

Global Reference Laboratory
 RE-SI-Q-GRL
 BL Silica

MAIL-RE-SI-Q-01

ANM 0144 - Recertification of IRM 100

Content

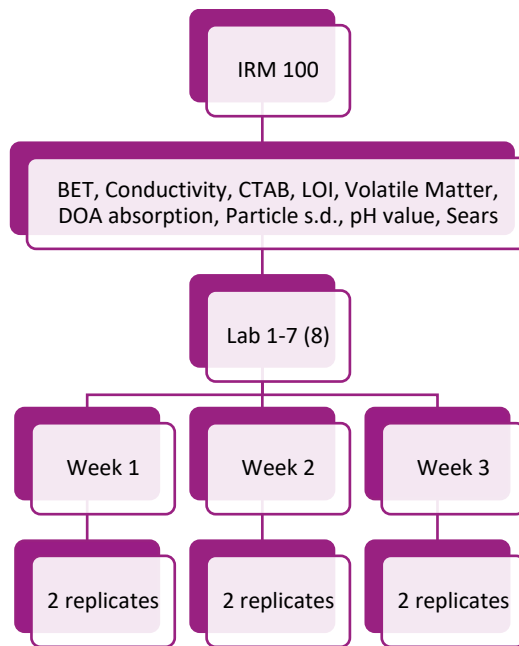
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Background

In 2015 IRM 100 has been released as commercially available Silica Reference Material. Distributor is Balentine Enterprises Inc.

Some characteristics showed ageing effects over time. In 2019 it was decided within the ASTM Round Table (coordinated by Jorge Lacayo-Pineda) that the IRM 100 material shall be recertified based on a Round Robin testing of ASTM members.

Study setup



Test characteristics

BET surface area MP	ASTM D1993
Conductivity, 4% (m/V)	ASTM D8300
CTAB surface area SSA	ASTM D6845
Volatile Matter, 2h/105 °C	ASTM D6738
Loss on ignition 2h/1000°C	ISO 3262-1
Oil absorption (DOA)	ISO 19246
Optional: Particle size, d50%	ISO 13320
pH 5 % (m/m)	ASTM D6739
Sears Number	ASTM D8016
Dynamic Void Volume	experimental

Summary

Based on the collected results (initial and latest measurement values) and approved by the ASTM Round Table, the following limits come into effect:

Specification parameters acc. ASTM D5900

	ASTM Designation	Unit	Mean	Within lab			Between labs		
				Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
BET SA	D1993	m ² /g	164.5	1.23	2.46	3.71	2.36	4.72	7.08
CTAB SA	D6845	m ² /g	162.8	0.67	1.34	2.00	2.29	4.59	6.88
Volatile Matter	D6738	%	3.2	0.12	0.23	0.35	0.13	0.27	0.40
pH (5%)	D6739		5.9	0.04	0.08	0.12	0.08	0.16	0.24
Conductivity (4%)	ISO 787	µS/cm	146.4	2.24	4.48	6.73	7.13	14.27	21.40
Loss on Ignition (orig. mat.)	ISO 3262	%	5.9	0.07	0.14	0.21	0.18	0.36	0.54

