Bayer Industry Services

Validation Study on the Calculation of Dry Matter by Determination of Dry Residue and Water Content and Loss on Ignition in Waste

Evaluation of the validation study on

prEN 14346 and

prEN 15169

organised by CEN/TC 292 / WG 5

sponsored by

German Federal States Program

“Water, Soil, Waste”

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Content

1 Scope

Determination of dry matter in waste materials is subject of standard prEN 14346:2005-08. Dry matter may be determined by drying the samples at 105 °C or calculated based on water content. For this reason, prEN 14346 describes both direct sample drying and determination of water. Water determination may be performed using an azeotropic distillation or a titration method according to Karl Fischer.

Loss on ignition in waste material is described in Standard prEN 15169:2005-02.

The present report describes a collaborative interlaboratory experiment which was performed in order to provide basic data concerning reproducibility and repeatability of both standards mentioned above.

2 Summary

An overview of the most important data (relative reproducibility and relative repeatability standard deviation) is given in the table below. In case of the determination of water by azeotropic distillation, not enough data are available to validate this method.

Standard	Sample	mean [% w/w]	relative reproducibility standard deviation SR (%)	relative repeatability standard deviation Sr (%)
prEN 14346 Dry matter by drying at 105°C	Contaminated soil	93,3	0,5	0,1
	Dredged sludge	66,4	1,0	0,4
	Nickel sludge	53,0	2,1	0,6
prEN 14346 Water by Karl Fischer Titration	Contaminated soil	6,4	16,6	3,5
	Destillation residue	9,0	9,3	2,2
	Dredged sludge	30,7	11,3	2,6
	Drilling solution	97,5	1,9	1,0
	Nickel sludge	38,8	11,6	1,9
	Waste oil	69,2	2,8	1,2
prEN 15169 Loss on ignition of dry matter	Contaminated soil	5,6	6,1	2,4
	Dredged sludge	11,4	8,5	2,0
	Nickel sludge	11,7	11,3	2,6

3 Organisation

- Sponsor:
  German federal states program „water, soil, waste“
  (Länderfinanzierungsprogramm “Wasser, Boden Abfall”)

- Project coordinator:
  Dr. Klaus Furtmann, Landesumweltamt Nordrhein-Westfalen, Düsseldorf/Germany

- Contractor:
  Bayer Industry Services GmbH, Leverkusen/Germany

- Sample materials:
  Members of CEN/TC 292 WG 5

4 Participants

Participating laboratories are given in table 1. They represent eight European countries..

Country	105°C drying	azeotropic distillation	Karl-Fischer titration	loss on ignition
A	9	1	1	9
B	1	0	0	1
D	11	3	7	11
DK	2	1	1	2
F	4	0	13	14
NL	1	0	1	1
FIN	3	2	1	3
SCG	2	1	1	2
Total	33	8	25	43

For details of the participating laboratories see table 1.

5 Samples

5.1 Type of samples and sample preparation

5.1.1 Contaminated Soil

The sample was taken from an old industrial site. It was sieved (2 mm) and spiked with activated charcoal (4 %, w/w) to ensure practical data for loss on ignition. Afterwards, it was homogenized by shaking in a plastic drum.

5.1.2 Dredged Sludge

The sample was taken from a stagnant riparian water zone at river Rhine. In order to obtain a sample ready for homogenisation it was pre-dried at room temperature for about 30 hours, sieved (2 mm) and homogenized in a plastic drum.

5.1.3 Nickel Sludge

This mixture of inorganic waste material consisted mainly of carbonates resulting from a metallurgical process. The material was passed through a 3 mm sieve. In order to obtain a sample ready for homogenisation it was pre-dried at room temperature overnight and homogenized in a plastic drum.

5.1.4 Drilling solution

This sample was an oil-water emulsion that has been used for drilling in metal processing. Laboratory samples were prepared while the sample (about 7 L) was homogenized in a 10 L- glass container by vigorous stirring.

5.1.5 Waste Oil

This sample was from an oil regeneration plant. It was a mixture of waste engine oil and hydraulic fluid. It was homogenized in the same way as in the case of the drilling solution.

5.1.6 Distillation residue

A mixture of organic compounds was from a pharmaceutical production process. It consisted of low boiling alcohols and ketones, e.g. methanol and acetone.

6 Homogeneity and stability

For investigation of homogeneity, repeated analyses from different sample vessels were performed. Data from homogeneity testing are given in table 2.

Stability and in-bottle homogeneity of samples was tested by 3 to 4 repeated analyses from one sample container. Additional analyses were performed during the experimental phase of the study. Results showed that all analytes were stable.

7 Shipping of samples

After homogenisation, samples were filled into screw cap glass bottles with a volume of 50 ml, 100 ml or 500 ml. typically, each glass bottle contained about 40 g, 80 g or 350 g of sample. Samples were shipped to the laboratories by truck or by air mail. Shipping started at Nov. 9th 2005. The latest sample shipping was finished at Dec 2nd 2005.

8 Analysis of samples

The three solid samples (contaminated soil, dredged sludge and Nickel sludge) were intended for analysis according to all methods. The liquid samples (drilling emulsion, waste oil, distillation residue) were analysed with the azeotropic distillation and the Karl-Fischer method.

	Drying at 105°C	Azeotropic distillation	Karl-Fischer titration	Loss on ignition
Contaminated soil	X	X	X	X
Dredged sludge	X	X	X	X
Nickel sludge	X	X	X	X
Drilling emulsion		X	X	
Waste oil		X	X	
Distillation residue		X	X	

9 Data Evaluation

Original laboratory results were evaluated according to DIN ISO 5725-2:2002-12. A commercial software package (ProLab/QuoData) was used for calculations and graphical data presentation. We thank Dr. Furtmann (Landesumweltamt Nordrhein-Westfalen, Düsseldorf/Germany) for technical assistance.

10 Results

10.1 Laboratory Data

Complete data delivered by participating laboratories are listed in table 3.

