of the paper substrate. RC Silicones can be coated on polycoated paper (PE) because blistering of the polycoat is not a concern.

As always, it is necessary to have a good silicone hold out with the paper used. If

silicone coatings of uniform thickness are desired, the paper should have a smooth closed surface. Alternative papers are often more porous and thus require higher silicone coat weights. With our Reduced Penetration Silicone (RPS) RC 1002 we provide a silicone that is able to siliconize slightly porous papers with low silicone coat weights. The absorbing effect of the porous surface can be partly compensated.

Pot life / Remaining silicone acrylates after the job

The guaranteed pot life of coater-ready free radical curing silicones is 72 hours, however, they can easily be stored for much longer time. Without UV light, free radical TEGO® RC Silicones will not cure. Store the unused portions of the blends in either lined drums or pails, or plastic containers. Keeping the following preconditions in mind pot life can be extended to several months:

- Prevent exposure to UV light. Use containers that prevent light from getting through to the silicone.
- Do not store the silicone outside where it can be exposed to direct sunlight or high temperatures.
- Allow air to get to the silicone blend.

The oxygen in the air prevents the silicone from curing. Allow headspace in the container, and reopen the container from time to time.

- If the blend will be sitting for a long period, you may want to stir the pail occasionally. This mixes air into the silicone, which helps prevent curing.
- Clearly identify the container to avoid confusion.

To make sure that the silicones are still good to be used, check the viscosity before use. It is also advisable to run a short trial with the material after longer storage time before starting bigger production campaigns.

Cleaning procedure

Using free radical curing silicones, pumps, tubes, or piping that are contaminated with silicones do not necessarily need to be flushed with solvents to clean them. As long as there is no direct sunlight reaching the silicones, they will not cure inside the tubes. Flushing will be required if you will be using other materials or silicones after running free radical RC Silicones.

Also, silicone acrylates will not cure on the roller for a production break overnight or for the weekend (as long as it is not exposed to direct sunlight). The coating remains liquid and does not plug up the cells. Therefore, there will be no build up on the doctor blade. However, the silicones will attract dust and dirt from the production environment. Therefore the rollers should be protected by a kind of cover against dirt and light during stand still. Of course, be-

fore longer stand-still of the coating line and changes between different coating or silicone systems, the coating system needs to be cleaned thoroughly.

To clean the coater head, drain the silicone from the coater head and piping into the holding tank. Keep the silicones in the holding tank or put them into a properly identified container to store it for future use.

The rollers and the silicone pan or closed chamber system should be cleaned by means of disposable towels and a suitable solvent such as isopropanol. If you need a thorough cleaning, stronger solvents like white spirit may be used to clean the roller. Please make sure that all regulations for the use of solvents are observed and that the solvent would not tend to swell the rubber rollers.



Cationic

Cationic curing silicones, products & formulations

Cationic curing takes place in a normal atmosphere, therefore, nitrogen inerting is not required. They can be cured with standard UV equipments as used in the printing industry. This is the cationic system's main advantage over free radical curing silicones. Therefore, cationic UV siliconizing is often the first choice when standard release values are required on suitable substrates (i.e. substrates which contain no ingredients that may poison the catalyst).

Poisoning effects by humidity and substrates as well as post curing are part of the inherent nature of cationic curing. Therefore, fast cure speed of both silicone base polymer and photocalyst is important. Cationic TEGO® RC Silicones contain the sensitizer isopropylthioxanthone (ITX), which gives rise to a substantial enhancement in possible cure speed.



TEGO® RC Epoxy Silicones - the basic silicone types

All cationic curing silicone polymers can be used as 100% products or in blends. They do not require an anchorage polymer. For UV curing, these silicones need the addition of a suitable photocatalyst.