Additional information

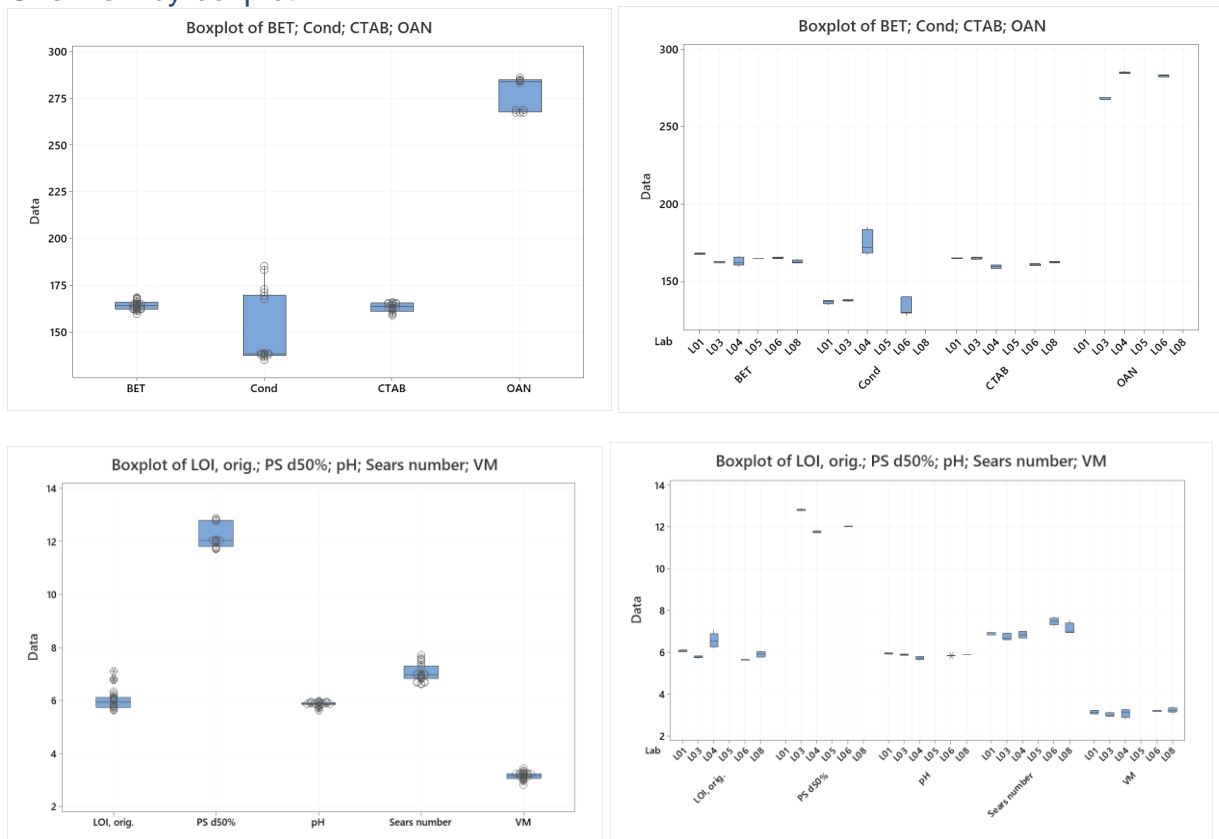
	ASTM Designation	Unit	Mean	Within lab			Between labs		
				Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
Sears Number	D8016	mL/1.5 g	7.0	0.17	0.34	0.51	0.31	0.62	0.93
Oil Absorption (DOA)	ISO 19246	mL/100g	279.1	0.73	1.46	2.18	7.44	14.88	22.32
Particle size, d50%	ISO 13320	µm	12.2	0.04	0.08	0.12	0.46	0.92	1.38

Beyond the agreed test plan, tentative data for dynamic void volume was determined:

	ASTM Designation	Unit	Mean	Within lab			Between labs		
				Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
Dyn. Void Volume, orig subst. @5 MPa	experimental	mL/100g	186.3	1,43	2,86	4,29	1,67	3,34	5,01
Dyn. Void Volume, degassed, @5 MPa	experimental	mL/100g	197.6	2,77	5,54	8,32	4,57	9,14	13,72

Results

Overview by boxplot



Regarding the boxplot diagrams the following characteristics are suspicious:

- Conductivity Lab 04 is significantly increased
- OAN Only 3 datasets, wide spread
- Loss on ignition Lab 04 shows high variation
- Particle size d50% Only 3 datasets

Descriptive statistics overview:

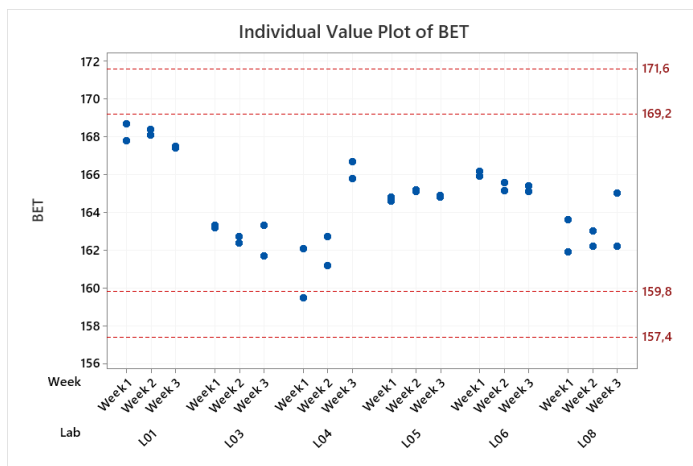
Variable	Lab	N	Mean	StDev	CoefVar	Minimum	Maximum	Range
BET	L01	6	168,0	0,51	0,3	167,4	168,7	1,3
	L03	6	162,8	0,64	0,4	161,7	163,3	1,6
	L04 ¹⁾	6	163,0	2,75	1,7	159,5	166,7	7,2
	L05	6	164,9	0,22	0,1	164,6	165,2	0,6
	L06	6	165,6	0,44	0,3	165,1	166,2	1,1
	L08	6	163,0	1,17	0,7	161,9	165,0	3,1
Cond	L01	6	136,9	1,30	1,0	134,9	138,5	3,6
	L03	6	138,1	0,59	0,4	137,0	138,7	1,7
	L04 ²⁾	6	174,9	7,51	4,3	167,4	185,3	17,9
	L05	0	*	*	*	*	*	*
	L06	6	133,3	5,47	4,1	128,2	140,4	12,2
	L08	0	*	*	*	*	*	*
CTAB	L01	6	165,2	0,46	0,3	164,5	165,7	1,2
	L03	6	165,3	0,59	0,4	164,6	166,1	1,5
	L04	6	159,9	0,97	0,6	158,7	160,9	2,2
	L05	0	*	*	*	*	*	*
	L06	6	161,2	0,70	0,4	160,2	161,9	1,8
	L08	6	162,6	0,50	0,3	161,9	163,1	1,2
LOI, dried s.	L01	6	3,0	0,08	2,6	2,9	3,1	0,2
	L03	6	2,8	0,05	1,7	2,8	2,9	0,1
	L04 ³⁾	6	3,6	0,51	14,1	3,2	4,3	1,1
	L05	0	*	*	*	*	*	*
	L06	6	2,5	0,04	1,6	2,5	2,5	0,1
	L08	6	2,8	0,19	6,8	2,6	3,1	0,5
LOI, original substance	L01	6	6,1	0,04	0,7	6,0	6,2	0,1
	L03	6	5,8	0,05	0,9	5,7	5,8	0,1
	L04 ³⁾	6	6,6	0,36	5,5	6,2	7,1	0,9
	L05	0	*	*	*	*	*	*
	L06	6	5,6	0,03	0,5	5,6	5,7	0,1
	L08	6	5,9	0,14	2,3	5,8	6,1	0,3
OAN	L01	0	*	*	*	*	*	*
	L03	5	268,0	0,70	0,3	267,4	268,8	1,4
	L04	6	284,7	0,78	0,3	283,7	286,0	2,3
	L05	0	*	*	*	*	*	*
	L06	6	282,6	0,71	0,2	281,5	283,6	2,1
	L08	0	*	*	*	*	*	*
PS d50%	L01	0	*	*	*	*	*	*
	L03	6	12,8	0,04	0,3	12,8	12,9	0,1
	L04	6	11,8	0,05	0,4	11,7	11,8	0,1
	L05	0	*	*	*	*	*	*
	L06	6	12,0	0,03	0,2	12,0	12,1	0,1
	L08	0	*	*	*	*	*	*
pH	L01	6	6,0	0,02	0,4	5,9	6,0	0,1
	L03	6	5,9	0,03	0,5	5,9	5,9	0,1
	L04	6	5,7	0,08	1,4	5,6	5,8	0,2
	L05	0	*	*	*	*	*	*
	L06	6	5,9	0,01	0,2	5,8	5,9	0,0
	L08	6	5,9	0,02	0,3	5,9	5,9	0,0

Sears number	L01	6	6,9	0,06	0,9	6,8	7,0	0,2
	L03	6	6,7	0,15	2,2	6,6	6,9	0,3
	L04	6	6,9	0,14	2,0	6,7	7,0	0,3
	L05	0	*	*	*	*	*	*
	L06	6	7,5	0,17	2,3	7,3	7,7	0,5
	L08	6	7,1	0,26	3,7	6,9	7,6	0,6
VM	L01	6	3,1	0,08	2,4	3,0	3,2	0,2
	L03	6	3,0	0,09	2,9	2,9	3,2	0,2
	L04	6	3,1	0,19	6,2	2,8	3,3	0,5
	L05	0	*	*	*	*	*	*
	L06	6	3,2	0,04	1,1	3,2	3,3	0,1
	L08	6	3,3	0,12	3,8	3,1	3,4	0,4