10.2 Summary of results

Performance characteristics of prEN 14346 and pr EN 15169 are given in Table 4.

11 Annex

Table 1: Participating laboratories

Institution/Organisation	City	City Code
Bayer Industry Services SUA PUA 1	Leverkusen	D-51368
SCORI chez CIMENTS CALCIA	Beaucaire	F-30300
ECN	Petten	NL-1755
Staatliches Umweltamt	Münster	D-48147
Landeslabor Brandenburg FB 03	Potsdam	D-14467
Universität für Bodenkultur	Wien	A-1190
SCORI	Airvault	F-79600
SCORI	St. Pierre la Cour	F-53410
Umweltbundesamt Fachgebiet III 3.4	Berlin	D-12307
Institute of Public Health	Belgrad	SCG-11000
SCORI	Barlin	F-62620
SCORI	Lillebonne	F-76170
IMAT-UVE GmbH	Mönchenglad-bach	D-41066
Österreichisches Forschungsinstitut	Wien	A-1110
Umweltbundesamt	Wien	A-1090
SCORI	Montalieu	F-38390
SITA FD	Nanterre	F-92758
Umweltlabor Dr. Axel Begert GmbH	Bachmanning	A-4672
BAM	Berlin	D-12489
SCORI	Frontignan	F-34110
UCL Umwelt Control Labor GmbH	Lünen	D-44536
Fernwärme Wien GmbH	Wien	A-1110
Consulting Engineers Paavo Ristola	Hollola	FIN-15870
TERIS RON	St. Maurice l'Exil	F-38556
Center of Ecotoxicological Research of	Podgorica	SCG-81000
Kommunekemi a/s	Nyborg	DK-5800
Bayer Industry Services SUA PUA 2	Dormagen	D-41538
Fernwärme Wien GmbH	Wien	A-1110
Lenzing AG ZB Umweltschutz	Lenzing	A-4860
Ekokem Oy Ab	Riihimäki	FIN-11101
Labo Services	Givors	F-69702
TU Dresden Institute of Waste Management	Pirna	D-01796
Dr. Weßling Laboratorien GmbH	Bochum	D-44793
SGS Institut Fresenius GmbH	Berlin	D-10245
SCORI	Givors	F-69701
Amt der Salzburger Landesregierung Abt. 16	Salzburg	A-5010
Chemcon GmbH	Wien	A-1020
VITO	Mol	B-2400
RTR Süd-Ouest	Oriolles	F-16480
VTT Processes	Espoo	FIN-02150
Force Technology	Brondby	DK-2605
SCORI	Xeuilley	F-54990

Table 2: Homogeneity and stability testing

		dry residue (105°C) %	water (Karl-Fischer titration) %	Loss on ignition %
contaminated soil	mean	92,94		6,04
	standard deviation	0,05		0,07
	relative standard deviation	0,06		1,23
Dredged sludge	mean	66,85		12,68
	standard deviation	0,18		0,24
	relative standard deviation	0,27		1,87
Nickel sludge	mean	53,39		14,03
	standard deviation	0,57		0,35
	relative standard deviation	1,08		2,46
Drilling emulsion	mean		98,03	
	standard deviation		0,71	
	relative standard deviation		0,72	
Waste oil	mean		69,83	
	standard deviation		0,22	
	relative standard deviation		0,31	
Distillation residue	mean		9,16	
	standard deviation		0,07	
	relative standard deviation		0,81	

Table 3: Detailed laboratory results.

All data given in % (w/w)

	Contaminated soil				Dredged sludge			
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)			prEN 15169
	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter
1	94,73			5,99	67,77			13,71
	94,8			5,55	66,77			13,22
2	93,20			5,70	66,10			11,10
	93,30			5,60	66,20			11,60
	93,10			5,80	66,10			11,20
3	92,70	6,08		5,87	65,85	29,32		13,43
	92,71	6,17		5,90	65,80	28,68		13,62
	92,71	6,32		6,10	65,70	29,32		14,18
4	93,03			87,69	66,36			57,60
	92,89			87,66	66,26			61,70
				87,95				58,50
5	93,19			5,75	66,43			11,87
	93,17			5,70	66,38			11,68
	93,35			5,50	66,66			11,39
6	94,10		5,64	5,50	66,10		32,90	11,50
	94,10		5,51	5,50	66,20		31,03	11,80
	94,30		5,61	5,50	66,40		32,91	11,85
7	92,83			5,61	66,30			11,26
	92,93			5,57	66,28			10,99
	93,00			5,56	66,04			11,01
8	93,15			5,08	66,19			10,26
	93,09			4,64	66,04			9,35
	93,14			4,49	66,09			10,13
9	92,74			5,55	65,90			11,50
	92,90			5,57	66,10			11,20
	93,02			5,50	65,60			11,20
10	93,23			5,41	66,46			10,74
	93,37			5,48	66,29			10,92
	93,22			5,38	66,53			10,92
12	93,00			5,90	67,00			12,30
	92,90			6,00	66,80			12,60
	92,90			6,10	66,60			12,90
13	93,10	5,10	6,77	5,50	66,00	32,40	32,68	11,30
	93,00	5,10	6,31	5,60	66,00	31,80	32,66	11,20
	93,20	5,50	6,54	5,50	66,10	32,70	32,67	10,80

