

THIXATROL[®] AS 8053

Highly effective organic thixotrope for high-performance MS polymer and hybrid sealants

Key Benefits

- ❖ Low processing temperatures
- ❖ High efficiency
- ❖ Supreme temperature storage stability
- ❖ Savings in process/costs

Introduction

THIXATROL® AS 8053 is a 100% active, seed resistant organic rheological additive based on a diamide technology. THIXATROL® AS 8053 provides high viscosity build, imparts thixotropy and allows application at high layer thicknesses .

Composition	Proprietary organic
Appearance	Fine off white powder
Bulk density	0.25 g/cm ³ 2.08 lb/gal
Density	1.02 g/cm ³
Melting point	120 - 130 °C
Mean particle size	Max. 5 µm

Key benefits

- Designed for low temperature activation, processing and packaging
- 100% active, active powder
- Highly efficient already at low loadings
- More effective in comparison to market reference
- Provides outstanding viscosity control, thixotropy and excellent slump and sag resistance when applied at high layer thicknesses.
- Cost efficient due to complexity reduction in production/processing
- Excellent storage stability of the readily formulated system
- Seed-resistant

Applications

- Hybrid sealants, both MSP and SPUR
- Industrial coatings
- Corrosion protective high solid epoxy primers (and solvent free)
- Two component high solid polyurethane top coat systems

Incorporation in MSP sealants

The low temperature activation of THIXATROL® AS 8053 makes sealant and paint manufacturing less complex. This consequently results often in a significant cost benefit.

THIXATROL® AS 8053 is added at the beginning of processing to the batch. Different to earlier process descriptions, all non sensitive ingredients can be loaded before activation. After activation at temperatures of below 50 °C, sensitive ingredients and the catalyst can be added for completion.

THIXATROL® AS 8053 is incorporated best with a vacuum mixer at the highest possible shear for the relevant time needed to obtain the required activation temperature. To determine the ideal activation temperature a ladder study is recommended up front to the processing.

The necessary activation temperatures for solvent-free MSP- and SPUR sealants are in a range of 35 -55 °C (95 -131 °F). For high solids and protective coatings (epoxy primers, polyurethane topcoats) are the temperature needs to be in a range of 30 - 60 °C (86 -140 °F).

Typical levels of use for effective sag and flow control are in a range from 1.0% to 3.5% related to the total system weight.

Processing

In terms of process control, the use of THIXATROL[®] AS 8053 provides several benefits in comparison to the typically used market standards. These differences in the current and the optimized processing with THIXATROL[®] AS 8053 conditions are described in the following figures (1,2).

