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PVC mixer tests
Reproducibility and influence of test conditions

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Mixer tests in PVC industry

A laboratory mixer is the ideal measuring tool for testing fusion behavior, compound stability, and processing behavior of PVC formulations. A laboratory mixer is very sensitive to any changes caused by the compound formulation or any changes in the dry blend components, such as the resin, additives used, and additional fillers. Laboratory mixers are also sensitive to changes that are caused by incorrect handling. To achieve reliable test results, it is imperative that the mixer is handled in a reproducible manner.

This application note shows examples of reproducible mixer tests and provides examples of how changes in the testing conditions influence measuring results.

For these investigations, the following equipment configuration was used:

- Thermo Scientific™ HAAKE™ PolyLab™ OS RheoDrive™ 7 OS
- Torque sensor for HAAKE PolyLab OS, 400 Nm
- PolySoft OS Mixer Test and Data Evaluation Software
- HAAKE Rheomix 600 OS
- Roller rotors
- Pneumatic ram
- Sample material—rigid PVC dry blend (typically used to produce window profiles)

Test procedure and measuring results

Typically, a PVC dry blend is a mixture of basic PVC resin, fillers, processing aids (e.g., softeners, stabilizers, inner and outer lubricants), and other additives. Each component influences the processing behavior of the compound. One important factor is fusion behavior. Changes in the fusion behavior have a direct effect on the degree of gelation of the final product. For a PVC window profile, this would result in a deterioration of the mechanical properties or problems in the welding behavior. Unlike a normal thermoplastic polymer, PVC does not melt when simply exposed to higher temperatures. PVC needs additional shearing and compression forces to agglomerate the PVC particles and to form a homogenous melt.

Figure 1: HAAKE PolyLab OS System—RheoDrive with Rheomix OS Laboratory Mixer.

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In a laboratory mixer, this fusion behavior can be studied by measuring the drive torque and recording this torque over the mixing time. The PolySoft OS Mixer Test and Data Evaluation Software (Figure 2) guides the user through this test, records the measurement data, and evaluates all points of interest of the resulting measurement curve by means of dedicated, pre-defined evaluation routines. This software also works as a measurement database and enables further statistical studies to be performed.

The following graph shows an evaluation of such a torque curve (Figure 3). At the beginning of the test, the loading of the PVC powder into the mixer causes an instantaneous increase of torque ("L"–Loading Peak). The powder then distributes within the mixer chamber, and some parts of the compound (e.g., waxes) melt due to the high mixer temperature. Both effects lead to a drop in torque and lead to a first minimum ("V"–Valley). Due to the increase in mass temperature and the introduced shear energy, the PVC starts to combine into bigger agglomerates. This causes an increase in viscosity, which leads to an increase in torque. This process results in a second torque maximum ("F"–Fusion Maximum). The PVC dry blend forms a homogeneous melt. Due to an additional temperature increase of the sample caused by frictional heating, the torque drops again until it reaches a constant torque. A balance between the temperature increase caused by dissipation and temperature decrease caused by heat conduction through the chamber wall is reached. The torque, which is adjusting here, is a relative value for the melt viscosity of the sample.

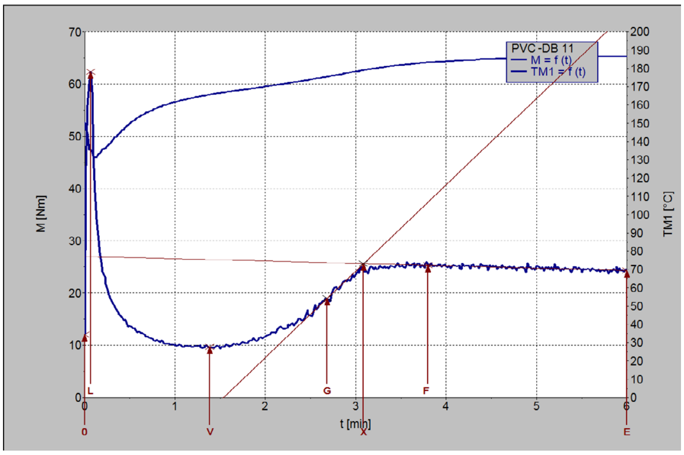


Figure 3: Evaluation of a mixer curve.