	Contaminated soil				**Dredged sludge**			
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)			prEN 15169
	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter
15	93,86		5,81		66,76		34,63	
	93,68		6,02		66,48		33,71	
	93,40		6,25		66,90		31,86	
16	92,00			5,35	66,00			10,51
	93,10			5,36	66,10			10,37
	93,00			5,25	65,80			10,46
17	93,62			5,93	66,59			12,33
	93,71			6,01	66,36			12,48
	93,62			5,92	66,47			11,97
18	94,26		5,85	5,60	6,77		7,13	5,90
	94,06		5,84	5,60	6,88		7,03	6,00
	94,23		5,85	5,70	7,18		7,08	5,90
20	92,95	5,79	6,85	4,03	66,14	35,48	30,22	10,51
	93,01	6,99	6,82	3,77	66,06	31,15	30,01	10,66
	93,07	7,58	6,86	4,33	66,02	31,47	30,21	10,41
21	93,00	8,50	9,40	6,10	65,60	39,00	37,60	13,20
	93,00	9,50	9,40	6,10	66,10	32,40	37,60	13,00
	93,00	9,00	9,40	6,20	66,00	32,00	38,30	13,50
24	93,70			5,20	66,00			10,10
	93,70			5,40	66,20			10,60
	93,70			5,50	66,30			10,40
25	93,15			5,80	66,21			11,28
	93,46			6,33	66,37			11,15
	93,60			5,98	67,02			11,20
26	93,58			5,28	68,01			10,30
	93,36			4,29	67,96			10,75
	93,49			5,55	68,84			10,73
30	93,08			5,38	66,04			11,20
	93,12			5,22	65,99			11,33
	93,24			5,25	66,01			10,87
31	92,80	6,80	4,04	5,28	65,90	34,00	26,50	10,76
	92,80	6,80	4,22	4,80	65,80	34,00	31,70	10,72
	92,80	6,80	4,65	5,20	65,90	34,00	33,40	10,81
33			6,90				31,00	
			6,10				28,40	
			6,10				29,70	
34			6,90				28,10	
			8,90				32,80	
			7,00				28,80	

	Contaminated soil				**Dredged sludge**			
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)			prEN 15169
	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter
36	92,80		6,55		65,97		32,85	
	92,98		6,56		65,69		31,06	
	92,76		6,88		65,79		31,76	
37	93,40		8,40		66,80		23,60	
	93,50		7,40		67,30		26,80	
	91,40		6,50		67,50		31,90	
38			7,08	12,00			27,60	41,00
			6,88	12,20			28,30	41,00
			7,21	12,00			28,35	41,00
39			6,6				26,60	
			6,80				25,20	
			6,10				25,70	
40			6,30	11,40			30,80	40,70
			6,50	11,50			30,90	40,90
			5,80	11,80			30,70	41,00
41			5,41				25,83	
			5,39				27,30	
			5,61				25,38	
42			6,80				25,65	
			6,30				26,35	
			6,50				25,75	
43	94,82			5,63	68,27			11,47
	94,15			5,69	67,50			11,81
	93,68			5,68	68,81			11,57
44	94,50				67,70			
	94,00				67,40			
	93,80				66,90			
47	93,13		6,50		66,97		31,51	
	93,14		6,71		66,29		30,31	
	93,10		6,67		66,30		31,31	
48	93,00			5,40	66,00	0,00	0,00	11,00
	93,00			5,60	66,00	0,00	0,00	11,00
	93,00			5,50	66,00	0,00	0,00	11,00
49	93,21	5,86	7,11	5,45	66,23	33,49	33,36	11,29
	93,11	6,21	7,08	5,49	66,40	30,05	33,65	11,20
	93,05	7,08	7,09	5,48	66,09	29,17	34,71	10,94
50	93,02	6,94		5,39	66,35	32,56		10,77
	93,06	6,88		5,15	66,06	32,54		10,63
	93,08	6,99		5,24	66,15	32,78		10,80

	Contaminated soil				**Dredged sludge**			
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)			prEN 15169
	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter	Method A Dry matter at 105°C	method B – azeotropic distillation	method C – Karl-Fischer titration	loss on ignition of dry matter
51	92,93	6,85		11,38	66,47	33,50		27,29
	93,03	6,76		11,28	66,06	33,20		27,33
	92,99	6,90		11,14	66,07	33,54		27,25
52	93,43			5,83	66,53			11,46
	93,48			5,89	66,42		98,76	11,42
	93,42			5,75	66,59			11,35

	Nickel sludge				**Waste oil**	
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)	
	Method A Dry matter at 105°C	method B azeotropic distillation	method C Karl-Fischer titration	loss on ignition of dry matter	method B azeotropic distillation	method C Karl-Fischer titration
1	54,8			22,87		
	54,26			22,28		
2	52,10			10,80		
	52,10			11,20		
	52,20			11,40		
3	51,69	41,18		22,27	69,74	
	51,30	40,41		22,67	61,70	
	51,50	40,58		22,60	67,52	
4	52,71			46,17		
	52,42			45,78		
	52,60			46,37		
5	54,27			13,46		
	53,94			12,98		
	53,01			12,36		
6	52,10		40,28	11,60		68,52
	53,10		42,05	12,60		68,61
	53,30		41,43	12,40		68,07
7	52,58			11,37		
	52,32			11,12		
	52,15			11,00		
8	53,18			11,06		
	52,99			10,93		
	53,01			10,26		

	Nickel sludge				**Waste oil**	
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)	
	Method A Dry matter at 105°C	method B azeotropic distillation	method C Karl-Fischer titration	loss on ignition of dry matter	method B azeotropic distillation	method C Karl-Fischer titration
9	51,90			11,10		
	52,10			10,90		
	52,00			10,80		
10	52,75			11,54		
	52,77			11,66		
	52,55			11,32		
12	54,00			13,60		69,50
	53,30			14,50		69,80
	53,00			14,00		69,70
13	52,40	37,90	41,15	11,10	70,7	84,24
	52,40	37,90	40,97	11,10	70,3	79,40
	51,90	42,00	42,94	10,80	69,2	82,53
15	54,71		48,17			70,79
	53,92		47,34			70,49
	53,48		49,37			70,69
16	51,90			9,94		
	51,90			10,22		
	51,80			9,94		
17	53,20			14,28		64,90
	53,27			13,71		65,51
	53,38			13,84		63,69
18	53,40		36,60	12,70		5,66
	53,66		36,80	12,60		5,78
	53,75		36,70	12,80		5,67
20	52,07	46,94	36,38	11,05	73,42	59,00
	51,79	46,69	36,37	10,44	70,22	59,21
	51,69	44,94	36,51	10,56	69,48	58,83
21	49,40	42,70	44,40	13,00	70,60	70,20
	51,70	43,80	43,90	12,80	71,60	69,90
	51,70	43,70	44,40	12,70	69,40	68,90
24	52,20			10,80		
	52,40			10,90		
	52,50			11,00		
25	53,52			11,88		
	52,28			11,49		
	53,26			11,17		
26	55,82			10,36		
	55,96			11,37		
	55,69			10,85		
30	52,42			11,66		
	52,51			11,62		
	53,11			11,58		

