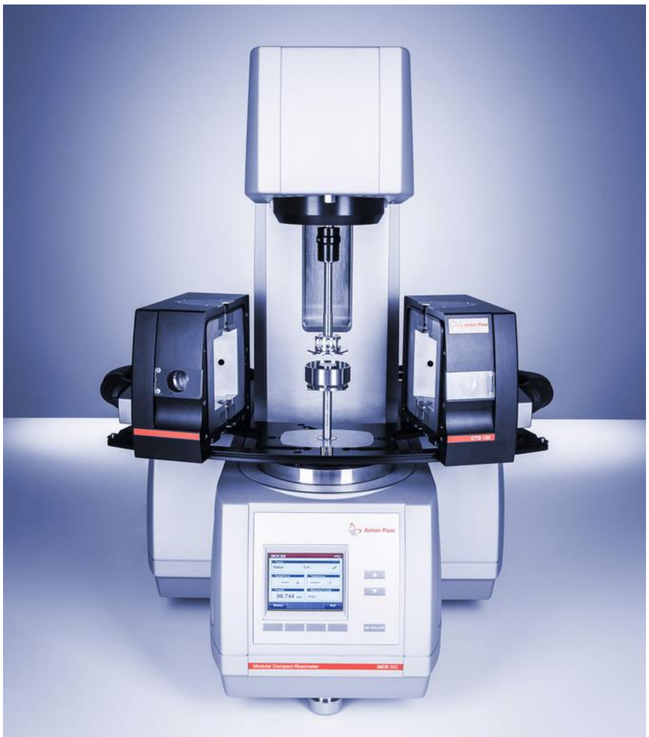
Round Robin: Powder Shear Cell

Relevant for: Shear Cell, Pharmaceutical Industry, Food Industry, Chemical Industry, Powder Flow, Powder Rheology

This application report summarizes the results of the round robin test with Anton Paar's Powder Shear Cell and the certified powder CRM 116. The reproducibility of shear stress points during yield locus analysis with reaching steady-state either automatically or time-based was examined.



1 Introduction

A round robin test is a method for external quality control of measurement methods with regard to their accuracy and precision. Identical instrument setups from different research institutes are examined. The participating laboratories receive identical samples, identical measurement instructions and the same time period for performing the tests. This allows the results and statements to be compared with each other.

This report focuses on yield locus measurements of certified limestone powder CRM 116 and the reproducibility and repeatability of normal stresses during pre-shear and shear-to-failure using Anton Paar's annular Powder Shear Cell. Each of the five participating laboratories performed shear measurements in triplicate under identical test conditions (automatic reaching and time-based reaching of steady-state) and at three different normal stresses. The tests performed at different normal stresses are referred to as stress levels 1-3 in the following text.

1.1 Yield Locus Analysis

The yield locus analysis is based on the Mohr-Coulomb theory and the measures of the failure plane. The measurement consists of two different steps: pre-shear and shear-to-failure. During pre-shear, the sample is consolidated at a given normal stress value σ_{pre} and then sheared until steady-state is reached in which the shear resistance and bulk density remain constant. During shear-to-failure, the normal stress acting on the sample is reduced to σ_{sh}. The sample begins to flow (fail) when a sufficiently large shear stress is reached. The point of incipient flow from various measurements is further used to determine the yield locus function. (1)

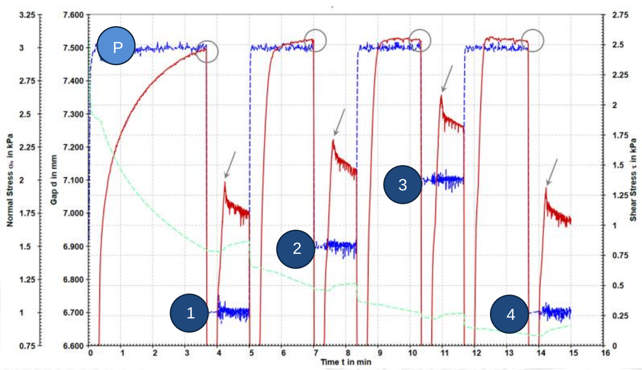


Figure 1: Typical shear sequence of limestone powder at 3 kPa preshear normal stress. The red line displays the shear stress progression during preshear (indicated by gray circles) and shear-to-failure (indicated by arrows). The blue line corresponds to the normal stress values. The four different levels of normal stresses at shear-to-failure are marked in dark blue circles (1-4). The normal stress at preshear is marked in light blue (P).

Tip: Further information about the measuring procedure can be found in the application report: “Introduction to Powder Rheology”.