Figure 1: Schematical representation of the current process conditions

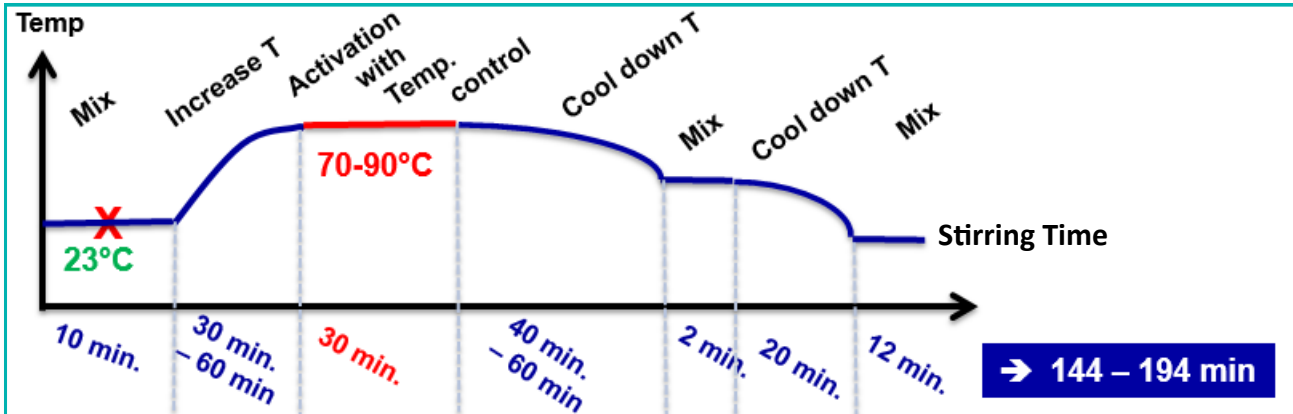
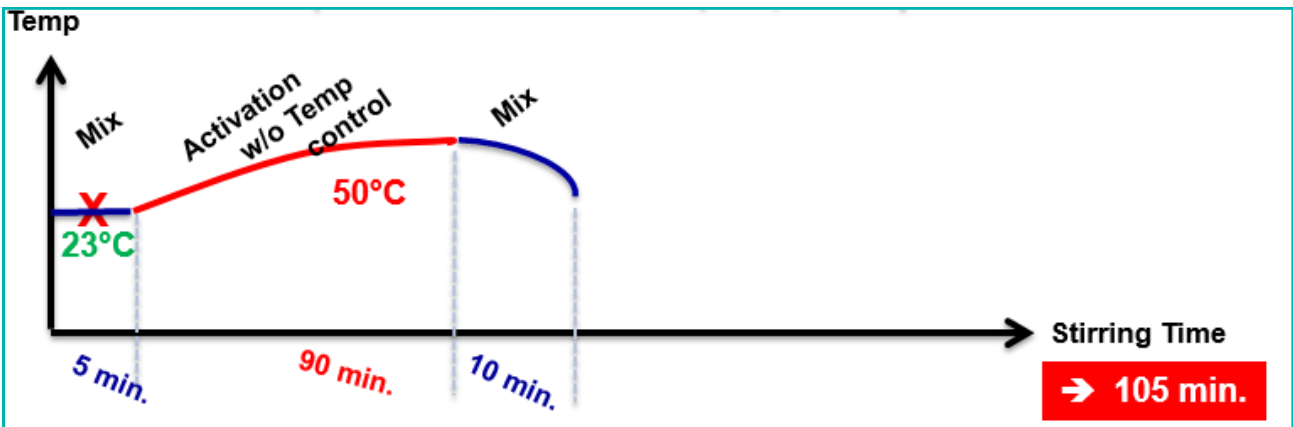


Figure 2: Schematical representation of the optimized process conditions



Note, the required activation time of 90 minutes which is displayed in figure 2, has been achieved with a lab kneader (for further information, please refer to page 4). In actual production conditions, where different equipment is used, the time might be considerably lower.

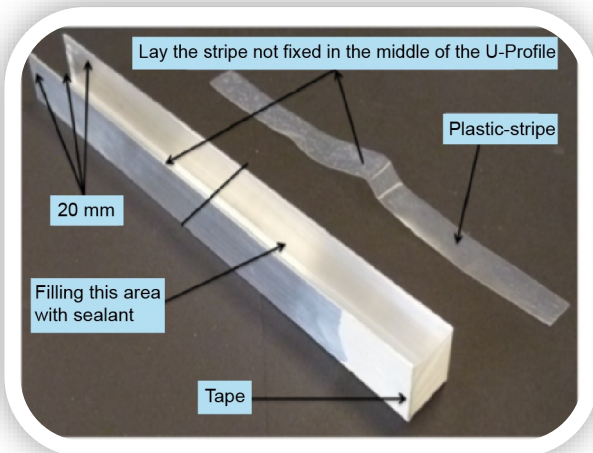
Under optimized conditions, as presented in figure 2, processing is less complex. Energy savings can be achieved due to lower process temperatures and the risk of failure with temperature sensitive ingredients is lower. Production is also faster due to the elimination of heating and cooling down phases.

These process conditions are applicable when using THIXATROL[®] AS 8053. This will be underlined by the results which are being presented in the following.

Preparation equipment and characterization methods



A planetary vacuum kneader was used for preparation



Evaluation of sag control

An aluminium U-profile, 20 × 20 mm - 25 cm long, was used. On the backside of the inside profile a polyethylene tape was placed. This tape has the possibility to slide. By using this tape the area of adhesion of sealant to aluminum is reduced and makes the test more severe. 12 cm of the profile (in horizontal position) were filled with the sealant. After that, the tape was removed and the U-profile was positioned in a vertically.



Rheological measurements

1. All rheological measurements were performed using the Anton-Paar MCR 300.
2. Flow curves were determined in a shear rate range from 0.1 s^{-1} to 100 s^{-1} , the shear stress at high shear rates of 100 s^{-1} was extracted out of the rheology curves.
3. Extrudability was measured rheologically.