	Nickel sludge				**Waste oil**	
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)	
	Method A Dry matter at 105°C	method B azeotropic distillation	method C Karl-Fischer titration	loss on ignition of dry matter	method B azeotropic distillation	method C Karl-Fischer titration
31	51,90	44,00	38,30	9,48	70,00	60,20
	52,10	45,00	38,30	9,11	68,00	66,50
	51,90	44,00	39,50	8,95	70,00	62,10
32						82,00
						83,00
						82,50
33			31,20			69,50
			29,90			70,40
			30,10			67,50
34			39,30			71,10
			34,10			70,70
			38,30			70,60
35						70,18
						70,20
						70,80
36	51,94		41,56			70,61
	52,34		41,68			70,60
	52,15		41,54			69,96
37	52,40		36,60			71,00
	53,40		38,80			69,80
	53,10		32,20			72,80
38			38,90	54,40		68,60
			39,90	54,60		69,50
			40,80	54,40		69,50
39			40,70			68,10%
			40,80			68,20
			40,20			68,90
40			39,30	53,00		66,70
			37,40	53,10		67,40
			38,20	53,20		65,70
41			32,75			71,28
			31,41			69,69
			33,19			69,92
42			32,00			71,30
			31,60			70,50
			32,50			70,90
43	55,35			13,49		
	56,17			13,69		
	56,04			13,65		
44	54,60					66,80
	52,90					63,70
	52,10					70,70

	Nickel sludge				**Waste oil**	
Lab. No.	prEN 14346 (water content)			prEN 15169	prEN 14346 (water content)	
	Method A Dry matter at 105°C	method B azeotropic distillation	method C Karl-Fischer titration	loss on ignition of dry matter	method B azeotropic distillation	method C Karl-Fischer titration
47	52,98		46,14			66,10
	53,15		44,42			66,29
	52,80		45,60			64,99
48	54,00			14,00		
	54,00			13,00		
	54,00			14,00		
49	52,63	41,60	41,30	11,13	45,11	69,80
	52,41	45,65	41,69	10,81	66,32	72,20
	52,12	41,80	43,46	10,75	65,02	69,20
50	52,42			11,02		
	52,42			10,88		
	52,25			10,70		
51	53,31	45,58		26,95	68,80	
	53,44	45,48		26,18	68,40	
	53,72	45,52		26,19	68,32	
52	52,90			12,80		68,70
	53,06			12,75		68,00
	53,24			12,86		70,00

	Drilling emulsion		**Distillation residue**	
Lab. No.	prEN 14346 (water content)		prEN 14346 (water content)	
	method B azeotropic distillation	method C Karl-Fischer titration	method B azeotropic distillation	method C Karl-Fischer titration
3	98,19		48,18	
	98,93		43,43	
	93,27		42,64	
6		94,40		9,06
		96,61		9,16
		94,80		9,04
12		99,00		9,00
		97,90		9,10
		98,80		9,10
13	91,7	97,71		
	97	98,85		
	96,4	99,97		
15		99,94		10,40
		98,74		10,23
		98,57		10,31

	Drilling emulsion		**Distillation residue**	
Lab. No.	prEN 14346 (water content)		prEN 14346 (water content)	
	method B azeotropic distillation	method C Karl-Fischer titration	method B azeotropic distillation	method C Karl-Fischer titration
18		99,40		10,00
		99,10		9,88
		99,30		9,97
20	99,94	98,13		9,16
	99,79	98,69		9,08
	99,52	97,46		9,14
21	97,60	97,60		10,10
	98,00	97,50		9,80
	97,30	96,00		9,60
31	96,00	100,00	98,00	7,20
	98,00	101,00	99,00	7,40
	98,00	99,00	98,00	7,30
32		100,00		9,00
		100,00		9,50
		99,00		9,00
33		99,50		8,30
		96,80		8,80
		99,10		8,50
34		97,20		9,00
		96,70		9,00
		97,30		9,00
35		99,50		8,66
		98,20		8,83
		99,50		8,94
36		96,93		9,84
		97,30		9,81
		97,20		9,47
37		96,80		8,70
		93,70		8,00
		93,70		8,40
38		96,99		9,18
		94,92		9,51
		95,53		9,48
39		96,90		9,00
		97,10		9,00
		96,80		9,10
40		98,40		9,69
		96,10		9,76
		97,60		9,74
41		96,12		9,10
		97,29		9,31
		97,80		9,33

	Drilling emulsion		**Distillation residue**	
Lab. No.	prEN 14346 (water content)		prEN 14346 (water content)	
	method B azeotropic distillation	method C Karl-Fischer titration	method B azeotropic distillation	method C Karl-Fischer titration
42		95,50		8,90
		96,20		8,80
		95,90		9,30
44		91,40		6,50
		95,40		6,10
		93,40		7,70
47		96,34		9,32
		97,23		9,23
		99,74		9,65
49	89,80	>97	5,00	9,40
	85,26	>97	14,67	9,00
	76,49	>97	8,51	9,30
51	97,94			
	97,32			
	97,82			
52		97,10		9,06
		96,40		9,08
		97,90		9,04