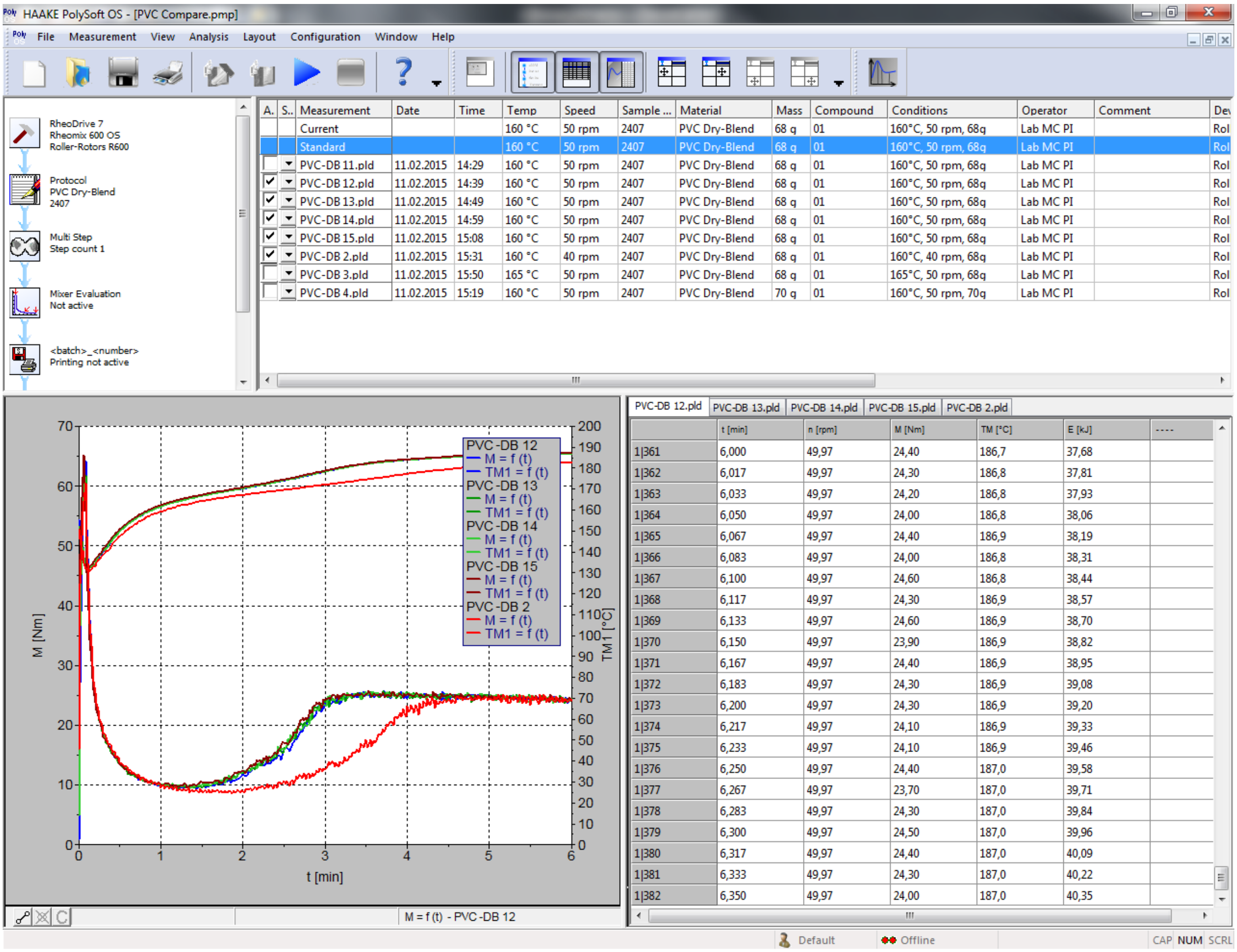


Figure 2: Screen capture—PolySoft OS Mixer Test and Data Evaluation Software.

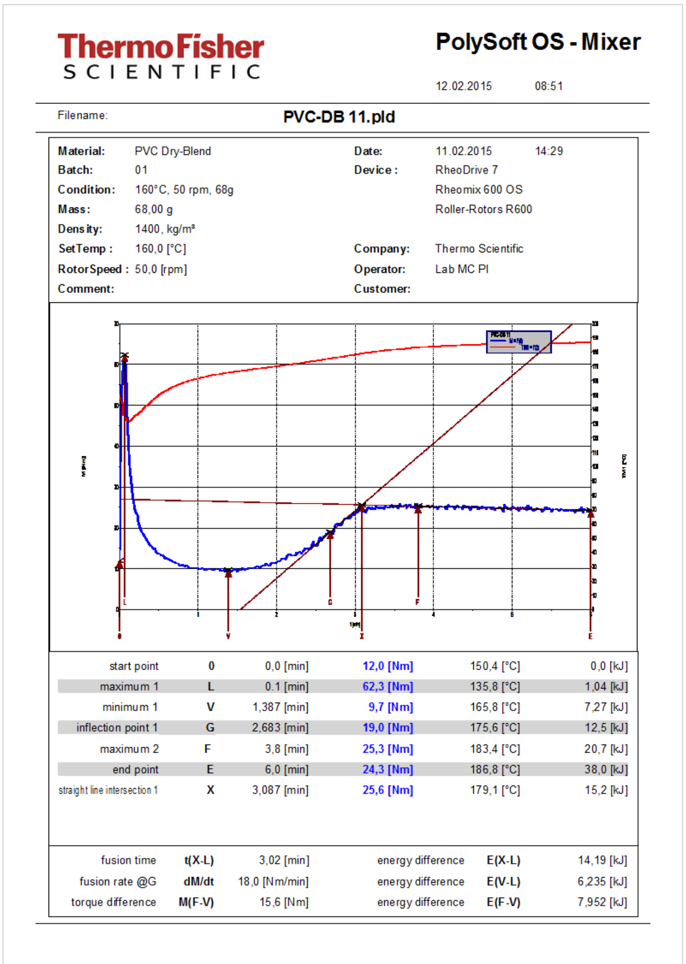
Additionally, Figure 3 shows a straight-line intersection (X) generated by the gradient of the point of inflection (G) and the gradient around the fusion point (F). Since X is easier to reproduce than the fusion maximum (F), X is often used to compare the different fusion behavior of samples. The software generates a measuring report that documents all the results (Figure 4).