Following data are available on request

1. Yield point measurement, to display the minimum shear stress to initiate flow
2. Oscillatory structure, to display the structure recovery time after the removal of shear

MS-Polymer sealant formulation and process instruction

The comparative study was performed in two parts. In the first part, the tested rheological additives were added at a 3.5% loading level in the following MS-polymer based sealant formulation. In the second part of the study, the loading level was set at 1.6%. This was done in order to make differences more visible.

Table 1: MS polymer sealant formulation

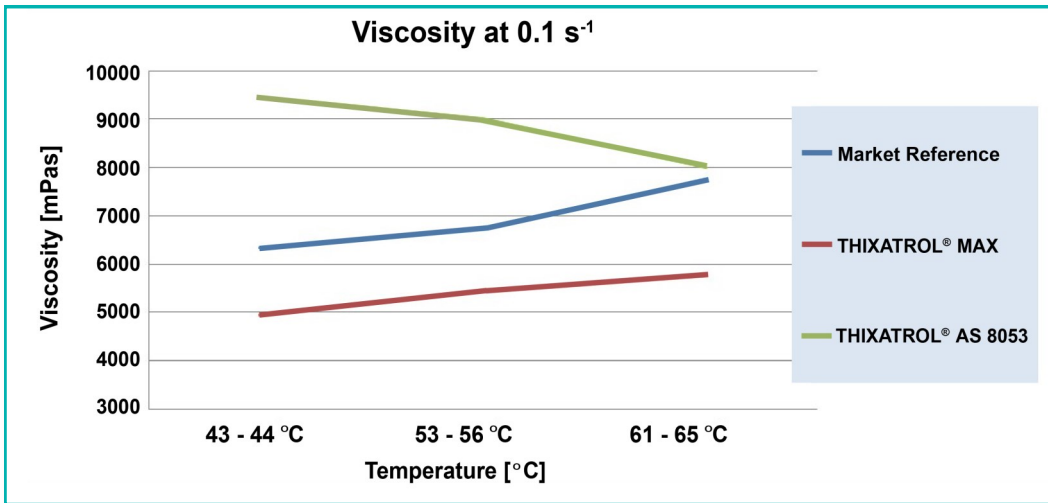
	Ingredients	Concentration	Function	Supplier
1	MS Polymer™ S203H	15.00	Binder	Kaneka
2	Carbital™ 110S	50.00	Extender	Imerys
3	Kronos® 2190	3.00	Pigment	Kronos International
4	Rheological Additive	3.50/1.60	Rheological additive	Elementis/other
5	Ms Polymer™ S303H	10.00	Binder	Kaneka
6	Jayflex™ DIUP	16.50	Plasticizer	ExxonMobile Chemical
Adding after cooling down, temperature depends on the activation temperature				
7	Dynasylan® VTMO	0.70	Water absorbent	Evonik Industries
Adding after cooling down, temperature depends on the activation temperature				
8	Dynasylan® DAMO-T	0.50	Adhesion promoter	Evonik Industries
9	Metatin™ Katalysator 740	0.30	Catalysator	DOW

Process instruction

1. Place ingredients 1 - 6 in the mixing beaker into the mixing container.
2. Mix the ingredients for 1 minute at 50 rpm without vacuum.
3. Stop mixing and scrape down adhering material from the mixing tools and the walls thoroughly.
4. Restart the mixer and turn the vacuum on. Increase the stirrer speed to 500 rpm and disperse the material for 90 minutes without additional temperature control.
5. Stop mixing, release the vacuum slowly and add ingredient 7.
6. Mix for 1 minute at 50 rpm.
7. Stop mixing again and add ingredients 8 -9 carefully.
8. Mix for 1 minute at 50 rpm then turn the vacuum on and mix further on for 10 min at 500 rpm.
9. Stop mixing, release the vacuum slowly and scrape down the material from the mixing tools and the walls thoroughly.

MS Polymer sealant activation temperature study

Figure 3: Viscosity measured at different temperatures (loading level at 3.5%)



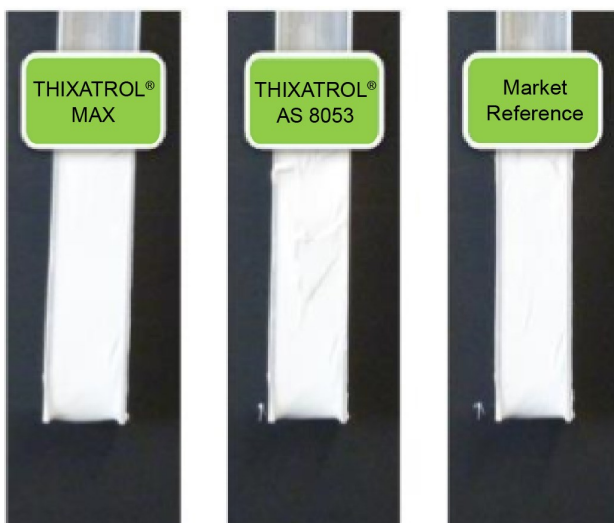
All organic thixotropes based sealants were evaluated at various activation temperatures of 45 °C, 55 °C and 65 °C to describe the differences in the activation requirements of the individual products.

