Dr. Dietmar Schulze
Powder testers & software

Dr. Dietmar Schulze GmbH
Am Forst 20
D-38302 Wolfenbüttel
Tel.: +49 5331 935490
Fax: +49 5331 978001
E-mail: mail@dietmar-schulze.de
www.dietmar-schulze.de

Last update: 29 May 2022

Round Robin Project

Shear tests on limestone powder CRM-116 with Ring Shear Testers RST-XS and RST-01.pc

1 Introduction

In 1977 the Working Party on the Mechanics of Particulate Solids (WPMPS) of the European Federation of Chemical Engineering (EFCE) started a project to compare results from Jenike Shear Cell tests performed in different laboratories [1,2]. The outcome of the study was a detailed description of the operation of the Jenike Shear Tester which was published as the "Standard Shear Testing Technique …" (SSTT) [3] and, later, has become an ASTM standard [4].

For the shear tests a limestone powder was used which is available under the name "CRM-116" from the Institute for Reference Materials and Measurements of the European Commission[*)]. The test results obtained in the 1980's with Jenike Shear Testers in five of the involved laboratories are distributed with the powder samples, i.e., for a series of yield loci measured at different stress levels the shear stresses at preshear and shear to failure are given (mean value and 95% confidence interval obtained from the tests in the five laboratories [2]; these results are called the "certified results" in the following). Thus, one can purchase a sample of CRM-116, run shear tests and compare the results with the certified values.

Since the certified results have been obtained with Jenike Shear Testers, they may be slightly different from those obtained with other test equipment. Furthermore, the shear tests for the certification were performed in a number of labs, whereas for the certified values only the results of the five "best" of the involved labs have been taken into account. If the results of all labs were used, the range of uncertainty would probably be broader.

In the document delivered with the CRM-116 powder [2], the individual test results of the labs, whose results have been used for the calculation of the certified results, are listed in tables. The shear stresses measured in some of these labs are in some cases close to the upper border of the range of uncertainty, whereas other labs obtained shear stresses which were more than 20% lower. The source of the problem may be the manual operation of the Jenike Shear Tester including the required manual preconsolidation procedure ("twisting" [3,4]). Thus, it can be concluded that the certified results represent a range of results obtained with Jenike Shear Testers. However, the aim of the distribution of the results and the publication of the SSTT [3] was to help people who start

[*)] Unfortunately, the samples are no longer available since approx. 2021. A similar material is eskal 500 from KSL Staubtechnik (https://www.ksl-staubtechnik.de).

working with a Jenike Shear Tester by allowing them to adjust their procedure and equipment to the expected results.

In the last years the Schulze Ring Shear Tester is increasingly used in universities as well as in industry. If CRM-116 powder is tested with an automatic Ring Shear Tester, the shear stresses show less scatter compared to the (manually operated) Jenike Shear Tester, i.e., with the automatic Ring Shear Testers narrower 95% confidence intervals are obtained (e.g., [5]). Furthermore, the Ring Shear Tester results are close to the upper border of the 95% confidence interval of the certified results. Thus, for the validation of Ring Shear Testers, e.g., PQ tests as required in Pharmaceutical Industry, the certified results obtained with Jenike Shear Testers may be not optimal. Therefore, it is proposed to generate Ring Shear Tester results on limestone powder CRM-116 based on a round robin test series performed by as many as possible participants. Due to the requirements of Pharmaceutical Industry, the study is focused on the small automatic Ring Shear Tester RST-XS, but also users of the Ring Shear Tester RST-01.pc are invited to take part.

2 Test procedure

All participants in the round robin project should follow the procedures outlined below.

1. The shear tests should be done using limestone powder CRM-116. This material is available from the Institute for Reference Materials and Measurements of the European Commission IRMM [6] (Comment 2022: see footnote on page 1).

2. In the test report of the Jenike Shear Tests on CRM-116 it is stated that air humidity has an influence on the flow properties, and the limestone powder was prepared by storing it at a defined air humidity and temperature prior to the tests [2]. From other shear tests on limestone powder one can see that the influence of air humidity is limited if the relative humidity does not exceed 60% RH. To keep things simple, the following procedure is proposed for the round robin tests: The powder sample (with the RST-XS, 0.5 kg may be sufficient for all tests; material can be reused a number of times, e.g., for future PQ testing) should be stored in contact to the lab atmosphere until equilibrium is reached, minimum two days (temperature between 20°C and 25°C, humidity between 30% RH and 50% RH). The lab atmosphere should be stable during this time. Temperature and humidity should be monitored and communicated with the test results (for example, this information could be entered in the field “order” in RST-CONTROL 95 prior to each test).