Reproducibility of mixer tests

To check the reproducibility of mixer tests, five tests were done under the same conditions, using the same PVC dry blend.

Test conditions	
Mixer temperature	160 °C
Rotor speed	50 rpm
Sample weight	68 g
Test time	6 minutes

Figure 5 shows the fusion curves of these five mixer tests within one graph. Clearly, it can be seen that the torque curves of these five tests are nearly identical, which shows the good reproducibility of the test method.



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PolySoft OS - Mixer

12.02.2015 08:51

Filename: PVC-DB 11.pld

Material:	PVC Dry-Blend	Date:	11.02.2015 14:29
Batch:	01	Device :	RheoDrive 7
Condition:	160°C, 50 rpm, 68g		Rheomix 600 OS
Mass:	68,00 g		Roller-Rotors R600
Density:	1400, kg/m³		
SetTemp :	160,0 [°C]	Company:	Thermo Scientific
RotorSpeed :	50,0 [rpm]	Operator:	Lab MC PI
Comment:		Customer:	

start point	0	0,0 [min]	12,0 [Nm]	150,4 [°C]	0,0 [kJ]
maximum 1	L	0.1 [min]	62,3 [Nm]	135,8 [°C]	1,04 [kJ]
minimum 1	V	1,387 [min]	9,7 [Nm]	165,8 [°C]	7,27 [kJ]
inflection point 1	G	2,683 [min]	19,0 [Nm]	175,6 [°C]	12,5 [kJ]
maximum 2	F	3,8 [min]	25,3 [Nm]	183,4 [°C]	20,7 [kJ]
end point	E	6,0 [min]	24,3 [Nm]	186,8 [°C]	38,0 [kJ]
straight line intersection 1	X	3,087 [min]	25,6 [Nm]	179,1 [°C]	15,2 [kJ]

fusion time	t(X-L)	3,02 [min]	energy difference	E(X-L)	14,19 [kJ]
fusion rate @G	dM/dt	18,0 [Nm/min]	energy difference	E(V-L)	6,235 [kJ]
torque difference	M(F-V)	15,6 [Nm]	energy difference	E(F-V)	7,952 [kJ]

Figure 4: Software report—fusion test of a PVC dry blend.

To achieve good reproducibility, it is important that a user must always use the mixer in the same manner. Changes in handling or mixing conditions will have an immediate effect on the test results. To show how small changes in the mixer settings or the testing conditions influence the measuring results, the following tests were done with the same PVC dry blend but with defined changes in the mixing conditions.

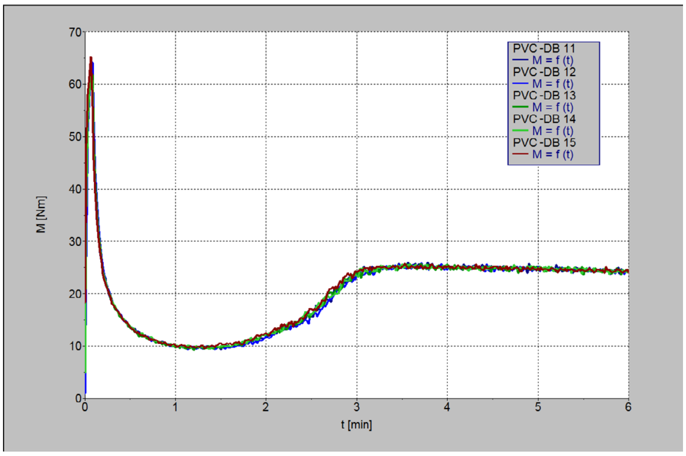

Figure 5: Reproduction of five mixer tests.