THIXATROL® AS 8053 provided significantly higher viscosities over a wide range of lower activation temperatures in comparison to the market references material. The observed effect results in less complex and more robust processing for the formulator. The packing can be without cooling directly after processing. This consequently results in significantly reduced production times.

For the following tests all additives were activated at a temperature of 50 °C.

Evaluation of the rheological activity of THIXATROL® AS 8053 at 3.5% loading level

Picture 1: Sag control test (loading level 3.5%)

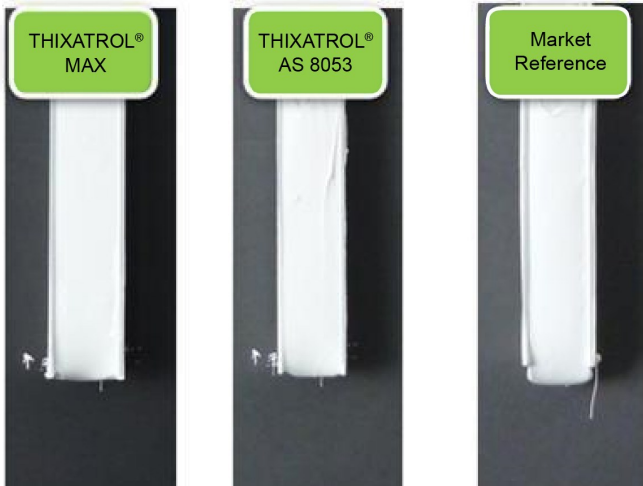


Sag control tests show that all the tested sealants perform well when the rheological additive was formulated at a concentration of 3.5% on total formulation.

In order to visualize the performance differences, the loading level will be reduced in the following tests.

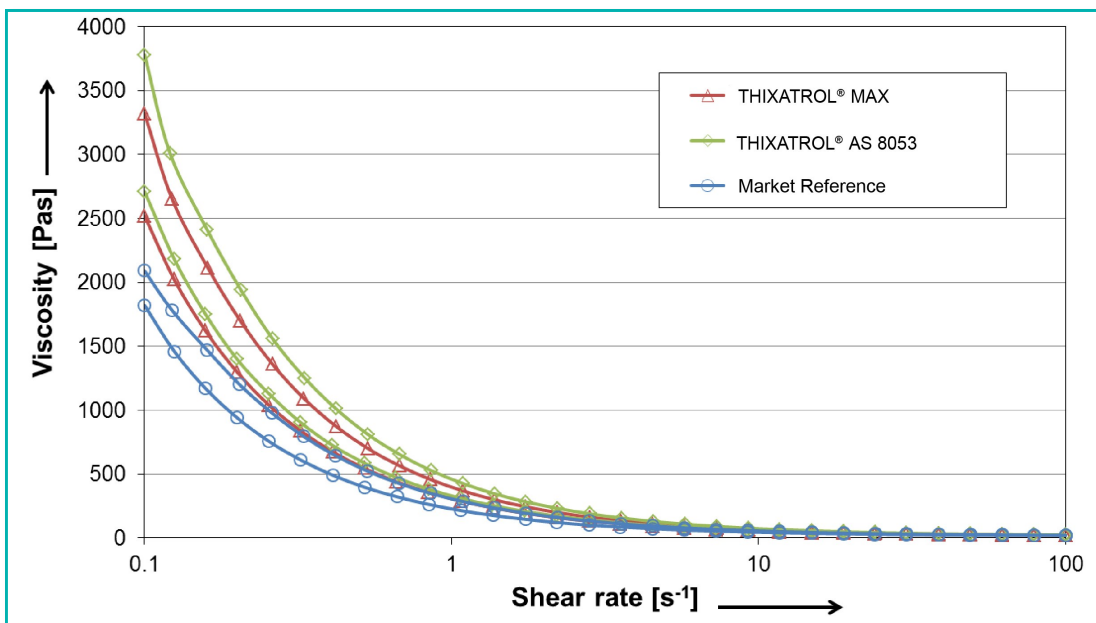
Evaluation of the rheological activity of THIXATROL® AS 8053 at 1.6% loading level