Table 4: Summary of performance characteristics of prEN 14346 and prEn 15169

Standard	Sample	O %	p	N	Outliers	m [% w/w]	sR	SR	sr	Sr
pr EN 15169 Loss on ignition of dry matter,	Contaminated soil	9	32	95	9	5,58	0,34	6,14	0,13	2,39
	Dredged sludge	12	33	98	12	11,39	0,97	8,50	0,23	1,98
	Nickel sludge	10	30	89	9	11,74	1,32	11,26	0,31	2,65
prEN 14346 Water by azeotropic Destillation	Contaminated soil	0	8	24	0	6,77	1,12	16,59	0,74	6,49
	Dredged sludge	0	8	24	0	32,44	2,29	7,07	1,83	5,65
	Drilling solution	14	7	21	3	97,37	2,12	2,18	1,80	1,85
	Nickel sludge	0	7	21	0	43,21	2,73	6,32	1,36	3,14
	Waste oil	14	7	21	3	69,14	2,20	3,19	1,12	1,62
prEN 14346 Water by Karl Fischer Titration	Contaminated soil	10	20	60	6	6,44	1,07	16,63	0,23	3,54
	Destillation residue	0	22	66	0	9,04	0,84	9,34	0,20	2,17
	Dredged sludge	18	17	51	9	30,69	3,48	11,32	0,78	2,55
	Drilling solution	0	23	69	0	97,48	1,85	1,90	1,01	1,04
	Nickel sludge	12	17	51	6	38,77	4,48	11,55	0,74	1,90
	Waste oil	14	21	63	9	69,19	1,97	2,85	0,84	1,21
prEN 14346 Water content calculated from	Contaminated soil	12	33	98	12	6,74	0,45	6,63	0,10	1,42
	Dredged sludge	3	33	98	3	33,57	0,66	1,95	0,26	0,77
	Nickel sludge	3	33	98	3	47,04	1,08	2,29	0,34	0,71
pr EN 14346 Dry matter	Contaminated soil	12	33	98	12	93,25	0,45	0,48	0,10	0,10
	Dredged sludge	0	33	98	0	66,44	0,67	1,01	0,24	0,37
	Nickel sludge	3	33	98	3	52,96	1,09	2,07	0,32	0,61

p Number of laboratories before elimination of outliers

N Number of observed values

O Percentage of outliers

m General mean

sR Estimate of the reproducibility standard deviation

sr Estimate of the repeatability standard deviation

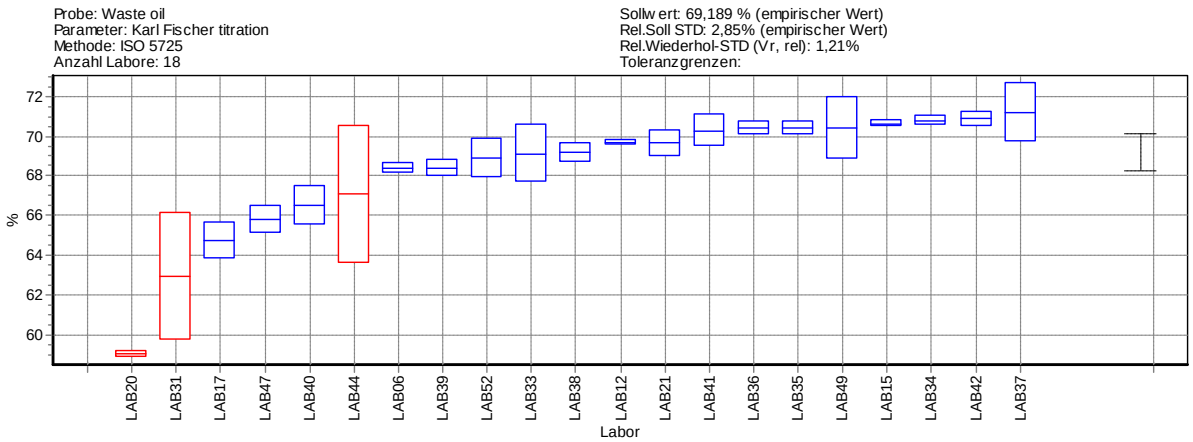
SR Estimate of the relative reproducibility standard deviation

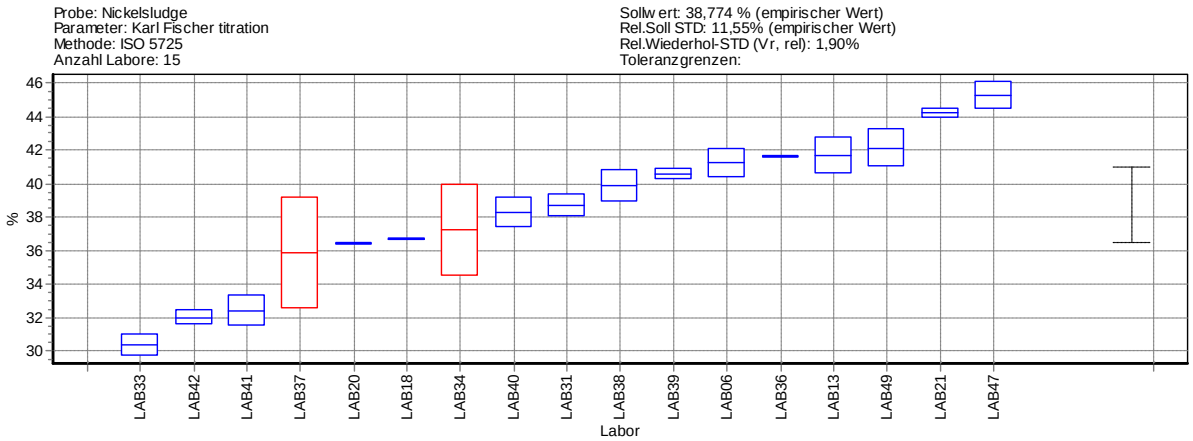
Sr Estimate of the relative repeatability standard deviation