- 1) Borderline high standard deviation, but remains in evaluation
- 2) Unexpected high result level; excluded from further evaluation
- 3) This group of replicates was excluded based on significantly higher standard deviation from comparison groups (> 3K)

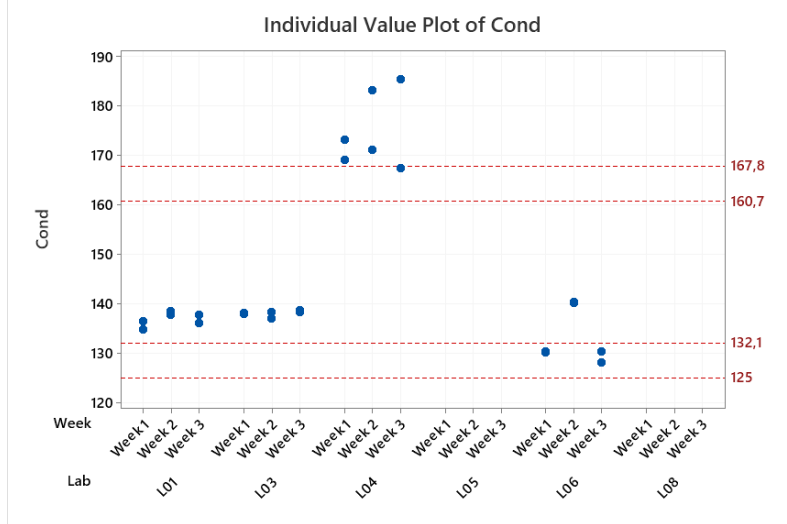
BET surface area

	Mean (AR-value) m ² /g	Within lab			Between labs		
		Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
		m ² /g	m ² /g	m ² /g	m ² /g	m ² /g	m ² /g
original BET SA	167,9	0,77	1,53	2,30	1,20	2,40	3,60
updated BET SA (=study data)	164,5	1,23	2,46	3,71	2,36	4,72	7,08



Conductivity

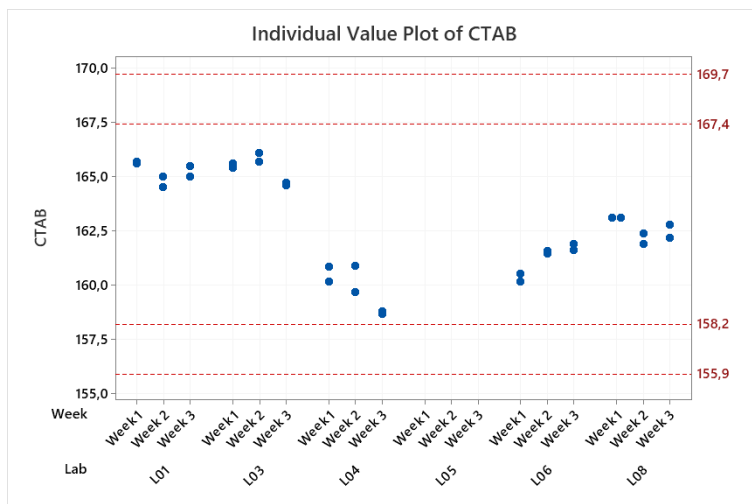
		Within lab			Between labs		
	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
	µS/cm	µS/cm	µS/cm	µS/cm	µS/cm	µS/cm	µS/cm
original Conductivity	146,4	2,24	4,48	6,73	7,13	14,27	21,40
study data	136,1	3,26	6,52	9,78	3,73	7,46	11,19
updated Conductivity	Originally certified values remain unchanged. The data count is low, and the formerly specified limits are confirmed and still applicable.						