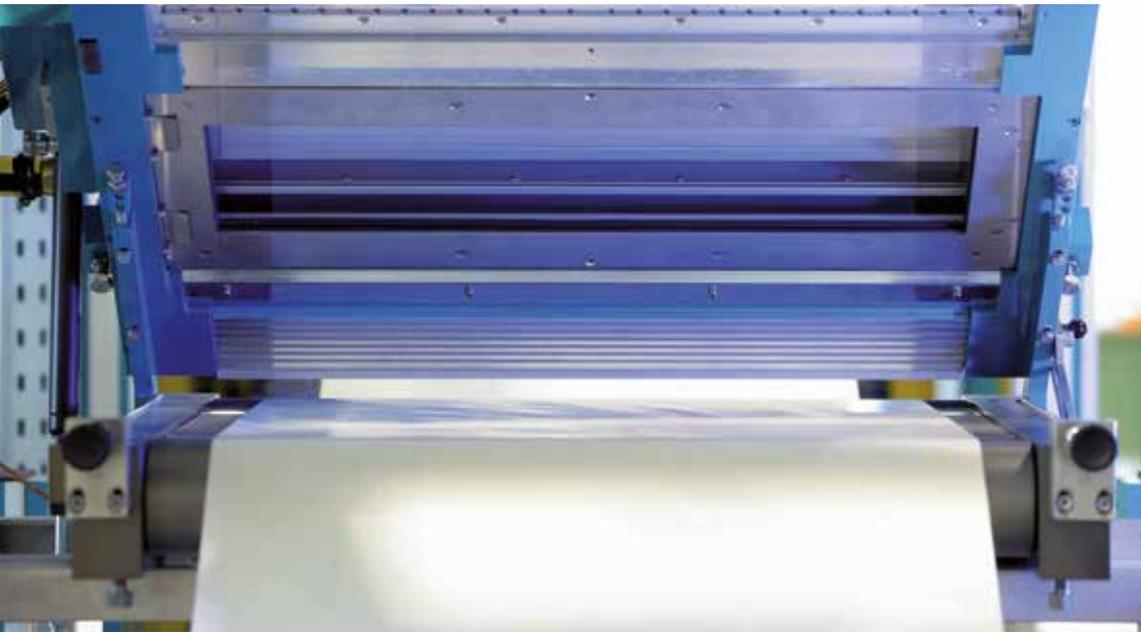
TEGO® RC Epoxy Silicones

- RC 1401 is used for premium re-release applications.
- RC 1403 is the standard easy release polymer. Compared to RC 1401, RC 1403 cures a little bit faster whilst the release level is slightly higher than RC 1401. RC 1403 is not available in the US and Canada.
- RC 1412 is used for tight release.

Our recommended photocatalyst

The photocatalyst TEGO® PC 1467 shows excellent compatibility with cationic curing silicones. The standard recommendation is to use an addition level of 1 - 2% photocatalyst. During the UV irradiation, it forms a strong acid which will initiate the cationic curing process via the functional epoxy groups of the silicone polymers.

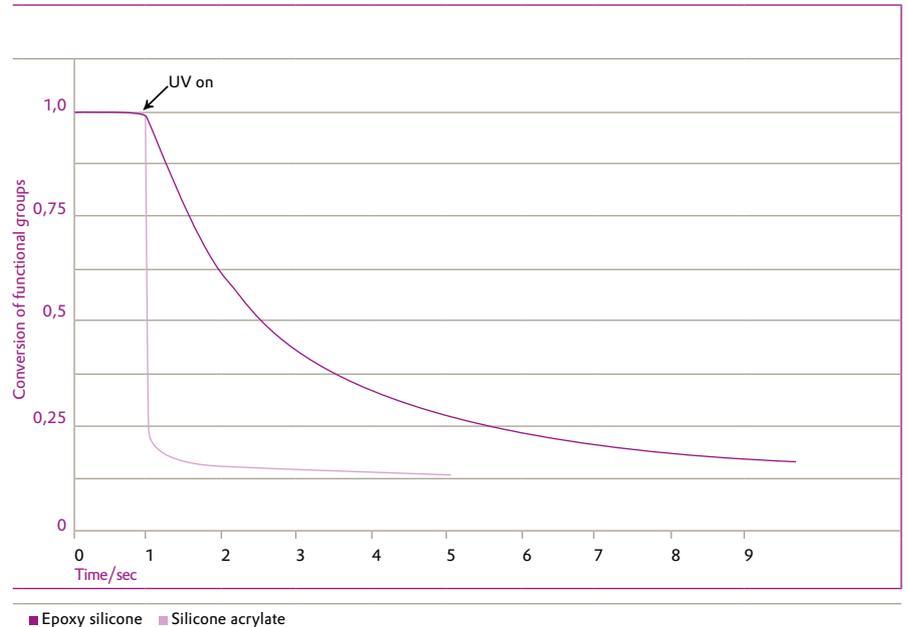
TEGO® PC 1467 offers very high efficiency and cure speeds. The addition level of PC 1467 may be reduced in applications, where non-poisoning substrates are used and low humidity is present.