2 Sample Preparation and Test Conditions

CRM 116 limestone powder from the European Commision’s Institute for Reference Materials and Measurements was used. The material is delivered with certified results for yield locus measurements which were determined with a Jenike shear tester. (2)

In order to obtain repeatable results, the limestone powder was sieved before being filled into the powder chamber. For sieving, all participants used an identical sieve. Furthermore, the effects of incorrect filling were reduced by an additional sample preparation step. This additional step includes a prior shear measurement at a normal stress of 1.5 kPa.

The tests were performed at ambient conditions without temperature or humidity control.

2.1 Measuring Conditions and Settings

The yield loci of the powder were measured by all participants at four different stress levels. The stresses in Table 1 correspond to the stress levels applied during the test. The testing includes automatic and time-based steady-state tests. These two measurement modes are described in section 2.1.1. Each shear measurement was performed with a triplicate repetition and fresh sample filling. This round robin test focuses on the measurement of the CRM 116 powder at three different stress levels (3, 6 and 9 kPa) to simulate low to high powder loading.

Table 1: Stress levels for yield locus function tests.

Stress level	Normal stress at the pre-shear points [kPa]	Normal stress at shear-to-failure points [kPa]			
	P	1	2	3	4
1	3.0	1.0	1.5	2.0	1.0
2	6.0	2.0	3.0	4.0	2.0
3	9.0	3.0	5.0	7.0	3.0

2.1.1 Automatic and Time-Based Steady-State

The difference between the settings of the automatic and time-based steady-state modes lies in the control of the number or quality of the measurement points.

In automatic mode, the quality of the measurement points is controlled by observing the results over 20 points, which must be within a specified tolerance range of ± 0.2 %. These constraints must be maintained beyond five averaging points to obtain a valid result. In contrast, in the time-based mode, only the number of measurement points for each measurement interval is specified, defining the duration of the measurement interval. In this test, the number of pre-shear points was set to 200 for the first pre-shear point and 100 for subsequent pre-shear points with a measurement duration of 1 second per point. The number of measurement points for the shear-to-failure curves was set to 200 (with a measurement duration of 0.3 seconds per point).

3 Measuring Setup

The measurements were performed using an MCR rheometer from Anton Paar equipped with an 18.9 mL Powder Shear Cell (Figure 2).

Figure 2: Anton Paar’s Powder Shear Cell with annular shear cell (18.9 mL) and overflow ring and shear system PSC43.

4 Results

The repeatability and reproducibility of the shear stress points were evaluated by calculating the mean values τ_m and the corresponding standard deviation:

$$\tau_m = \frac{1}{n}\sum_{i=1}^{n}\tau_i$$

Equation 1: Equation for calculating the average shear stress. τ_i represents the individual shear stress points and n represents the number of measurements.

$$s = \sqrt{\frac{\sum_{i=1}^{n}(\tau_i - \tau_m)^2}{n-1}}$$

Equation 2: Equation for calculating the standard deviation between the shear stress points.

4.1 Repeatability

Repeatability is used to measure the variations in successive measurements under constant conditions (same instrument and operator). The data of one testing laboratory were used for this purpose. For the determination of the repeatability, a fivefold determination was performed with a constant operator and under automatic steady-state measurement conditions. The mean value of the shear stress at the shear-to-failure points is given in Table 2 together with the observed standard deviation and the relative standard deviation.

Table 2: Results of the fivefold determination under constant measurement conditions (constant operator and measurement laboratory) and with automatic steady-state settings.

Stress level	Consolidation normal stress [kPa]	Mean shear stress at stress point [kPa]	Standard deviation of shear stress values [Pa]	Relative standard deviation of shear stress values [%]
1	1.0	1.19	35.9	2.99
	1.5	1.60	44.8	2.80
	2.0	1.97	47.4	2.41
	3.0	2.41	48.9	2.03
2	2.0	2.19	68.4	3.13
	3.0	2.94	66.4	2.26
	4.0	3.66	55.4	1.51
	6.0	4.66	104	2.23
3	3.0	3.03	69.7	2.30
	5.0	4.57	76.7	1.68
	7.0	5.98	122	2.04
	9.0	6.75	108	1.60

4.2 Reproducibility

Reproducibility is a measure of whether the specified measurement results (certified standard) can be achieved by a group of different research teams following the same method. This shows that the results obtained are not the consequence of the special circumstances in a research laboratory.

The following histograms show the mean value of the shear stresses at different normal stress levels. Each histogram contains the certified reference value with the permissible uncertainty range of the powder CRM 116 and the calculated mean values, which can be used for automatic and time-based steady-state measurements.