L04 has been excluded from evaluation

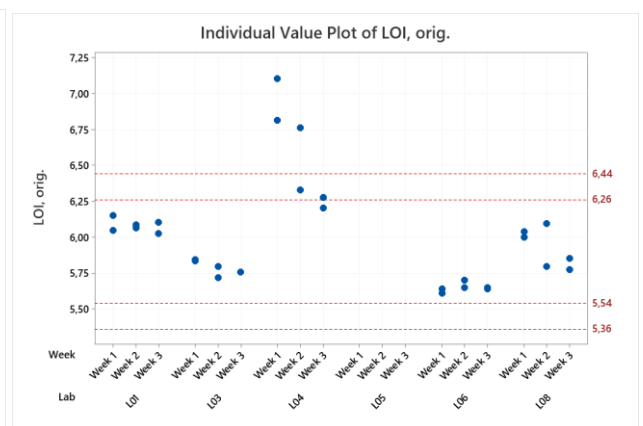
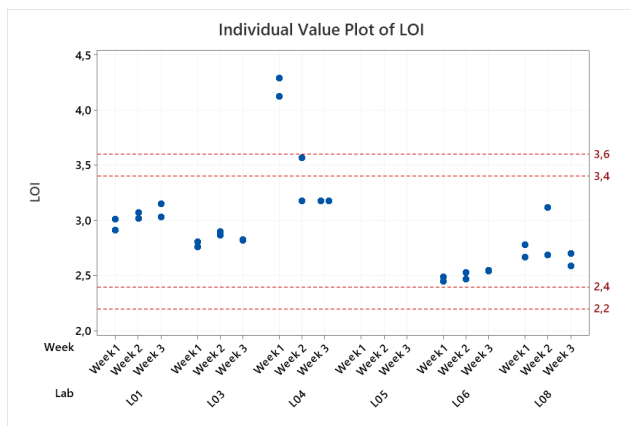
CTAB surface area

		Within lab			Between labs		
	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
	m ² /g	m ² /g	m ² /g	m ² /g	m ² /g	m ² /g	m ² /g
original CTAB	165,1	1,14	2,28	3,43	1,67	3,34	5,01
updated CTAB (=study data)	162,8	0,67	1,34	2,00	2,29	4,59	6,88



Loss on Ignition

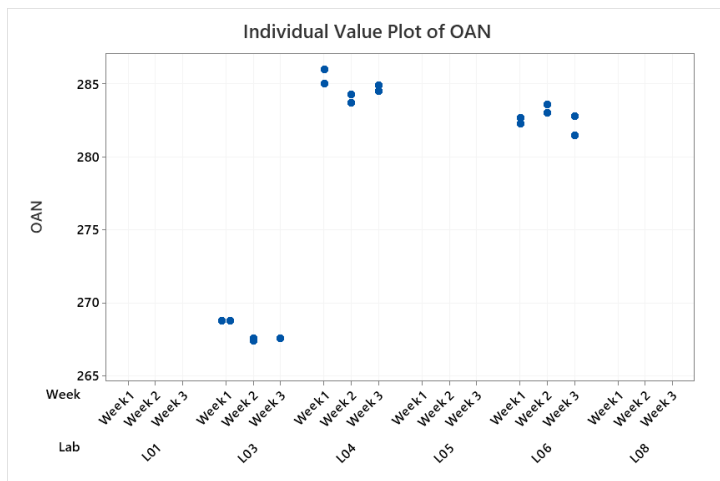
		Within lab			Between labs		
	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
	%	%	%	%	%	%	%
original LOI	2,9	0,10	0,20	0,30	0,23	0,47	0,70
study data	2,8	0,10	0,20	0,30	0,24	0,48	0,72
LOI, original substance	6,0	0,07	0,14	0,21	0,18	0,36	0,54
updated LOI	The formerly specified limits are confirmed and still applicable. With new revision, LOI based on original substance is reported instead of based on dry substance						



L04 has been excluded from evaluation

Oil absorption number

		Within lab			Between labs		
	Mean (AR-value)	Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
	mL/100g	mL/100g	mL/100g	mL/100g	mL/100g	mL/100g	mL/100g
original OAN	265,6	1,40	2,79	4,19	2,61	5,21	7,82
study data	279,1	0,73	1,46	2,18	7,44	14,88	22,32
updated OAN	New values are stated, but only informational. The observed differences and low count (3 labs) can't be assessed reasonably.						