Influence of the mixer speed

Test conditions	
Mixer temperature	160 °C
Rotor speed	40 rpm and 50 rpm
Sample weight	68 g
Test time	6 minutes

Figure 6 shows the influence of the mixer speed on the measuring results. The reduction of the rotor speed results in a much longer fusion time. The reduced rotor speed introduced less shear energy into the sample.

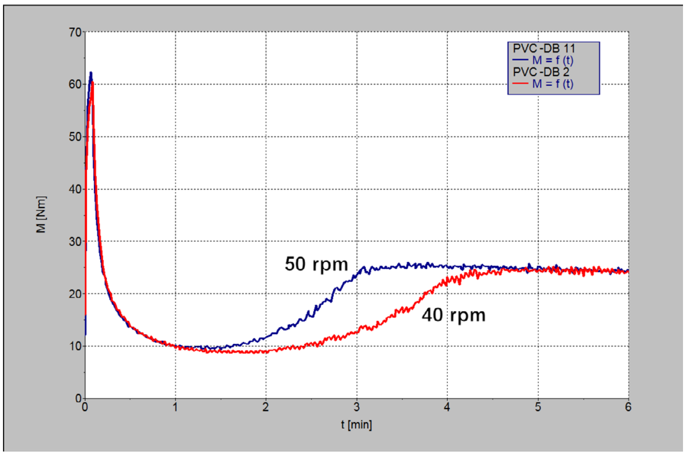

Figure 6: Comparison of different rotor speeds.

Influence of the mixer load

Test conditions	
Mixer temperature	160 °C
Rotor speed	50 rpm
Sample weight	68 g and 70 g
Test time	6 minutes

The heavier sample weight of an additional 2 g of material caused a much shorter fusion time (Figure 7). This is because the higher sample volume resulted in more shearing. It is important to note that small mistakes made during the weighing of the sample do have a significant effect on the measurement.

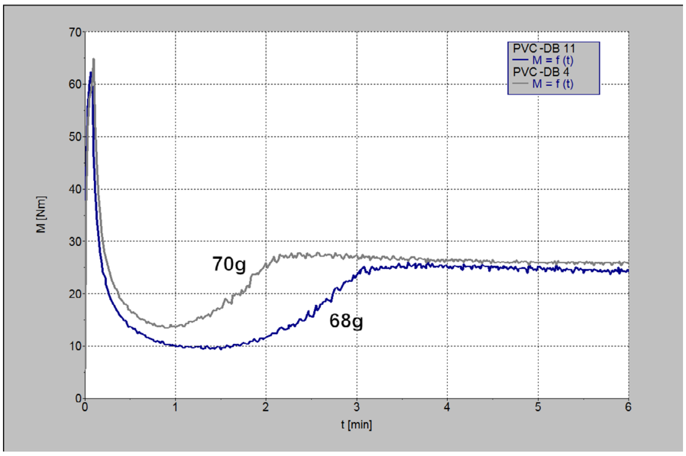

Figure 7: Comparison of different mixer loads.

Influence of the mixer temperature

Test conditions	
Mixer temperature	160 °C and 165 °C
Rotor speed	50 rpm
Sample weight	68 g
Test time	6 minutes

An increase of just 5° C caused a much faster fusion of the sample (Figure 8). For a user, this means that he must make sure that the sample is fed at the same temperature. To ensure this, the user must take special care that the cleaning procedure and cleaning time are reproducible.

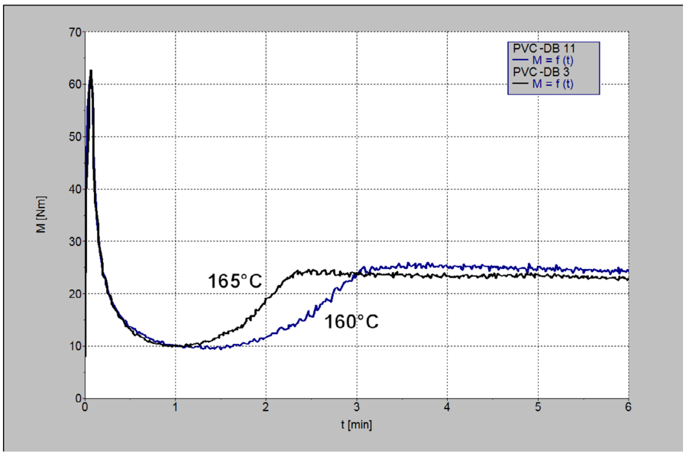

Figure 8: Comparison of different mixer temperatures.

Summary

These results show that testing with the laboratory mixer is reproducible, very sensible to changes in compound formulations, but also very sensitive to changes in handling and measuring conditions. When handled in a reproducible manner, the HAAKE PolyLab System, in combination with a laboratory mixer, is a reliable tool to characterize the processing behavior of PVC dry blend.

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