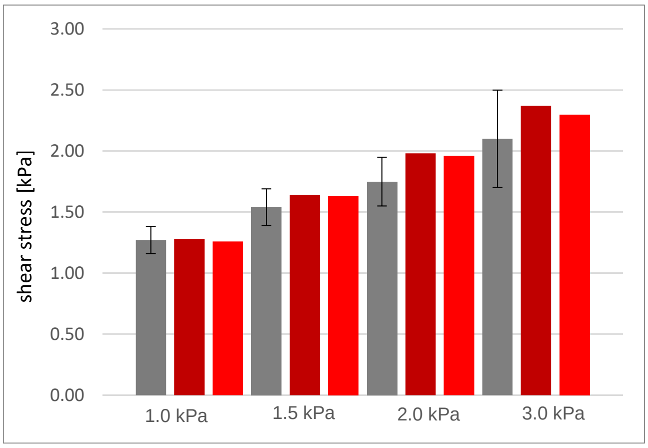


Figure 3: Stress points at shear level 1 (preshear: 3 kPa) in the automatic steady-state mode (dark red) and in the time-based steady state mode (light red). The reference value from the CRM certificate with the permissible uncertainty is shown in gray.

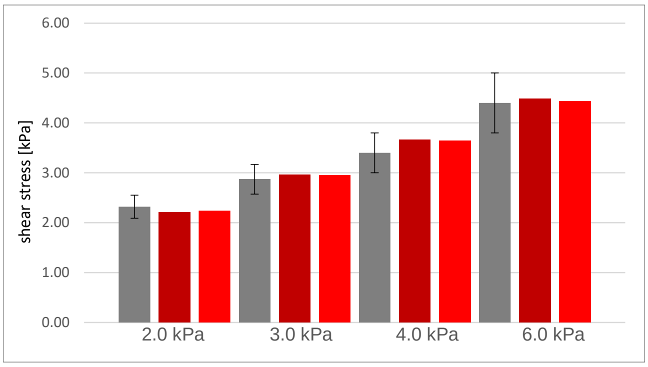


Figure 4: Stress points at shear level 2 (preshear: 6 kPa) in the automatic steady-state mode (dark red) and in the time-based steady state mode (light red). The reference value from the CRM certificate with the permissible uncertainty is given in gray.

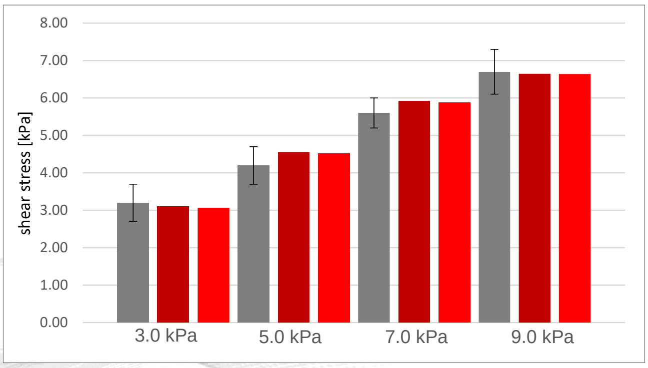


Figure 5: Stress points at shear level 3 (preshear: 9 kPa) in the automatic steady-state mode (dark red) and in the time-based steady state mode (light red). The reference value from the CRM certificate with the permissible uncertainty is given in gray.

Both, the automatic and the time-based steady-state values are within their mean shear stress values in the limited uncertainty range defined by the certificate of the limestone powder.

4.3 Automatic vs Time-Based Steady-State

The measured shear stress points τ are plotted against the normal stress σ_n in order to obtain the course of the measuring points. The diagrams represent the number of points corresponding to a yield locus test. In addition to plotting the average point, the standard deviation range around the mean value ($\tau_m \pm s$) is shown by dotted lines.

Note: The course of the yield curve does not correspond to the yield locus function.

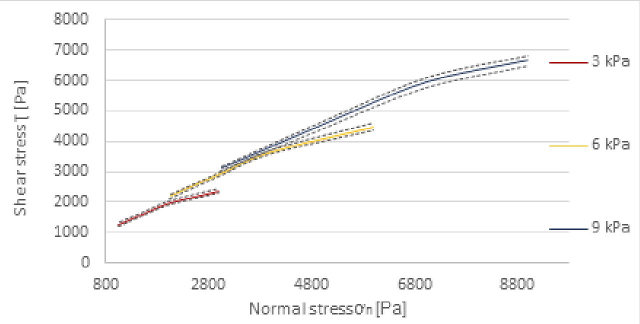


Figure 6: Stress points $\tau_m \pm s$ at shear level 1-3 in automatic steady-state mode.