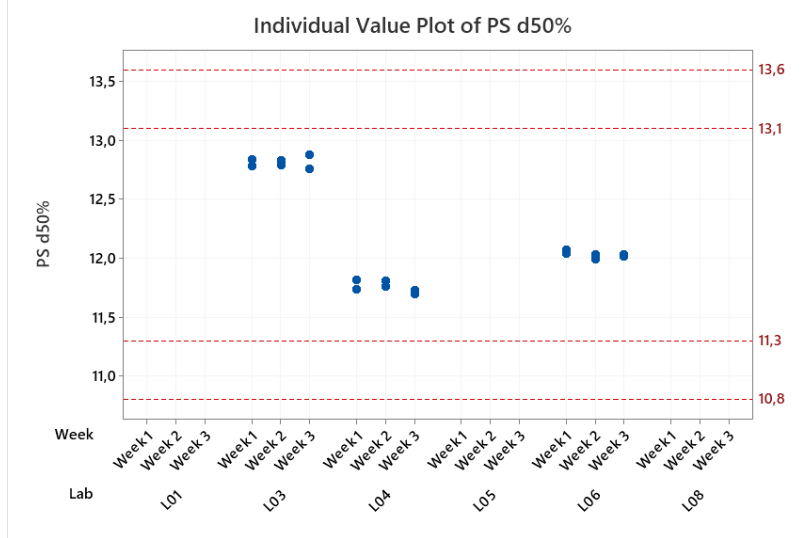
Date: 03.02.2021

Global Reference Laboratory

Responsible person: Timo Bahlke

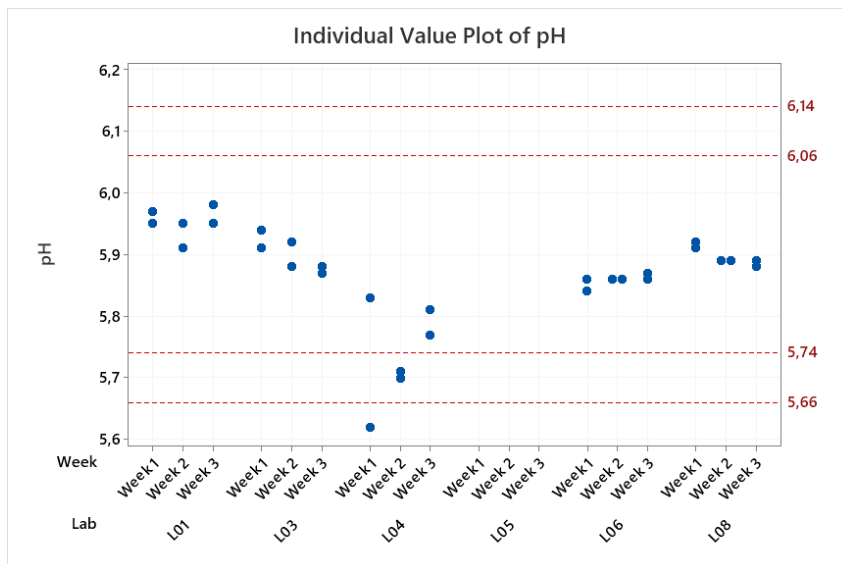
Particle size, d50%

	Mean (AR-value)	Within lab			Between labs		
		Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
		µm	µm	µm	µm	µm	µm
original PS, d50%	12,2	0,20	0,39	0,59	0,65	1,30	1,95
study data	12,2	0,04	0,08	0,12	0,46	0,92	1,38
updated PS, d50%	Originally certified values remain unchanged. The data count is low, and the formerly specified limits are confirmed and still applicable.						



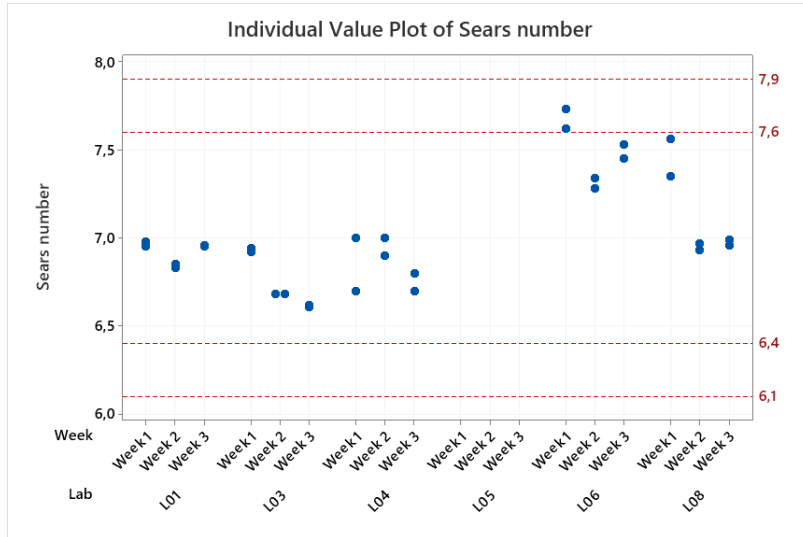
pH value

	Mean (AR-value)	Within lab			Between labs		
		Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
original pH	5,5	0,04	0,08	0,12	0,12	0,25	0,37
updated pH (=study data)	5,9	0,04	0,08	0,12	0,08	0,16	0,24