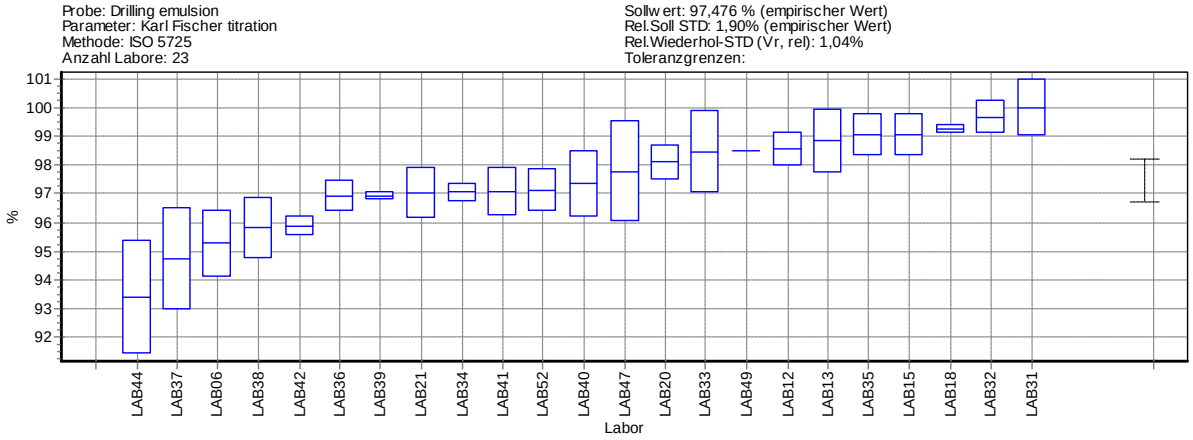
Graphical data presentation

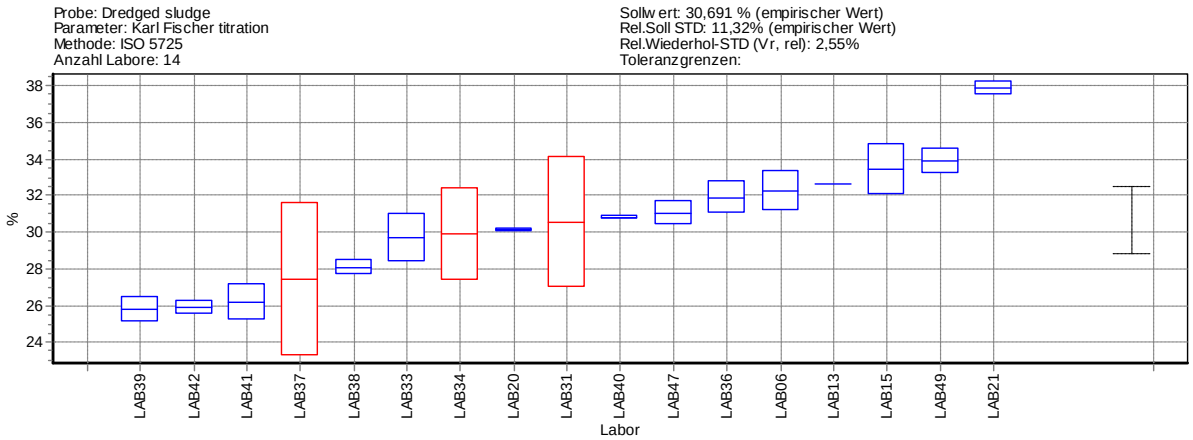
Translations: Probe (sample)
Anzahl Labore (number of laboratories)
Sollwert (mean value)
Rel. Soll-Std. (relative reproducibility standard deviation)
Rel. Wiederhol-STD (relative repeatability standard deviation)