Post-curing effects and curing speed

The cure speed of cationic polymerisation is generally slower when compared to free radical curing silicones. After exiting the UV source area, the cationic polymerisation continues in a post-curing process without further UV irradiation. The following graph illustrates the curing speed behaviour for radical and cationic curable systems. The silicones used in this study have the same degree of functionality.

Post-curing effect and curing speed



The degree of cure directly behind the UV unit, also called "initial cure", is very important as this has a major effect. This can be detected by simple finger rub tests, even in-line. A low initial cure causes an insufficiently cured surface prior to rewind. A silicone with insufficient initial cure may be damaged by the rewind process and thus yield varying release values. After one day storage time, the silicone will be fully cured in most cases due to post curing. Thus, subsequent adhesion values are usually very high after sufficient post-curing time.

During in-line converting processes, special care needs to be exercised. The degree of silicone cure prior to in-line adhesive coating has an effect on the final release values of the laminate. Silicone

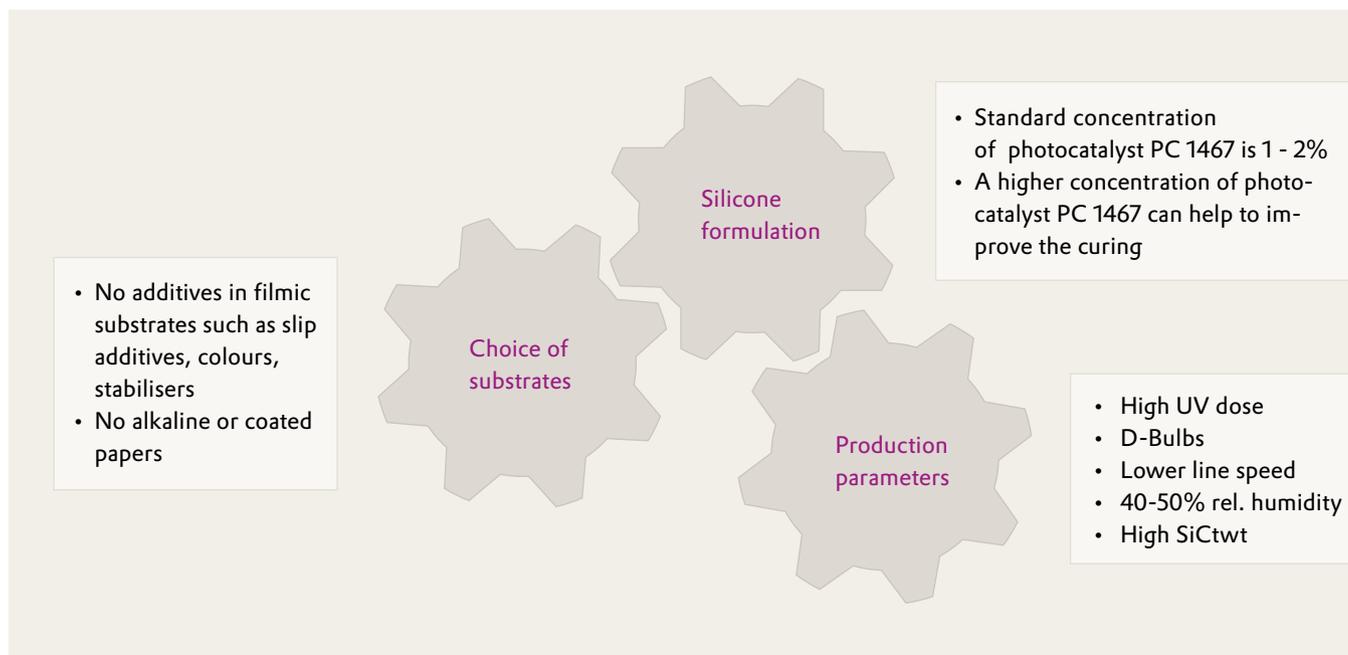
transfer due to insufficient cure can result in low release values and reduced adhesive tack. Therefore, the initial cure is very important for high quality release liner. The (initial) cure speed of cationic curing silicones can change using different substrates and different curing conditions.