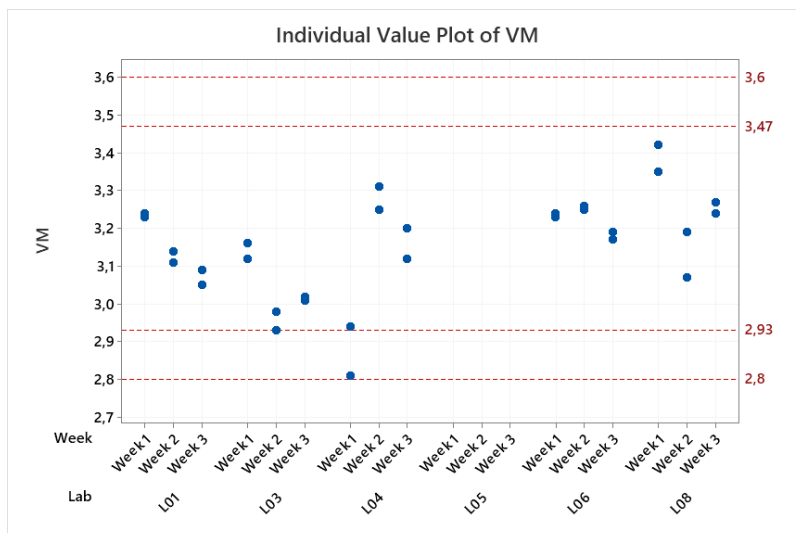
Sears number – newly introduced

	Mean (AR-value) mL/1,5 g	Within lab			Between labs		
		Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
		mL/1,5 g	mL/1,5 g	mL/1,5 g	mL/1,5 g	mL/1,5 g	mL/1,5 g
Sears Number	7,0	0,17	0,34	0,51	0,31	0,62	0,93



Volatile matters

	Mean (AR-value) %	Within lab			Between labs		
		Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
		%	%	%	%	%	%
original VM	3,2	0,11	0,21	0,32	0,16	0,32	0,48
study data	3,2	0,12	0,23	0,35	0,13	0,27	0,40
updated VM	Originally certified values remain unchanged. The formerly specified limits are confirmed and still applicable.						



Dynamic Void Volume Analysis - experimental

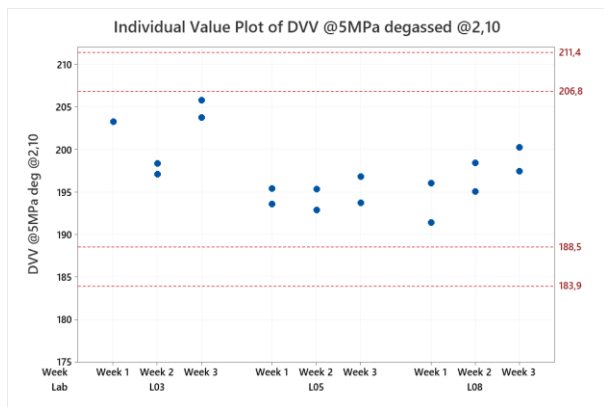
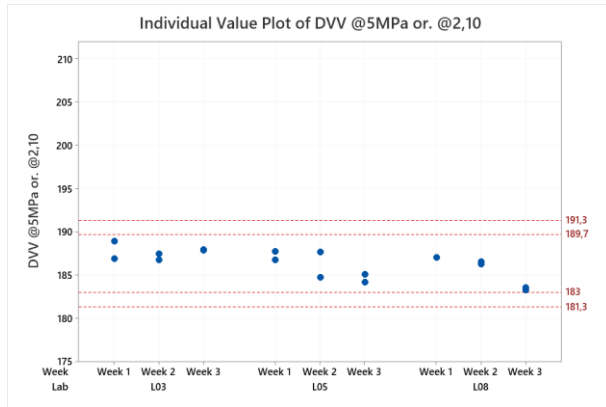
Beyond the agreed test plan, tentative data for dynamic void volume was determined. Count is low (n = 18), three laboratories participated. The indicated limits are therefore only for orientation.