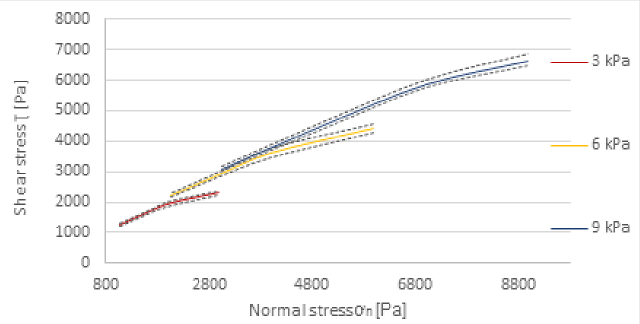


Figure 7: Stress points $\tau_m \pm s$ at shear level 1-3 in time-based steady-state mode.

From previous examinations of powder shear testers (3) it is known that annular shear cells have a higher accuracy and a lower standard deviation. The relative standard deviation in % is calculated from the ratio of the standard deviation and the mean shear stress. Based on these data, the range of the relative standard deviation is 1.8 up to 3.9 % in automatic steady-state mode. For time-based steady-state mode measurements, a relative standard deviation of 2.6 up to 4.3 % was determined. The numerical values can be found in the appendix (Table 2 and Table 4).

5 Conclusion

In summary, the round robin test shows that the Powder Shear Cell offers high reproducibility and repeatability of shear stresses for accurate yield locus measurements and analyses. Both measurement modes showed similar relative standard deviations with a slightly lower deviation of the shear stress values in the automatic steady-state measurement mode. Nevertheless, both measurement modes lead to good and reliable results, which correspond to the certified limestone powder range.

6 References

1. **Schulze, Dietmar.** Flow Properties of Powders and Bulk Solids. *Powders and Bulk Solids.* s.l. : Springer, 2021, Vol. 2nd ed.

2. **Joint Reseach Center.** ***Certified reference material catalogue.*** [Online] https://crm.jrc.ec.europa.eu/p/40455/40468/By-material-matrix/Other-manufactured-materials/BCR-116-LIMESTONE-POWDERS-for-shear-testing/BCR-116. (Date of viewing the webpage: 24.05.2022)

3. **Schulze, Dietmar.** *Shear tests on limestone powder CRM-116 with Ring Shear Tester RST-XS and RST-01.pc.* 16 March 2009.

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Appendix

Table 2: Determination of the reproducibility of the measurement result using automatic steady-state settings. The data were obtained in triplicate from different laboratories and different operators.

Stress level	Consolidation normal stress [kPa]	Mean shear stress at stress point [kPa]*	Standard deviation of shear stress values [Pa]	Relative standard deviation of shear stress values [%]
1	1.0	1.28	50.0	3.92
	1.5	1.64	48.3	2.95
	2.0	1.98	61.2	3.09
	3.0	2.37	73.0	3.09
2	2.0	2.21	71.9	3.26
	3.0	2.96	67.7	2.29
	4.0	3.66	79.4	2.17
	6.0	4.48	128.2	2.86
3	3.0	3.10	54.8	1.77
	5.0	4.56	138	3.03
	7.0	5.92	183	3.10
	9.0	6.65	179	2.70

*all mean shear stress values are within the 95% certainty range (3, 6, 9 kPa) of the CRM limestone powder. Only at stress level 1 (pre-shear normal stress of 3 kPa and a shear-to-failure normal stress of 2 kPa) a variance of 0.03 kPa was found.

Table 3: Determination of the reproducibility of the measurement result using time-based steady-state settings. The data were obtained under triple determination from five different laboratories and different operators.

Stress level	Consolidation normal stress [kPa]	Mean shear stress at stress point [kPa]**	Standard deviation of shear stress values [Pa]	Relative standard deviation of shear stress values [%]
1	1	1.26	42.0	3.33
	1.5	1.63	65.2	4.00
	2	1.96	83.2	4.24
	3	2.30	85.9	3.73
2	2	2.24	68.9	3.08
	3	2.95	92.9	3.15
	4	3.64	157	4.32
	6	4.43	138	3.12
3	3	3.07	87.4	2.85
	5	4.52	116	2.57
	7	5.87	158	2.68
	9	6.64	238	3.59

**all mean shear stress values lie within the 95% certainty range (3, 6, 9 kPa) of the CRM limestone powder. Only at stress level 1 (pre-shear normal stress of 3 kPa and a shear-to-failure normal stress of 2 kPa) a variance of 0.01 kPa was found.