   If a humidity chamber is available, it is recommended to use it for the storage of the limestone powder prior to the tests. Temperature and humidity should be adjusted to 20°C and 52% RH (according to proposal in the report of the Jenike Shear Tests [2]). 52% relative humidity can be attained also by equilibrating the powder with a saturated solution of sodium dichromate at 20°C (see Section 7.2 in [2]). Furthermore, it would be very interesting and appreciated much if those participants in the round robin project, who have the ability to store the powder in a humidity chamber, run the shear tests also with a powder specimen equilibrated at 30% RH and 40% RH (all at 20°C).

3. Yield loci should be measured by all participants at two different stress levels as follows (stresses correspond to stresses applied in the Jenike Shear Tester project; just the number of different stresses is reduced):

Stress level	normal stress at preshear [Pa]	normal stress at shear to failure [Pa]			
		1st point	2nd point	3rd point	4th point
1	3000	1000	1500	2000	1000
2	6000	2000	3000	4000	2000

It would be appreciated if tests are also performed voluntarily at higher stress levels according to the table below (these additional tests will enhance the base of the round robin):

Stress level	normal stress at preshear [Pa]	normal stress at shear to failure [Pa]			
		1st point	2nd point	3rd point	4th point
3	9000	3000	5000	7000	3000
4	15000	5000	7000	9000	5000

Control Files (*.CTF) containing the stresses listed above can be downloaded at www.dietmar-schulze.de/roundrobin.html. If these files are copied in subfolder “CTRL” in the RST-CONTROL program folder, the normal loads for the test can be directly imported from these files (please refer to Section 6.2.1.1 in the RST-CONTROL 95 manual).

In the RST-CONTROL 95 software ”N-RHOB-correction” should be activated (in menu “Preferences” in RST-CONTROL 95 select “Test procedure”. After the window “Test procedure settings” has appeared, activate “N-RHOB-correction” on tab sheet “Parameters”). All other parameters (shear velocity, tolerance, patience) should be set to their default values.

4. Ensure that the Ring Shear Tester is adjusted properly. Especially the horizontal position of the lid of the shear cell must be correct, i.e., the lid must be centric to the shear cell and must not touch the shear cell side walls (see Section 7 in the Ring Shear Tester manual).

   For all yield locus tests a standard shear cell should be used (RST-XS: shear cell XS-Mr; RST-01.pd: shear cell M). Use a freshly prepared cell for each yield locus test. Do not run two tests with the same filled cell.

5. Finally the test results should be saved as EIF files and e-mailed to Dietmar Schulze for statistical evaluation. If the range of results is acceptable, even if the ambient conditions were different within the given range, mean values and confidence intervals will be calculated similar to the Jenike Shear Tester results and distributed to all round robin partners. These results may then be the base for PQ testing and similar applications.

   From the scientific point of view, a publication of the test results may be of interest to the powder technology community. Submission of data will signify acceptance of the fact that data will be made public. Please indicate if your company agrees to be named in a publication as a participant in the round robin project. It is not intended to link individual test results to names of participants.

6. Timing: The goal is to finish all shear tests by May 31st, 2008. This leaves enough time to order limestone CRM-116 and to run all tests. However, it is appreciated if test results are sent before the deadline. Test results received after the deadline will be included in the data base at a later time.

3 References

[1] Carson, J.W., Wilms, H.: Development of an international standard for shear testing, Powder Technol. 167 (2006), pp 1-9

[2] Akers, R.J.: EUR14022 – The certification of a limestone powder for Jenike shear testing. Publ. by the Commission of the European communities (1992), pdf is available on *www.irmm.jrc.be* (see [6])

[3] The Institution of Chemical Engineers (Publ.): Standard shear testing technique for particulate solids using the Jenike shear cell. Rugby, England, UK (1989)

[4] ASTM Standard D6128-00: Standard test method for shear testing of bulk solids using the Jenike shear cell. ASTM International, *www.astm.org* (2000)

[5] Verlinden, A: Experimental assessment of shear testers for measuring flow properties of bulk solids. Ph.D. thesis, Univ. of Bradford, UK (2000)

[6] IRMM: Institute for Reference Materials and Measurements of the European Commission, Web link: *http://www.irmm.jrc.be/rmcatalogue/searchrmcatalogue.do* (enter material ”limestone powder”).