To ensure optimum curing speed for your set up, follow these recommendations:

- Use suitable substrates for cationic polymerisation. Your local technical service team will help you to evaluate the suitability of your substrates by running performance tests.
- Higher UV power can help to provide better initial cure.
- Running lower line speed will increase the time gap from the UV curing station

to the rewinding station. This helps to give some additional time to complete the curing reaction.

- If possible, run cationic curing silicone in a low humidity area.
- Curing of higher coat weights might be easier than the curing of low coat weights. Small amounts of potential inhibiting substances in the substrates are "diluted" in a higher volume of silicone and therefore will influence the curing process to a lower extent.



How to enhance the degree of cure of cationic curing silicones



Siliconizing of plastic films

Due to the low heat impact also cationic curing silicones are commonly used for coatings on plastic films. These films can be made of different materials such as polypropylene, polyethylene or polyester. It is important for these substrates that they are free of poisoning ingredients to guarantee complete curing and good anchorage to the substrate. Such ingredients include:

Potentially poisoning ingredients

- Colours, dyes and pigments
- Alkaline, base, amine derivatives
- Anti-oxidants from plastic like
 - Sulfur products e.g. thio compounds and sulphides
 - Phosphorous compounds e.g. phosphines, phosphites
- Metal soaps, Zn or Ca stearate, tin compounds
- Alcohol, water, polyether, ether

The decision whether a substrate is appropriate for siliconizing with cationic UV curing silicones can be easily made after some initial coating trials. Please send some sheets of the particular substrate to your technical service team for free trials on one of our technicum lines in Shanghai, Essen or Hopewell.

Siliconizing of paper substrates

The standard pulping process for the production of common paper grades is alkaline. Since the mechanism of cationic polymerisation is initiated via an acid, most paper substrates are not suitable to be coated with cationic curing silicones. The residual basic components in the paper will react with the acid formed by the photocatalyst, thus the curing process will not be initiated properly.

There are specialty papers manufactured in acidic pulping processes. These papers can be used with cationic curing silicones providing the same advantages as free radical curing UV silicones (excellent paper lay-flat, no rehumidification).

Polyolefin coated kraft papers (PEK) are coated with a thin layer of plastic film to prevent coatings from penetrating into

the paper fibres. Thus the silicone layer will be in contact to the thin filmic layer on top of these papers. Therefore, many PEKs are suitable for cationic siliconizing, too. The polyolefin used for the coating of the kraft paper should be free of poisoning ingredients for the cationic curing silicones as described before.

Pot life / Remaining epoxy silicones after the job

Like free radical curing RC Silicones, blends of epoxy silicones with photocatalyst added have a guaranteed pot life of 72 hours. In contrast to silicone acrylates, it is much more complicated to extend the pot life of ready-to-use blends longer than this.

However, keeping the following preconditions in mind, even the potlife of cationic curing silicones can be extended to weeks or even months:

- Prevent exposure to UV light. Use containers that prevent light from getting through to the silicone.
- Store the material below 30°C. Above 30°C polymerization will start resulting in higher viscosities over time.
- Do not store the silicone outside where it can be exposed to direct sunlight or high temperatures.
- Clearly identify the container to avoid confusion.

To make sure that the silicones are still good to be used, check the viscosity before use, e.g. by means of a Ford cup flow time measurement. It is also advisable to run a short trial with the material after longer storage time before starting bigger production campaigns.

Cleaning procedure

In contrast to silicone acrylates, epoxy silicones left on the coating equipment, holding tank and pipes must be removed prior to a lengthy downtime (overnight or weekend breaks) in order to avoid silicone build up. Therefore it is recommended to clean the whole equipment after every use.

To clean the coater head, drain the silicone from the coater head and piping into the holding tank. Keep the silicones in the

holding tank or put them into a properly identified container. The silicone blend should be used in the shortest possible time or disposed immediately to avoid gelation in the holding tank or container.

The rollers and the silicone pan or closed chamber system should be cleaned by means of disposable towels and a suitable solvent such as isopropanol. If you need a thorough cleaning, stronger solvents like white spirit may be used to clean the roll-

er. Please make sure that all regulations for the use of solvents are observed and that the solvent would not tend to swell the rubber rollers.