Picture 2: Sag control test (loading level 1.6%, activation at 50 °C)



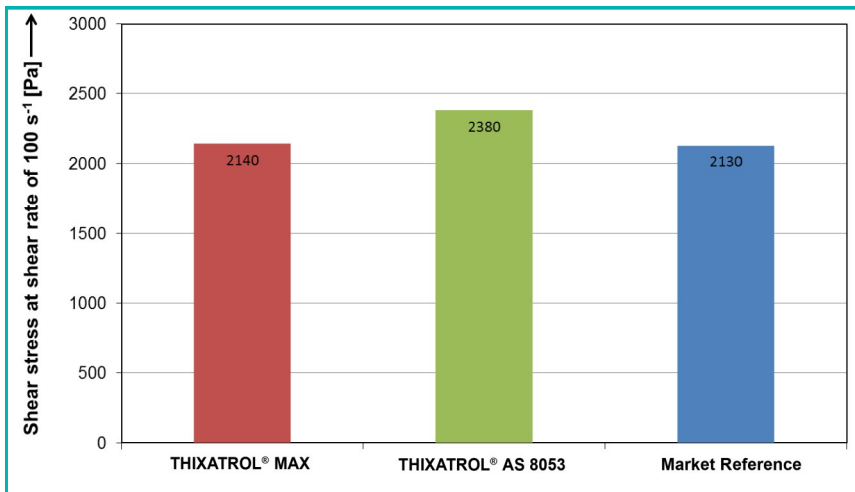
THIXATROL® AS 8053 was evaluated in comparison to THIXATROL® MAX and a market benchmarks at a concentration of 1.6% on total formulation. Sag control tests were conducted directly after the production of the sealant. THIXATROL® AS 8053 and THIXATROL® MAX demonstrate strong sag stability at this lowered concentration. The parallel tested market references show initially remarkable signs of sagging under this conditions.

Figure 4: Flow curves (loading level 1.6%, activation at 50 °C)



The flow curves presented in figure 4, are describing the exact viscosity development at the relevant shear rates. The down curve shows the disruption, and the back curve displays the recovery of the viscosity. When activated at 50 °C, the sample formulated with THIXATROL® AS 8053 demonstrates the highest low shear viscosity. The values are slightly higher than those achieved using THIXATROL® Max at equal conditions. Both THIXATROL® grades are clearly outperforming the parallel tested market reference sample.

Figure 5: Shear stress at 100 s⁻¹ to demonstrate the extrudability



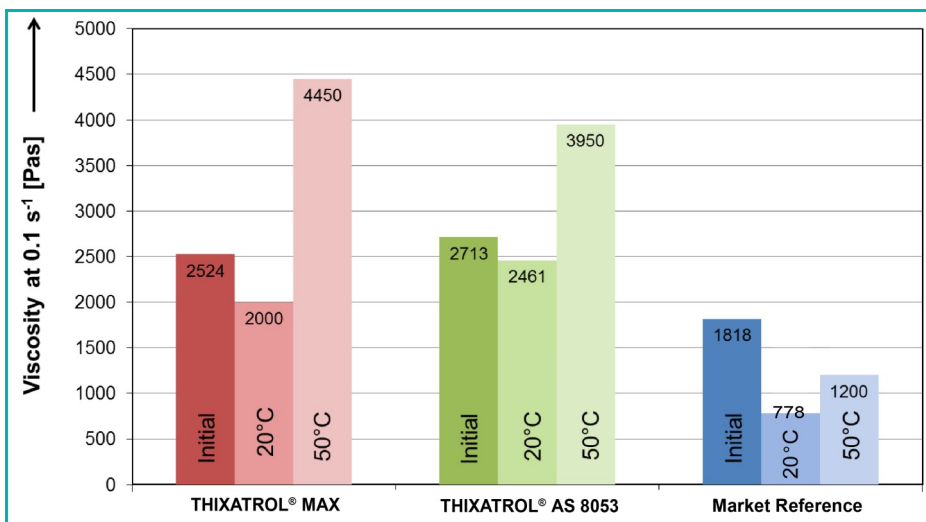
In figure 5, the shear stress at higher shear conditions of 100 s⁻¹ is plotted as an equivalent to the extrudability of the sealant.