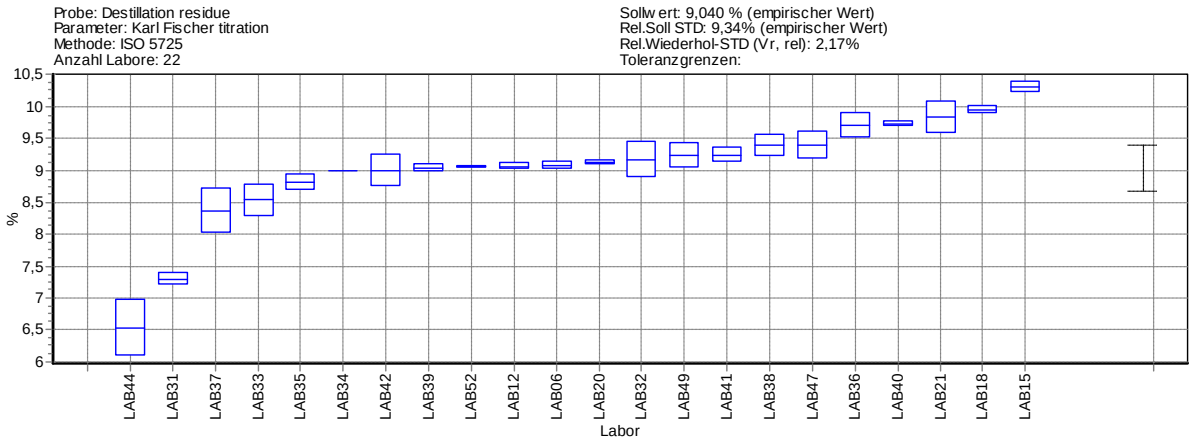
Karl Fischer Titration

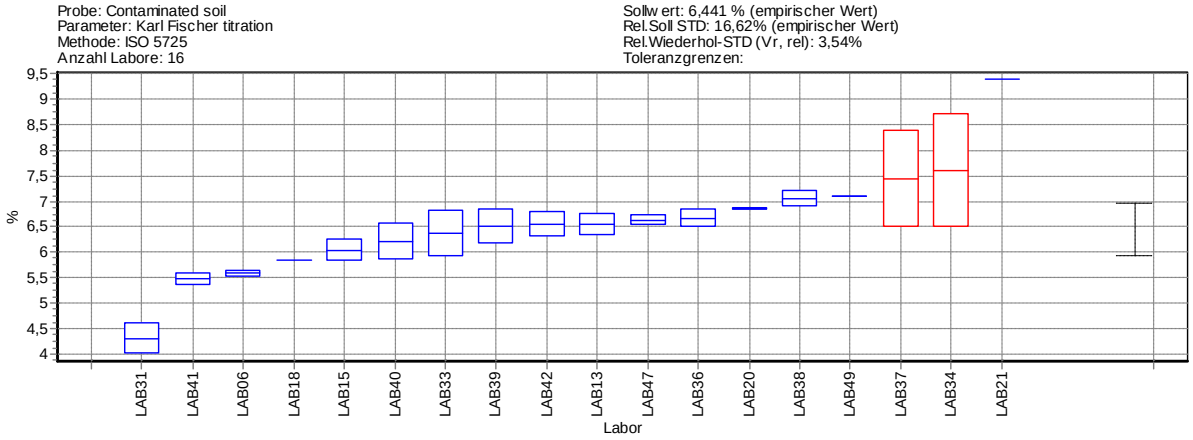




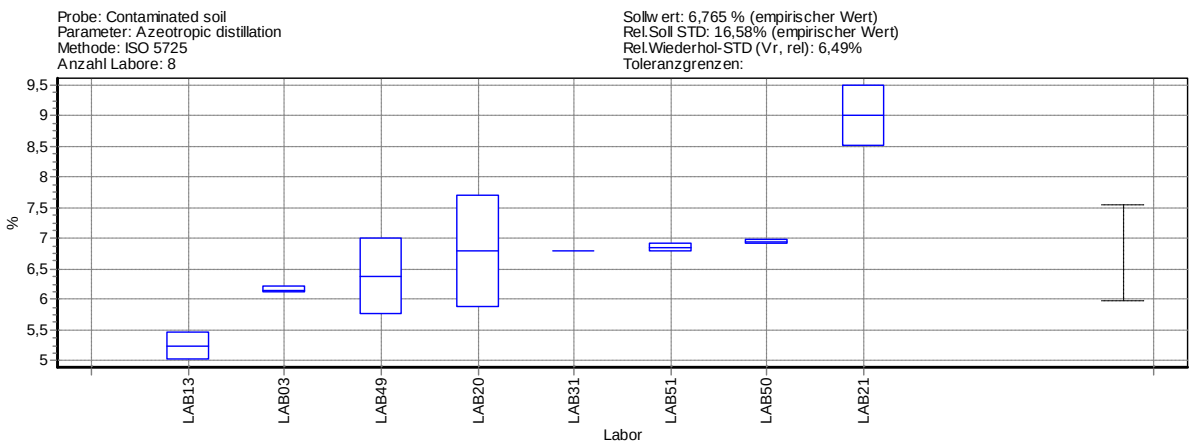

Probe: Drilling emulsion
Parameter: Karl Fischer titration
Methode: ISO 5725
Anzahl Labore: 23
Sollwert: 97,476 % (empirischer Wert)
Rel.Soll STD: 1,90% (empirischer Wert)
Rel.Wiederhol-STD (Vr, rel): 1,04%
Toleranzgrenzen:
%
101
100
99
98
97
96
95
94
93
92
LAB44 LAB37 LAB06 LAB38 LAB42 LAB36 LAB39 LAB21 LAB34 LAB41 LAB52 LAB40 LAB47 LAB20 LAB33 LAB49 LAB12 LAB13 LAB35 LAB15 LAB18 LAB32 LAB31
Labor
ProLab 2005

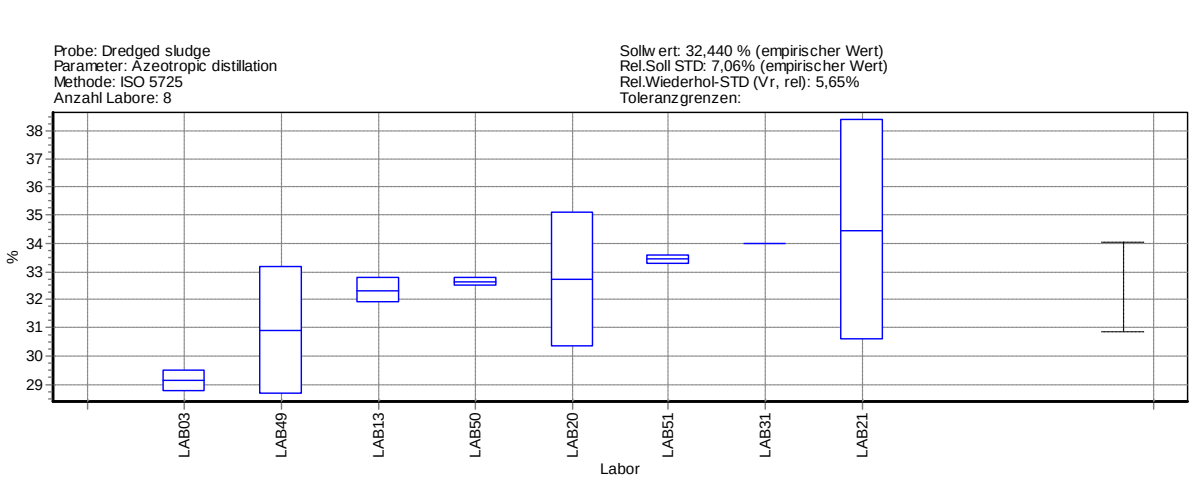

Probe: Dredged sludge
Parameter: Karl Fischer titration
Methode: ISO 5725
Anzahl Labore: 14
Sollwert: 30,691 % (empirischer Wert)
Rel.Soll STD: 11,32% (empirischer Wert)
Rel.Wiederhol-STD (Vr, rel): 2,55%
Toleranzgrenzen:
%
38
36
34
32
30
28
26
24
LAB39 LAB42 LAB41 LAB37 LAB38 LAB33 LAB34 LAB20 LAB31 LAB40 LAB47 LAB36 LAB06 LAB13 LAB15 LAB49 LAB21
Labor
ProLab 2005