Test Methods

Quality tests

Off machine tests are useful to gather first information on the silicone coating quality right after production. These tests should be known to the operators and performed frequently, e. g. every jumbo roll:

Smear test

Rub the silicone surface with your finger. There should be no sign of liquid silicone (= smear).

The smear test is a good indication for the initial cure of **cationic** curing silicones. Very slight smear/marks in the silicone layer from the finger might be acceptable and should disappear within one day after production (postcuring).

If smear is observed on **free radical** curing silicones, there is something wrong in the curing process. Check the formulation (photoinitiator), the UV bulbs and reflectors and the inerting setup.

Rub-off test

Rub the silicone surface a bit harder with your finger. There should be no rub-off of any dry particles of silicone. Please note that if you rub hard enough, you may abrade the surface, especially at higher coat weights. This is not a sign of poor cure or anchorage failure. It is the nature of solventless silicones to have a more rubber-like behavior.

The anchorage of **cationic** curing silicones may improve within the first 24 hours after production due to postcuring of the silicone layer in contact with the substrate.



Loop test

Loop test

Apply an approximately 20 cm (8 inch) piece of TESA tape 4154 on the cured silicone coating, peel it off and form a loop by putting the adhesive coated sides together. Opening the loop should require a certain level of force. In case of a transfer of liquid (uncured) silicone, a considerable reduction of the release force will be noticed.

Dye stain test

A test ink of 1% methylene blue in water is applied for one minute onto the silicone coated surface by means of a Cobb tester. The blue ink will stain any uncoated areas of the paper. A certain level of staining may be acceptable. A standard should be defined for each product. This method works only on paper substrates, not with poly-coated papers or films.

Microscope

To evaluate the silicone coverage on filmic substrates, a coaxial illuminated microscope with a 50x magnification can be very helpful. A small, portable and battery operated microscope, can be used on the production line for quality control.



Test ink and Cobb tester for the dye stain test.



Coaxial illuminated microscope.

Release and subsequent adhesion test

For a full quality control, release and proper cure tests need to be done. For the European label industry FINAT test methods FTM 3, 4, 10 (release) and FTM 11 (subsequent adhesion) are often used. The European tape industry also employs AFERA test methods. In the US and in Asia, there are various ASTM, TLMI and/or PSTC methods used.

For most of the different test methods we have more detailed information at hand. Please consult with our technical service teams for assistance.

The subsequent adhesion test (FTM 11) is a good indication if the test tape has been contaminated by liquid silicone residues, i.e. the test can indicate insufficient curing. Due to the postcuring of **cationic**

curing silicones, subsequent adhesion tests can improve to a great extent within the first day after production. Therefore, this test is not meaningful in this period of time. After 24 hours, the postcuring should finally have taken place, therefore, the subsequent adhesion will normally show very high (good) numbers. Hence, this test method is not suitable as quality control test for **cationic** curing silicones.



Table top release tester.



X-ray tester to determine silicone coat weights.

Silicone coat weight measurement

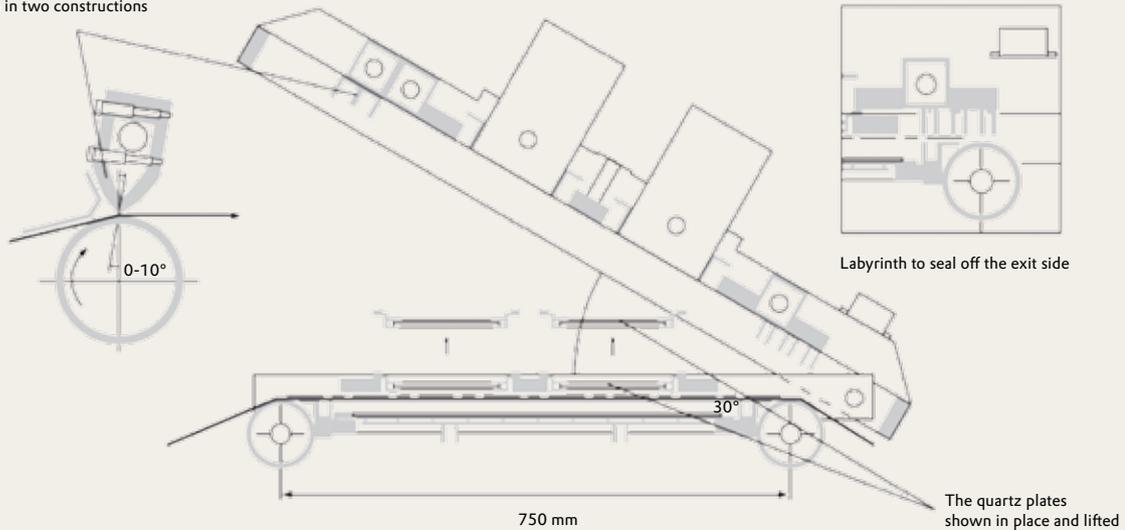
The silicone coat weight can be measured with a precision balance (4 decimal points) by washing off the silicone by means of a solvent. This can only be made on smooth filmic substrates and without corona treatment.