The results are based on discussed test conditions:

Skeletal density: 2,10 g/cm³

Testing of original material, without consideration of contained moisture at all ("original substance", no moisture or mass correction) and degassed sample (dried before testing).

	ASTM Designation	Unit	Mean	Within lab			Between labs		
				Sr	2 x Sr	3 x Sr	SR	2 x SR	3 x SR
Dyn. Void Volume, orig subst. @5 MPa	experimental	mL/100g	186.3	1,43	2,86	4,29	1,67	3,34	5,01
Dyn. Void Volume, degassed, @5 MPa	experimental	mL/100g	197.6	2,77	5,54	8,32	4,57	9,14	13,72



Raw data

Lab	Week	BET	Cond.	CTAB	LOI	DOA/OAN	PS d50%	pH	Sears	VM
L01	W 1	168,7	134,9	165,7	2,9			6,0	7,0	3,2
L01	W 1	167,8	136,5	165,6	3,0			6,0	7,0	3,2
L01	W 2	168,1	138,5	165,0	3,0			5,9	6,9	3,1
L01	W 2	168,4	137,7	164,5	3,1			6,0	6,8	3,1
L01	W 3	167,4	136,2	165,0	3,0			6,0	7,0	3,1
L01	W 3	167,5	137,7	165,5	3,2			6,0	7,0	3,1
L03	W 1	163,3	138,2	165,6	2,8	268,8	12,8	5,9	6,9	3,2
L03	W 1	163,2	138,0	165,4	2,8	268,8	12,8	5,9	6,9	3,1
L03	W 2	162,7	137,0	166,1	2,9	267,4	12,8	5,9	6,7	3,0
L03	W 2	162,4	138,4	165,7	2,9	267,6	12,8	5,9	6,7	2,9
L03	W 3	163,3	138,7	164,7	2,8	267,6	12,9	5,9	6,6	3,0
L03	W 3	161,7	138,4	164,6	2,8		12,8	5,9	6,6	3,0
L04	W 1	159,5	173,2	160,2	4,3	285,0	11,8	5,8	6,7	2,9
L04	W 1	162,1	169,1	160,9	4,1	286,0	11,7	5,6	7,0	2,8
L04	W 2	161,2	183,1	160,9	3,6	283,7	11,8	5,7	6,9	3,3
L04	W 2	162,7	171,2	159,7	3,2	284,3	11,8	5,7	7,0	3,3
L04	W 3	165,8	185,3	158,7	3,2	284,9	11,7	5,8	6,7	3,1

Date: 03.02.2021

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Responsible person: Timo Bahlke

L04	W 3	166,7	167,4	158,8	3,2	284,5	11,7	5,8	6,8	3,2
L05	W 1	164,8								
L05	W 1	164,6								
L05	W 2	165,1								
L05	W 2	165,2								
L05	W 3	164,9								
L05	W 3	164,8								
L08	W 1	163,6		163,1	2,7			5,9	7,4	3,4
L08	W 1	161,9		163,1	2,8			5,9	7,6	3,4
L08	W 2	163,0		162,4	2,7			5,9	6,9	3,2
L08	W 2	162,2		161,9	3,1			5,9	7,0	3,1
L08	W 3	162,2		162,2	2,6			5,9	7,0	3,3
L08	W 3	165,0		162,8	2,7			5,9	7,0	3,2

Lab	Week	DVV @5MPa	
		orig. @2,10	deg. @2,10
L03	W 1	188,9	203,3
L03	W 1	186,9	203,2
L03	W 2	186,8	198,4
L03	W 2	187,4	197,1
L03	W 3	187,8	203,7
L03	W 3	187,9	205,8
L05	W 1	187,7	195,4
L05	W 1	186,7	193,6
L05	W 2	187,6	195,4
L05	W 2	184,8	192,9
L05	W 3	184,2	193,7
L05	W 3	185,1	196,8
L08	W 1	187,0	196,0
L08	W 1		191,4
L08	W 2	186,2	195,0
L08	W 2	186,5	198,4
L08	W 3	183,5	197,4
L08	W 3	183,2	200,2