Data shown have been extracted from the in figure 4 shown measurement and displays the relevant force detected as shear stress at a shear rate of 100 s⁻¹. This forces were taken as the equivalent to the force necessary to extrude the material out of a cartridge.

All tested samples show force values in the same range. Therefore, the extrudability of the individual samples has to be expected very similar. The slightly higher value of the sample containing THIXATROL® AS 8053 can be explained by the slightly higher viscosity values in to comparison to both furtherly tested samples. This underlines the higher efficiency of THIXATROL® AS 8053.

The samples with the market reference and THIXATROL® Max are both acting equally. However, note that the viscosity of the sealant with the market reference shows noticeably lower viscosities at low shear rates.

Figure 6: Viscosity after storage (initial, 20 °C, 50 °C)

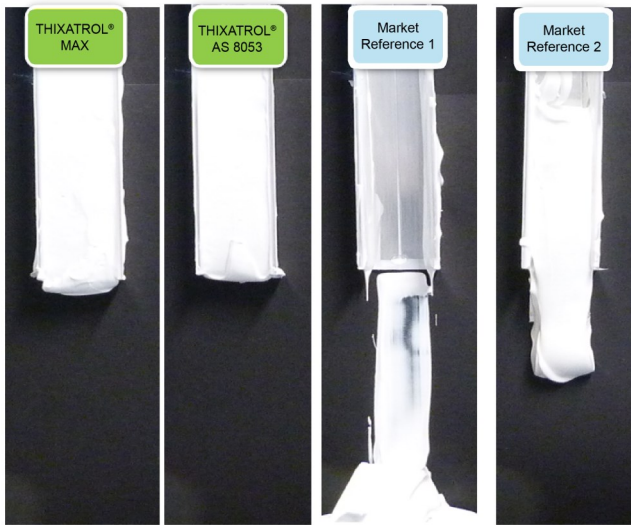


In figure 6, the viscosities at 0.1 s⁻¹ of the recovery curves before (initial) and after storage (6 weeks at 20 °C, 3 weeks at 50 °C) are presented.

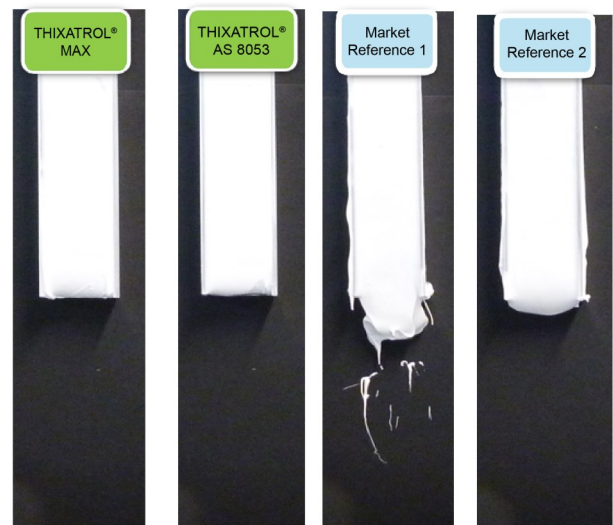
Samples formulated with THIXATROL® Max and THIXATROL® AS 8053 show a viscosity increase when stored at elevated temperature of 50 °C. Storage at room temperature of 20 °C had only minor influence on the sealants viscosity.

In case of the market reference, a significant viscosity loss has been noticed at both storage temperatures.

Picture 3: Sag resistance after 3 months of storage at 20 °C



Picture 4: Sag resistance after 2 months of storage at 50 °C



Samples containing THIXATROL® AS 8053 and THIXATROL® MAX show excellent sag stability after storage at room and elevated temperature. Only a slight indication to flow has been seen with THIXATROL® Max after 3 months at room temperature.

Material formulated with the market reference rheology modifier demonstrate severe failure of sag stability especially after 3 months at room temperature.

THIXATROL® AS 8053 performance summary

THIXATROL® AS 8053 is a highly effective organic thixotrope for MS sealants and hybrid sealants which provides

- Outstanding performance after low temperature activation down to 40 °C, THIXATROL® AS 8053 clearly outperforms current market references
- Excellent low-shear viscosity build
- Good extrudeability, determined rheologically as shear stress at higher shear rates
- Superior structure storage stability



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