A more versatile method is the X-ray analysis that gives a signal proportional to the Si-atoms concentration in the coating. TEGO® RC Silicones are silicone acrylates or epoxy functional silicones. These organic groups must be considered when determining the actual silicone coat weight. Since calibration curves in X-ray units are generally based on 100% polydimethylsiloxanes, the readings observed must be adjusted to calculate the

actual silicone coat weight when using TEGO® RC Silicones. For this, we have introduced the "RC-Number" which is a measure for the organic content of the silicone (or silicone blend).

Extremely high Si blank values (as found with clay coated paper and pigmented films) can cause errors in the coat weight determination. Very porous papers can absorb silicones which results in lower readings. On thin filmic substrates, make sure to measure on the silicone coated side. Thin substrates will give readings even on the uncoated side by scanning through the substrate. This effect will lead to erroneous readings on double side coated thin films, too.

The nitrogen knife shown in two constructions



Labyrinth to seal off the exit side

The quartz plates shown in place and lifted

Example of an inerted UV unit

Maintenance Work on (Inerted) UV Units

The quality of a silicone coating is dependent on the conditions of curing. For a good silicone cure, a sufficient amount of UV light and – for free radical curing - good inerting conditions are required. Using too much UV light cannot harm the properties of both UV silicone systems.

However, using too little UV light, e. g. by reduced transparency of the quartz plates or worn-out UV-bulbs, can reduce the degree of cure.

This is an example of a maintenance plan for an inerted UV unit. This maintenance

schedule can be used as a guideline for operators and the engineering department. For non inerted UV units, just ignore the recommendations for the maintenance of the nitrogen supply system.

Action	How Frequently	Responsible
Clean quartz plate from chamber inside with cleansers like Sidol (Germany), Brasso (UK) or Soft-scrub/ Barkeepers Friend (US). Avoid cleansers using quartz or feldspar as abrasives.	Every week of full production (using TEGO® Photoinitiator A18). More often if there is a faster build-up of layers on the quartz plate observed.	Operators
Clean the inside of the nitrogen knife by means of an old plastic credit card (approximately 0.8 mm thickness). Do not use metal tools to avoid scratching the inside of the nozzle.	Once a day to once a month depending on deposits inside the barrier nozzle.	Operators
Clean quartz plate from UV lamp side with isopropanol.	Every month to six months depending on dust layers on the quartz plate.	Operators
Clean reaction chamber inside with a damp rag to remove dust. Any cured layers on the water cooled base plate can be left on as long as cooling efficiency is not reduced.	Once a month depending how quickly the dust appears. The purpose is to eliminate loose powder and dust.	Operators
Check the distance of the nitrogen knife to the web (1.6 - 1.8 mm uniform across web) and opening gap of the knife (0.8 mm across web).	First time after three months then once a year.	Engineering Department
Calibrate oxygen meter according to manufacturer procedure.	Once a year.	Engineering Department
Clean UV bulbs and reflectors according to manufacturer procedure.	Depending on dust build-up.	Engineering Department
Replace UV bulbs.	According to manufacturer advice. Possibly every 1000 to 2000 hours of operation.	Engineering Department
Replace or clean air filter on the UV unit.	According to manufacturer procedure.	Engineering Department
Ensure proper water and air flow through UV dryer cooling system.	According to manufacturer procedure.	Engineering Department

Please double-check with the equipment manufacturer for recommended procedures and possible further maintenance.

www.evonik.com/tego-rc



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