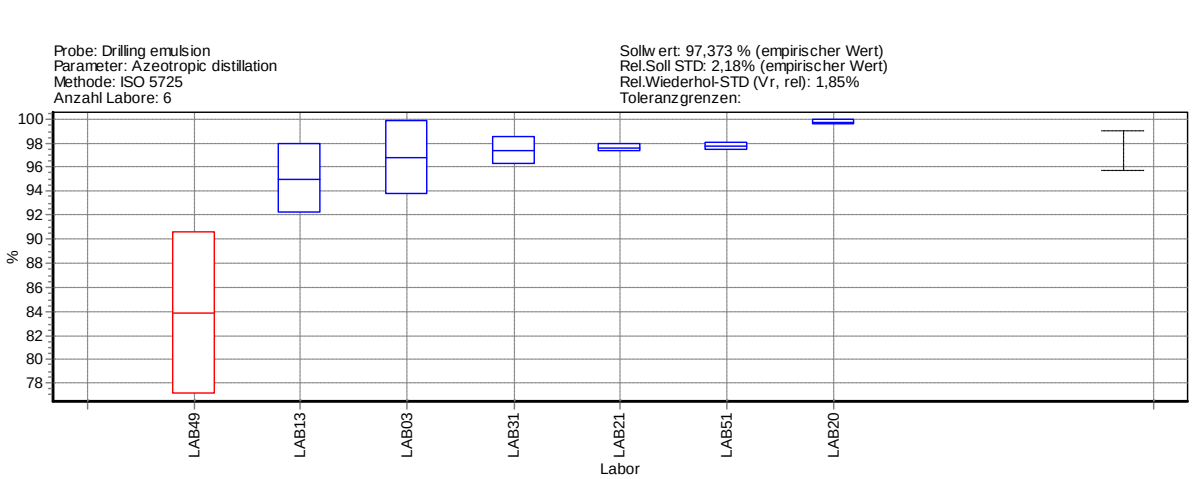

Probe: Destillation residue
Parameter: Karl Fischer titration
Methode: ISO 5725
Anzahl Labore: 22
Sollwert: 9,040 % (empirischer Wert)
Rel.Soll STD: 9,34% (empirischer Wert)
Rel.Wiederhol-STD (Vr, rel): 2,17%
Toleranzgrenzen:
%
10,5
10
9,5
9
8,5
8
7,5
7
6,5
6
LAB44 LAB31 LAB37 LAB33 LAB35 LAB34 LAB42 LAB39 LAB52 LAB12 LAB06 LAB20 LAB32 LAB49 LAB41 LAB38 LAB47 LAB36 LAB40 LAB21 LAB18 LAB15
Labor
ProLab 2005

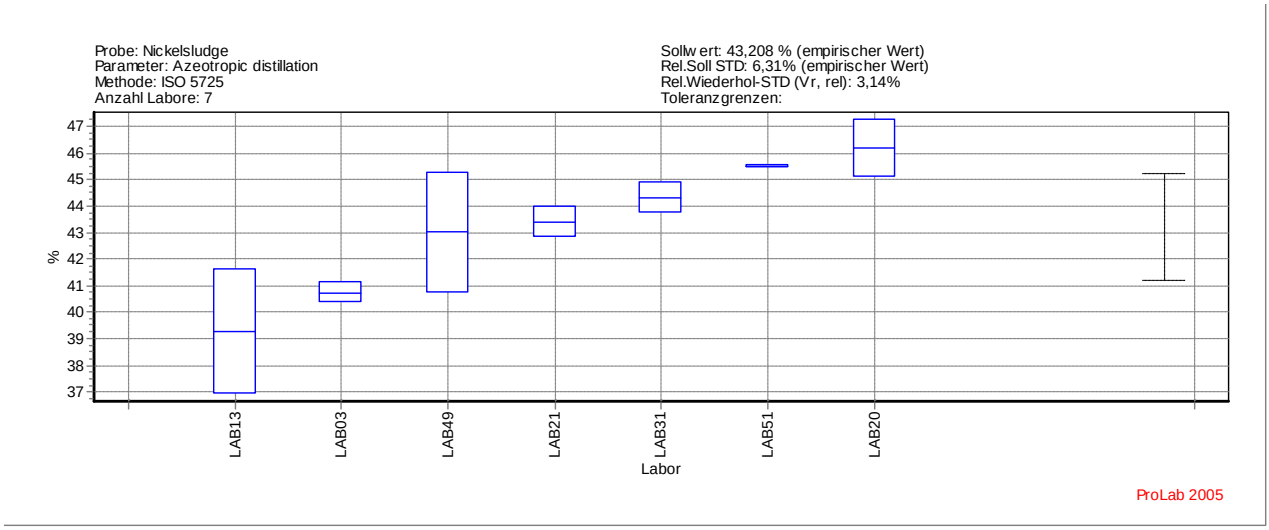

Probe: Contaminated soil
Parameter: Karl Fischer titration
Methode: ISO 5725
Anzahl Labore: 16
Sollw ert: 6,441 % (empirischer Wert)
Rel.Soll STD: 16,62% (empirischer Wert)
Rel.Wiederhol-STD (Vr, rel): 3,54%
Toleranzgrenzen:
%
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
LAB31
LAB41
LAB06
LAB18
LAB15
LAB40
LAB33
LAB39
LAB42
LAB13
LAB47
LAB36
LAB20
LAB38
LAB49
LAB37
LAB34
LAB21
Labor
ProLab 2005

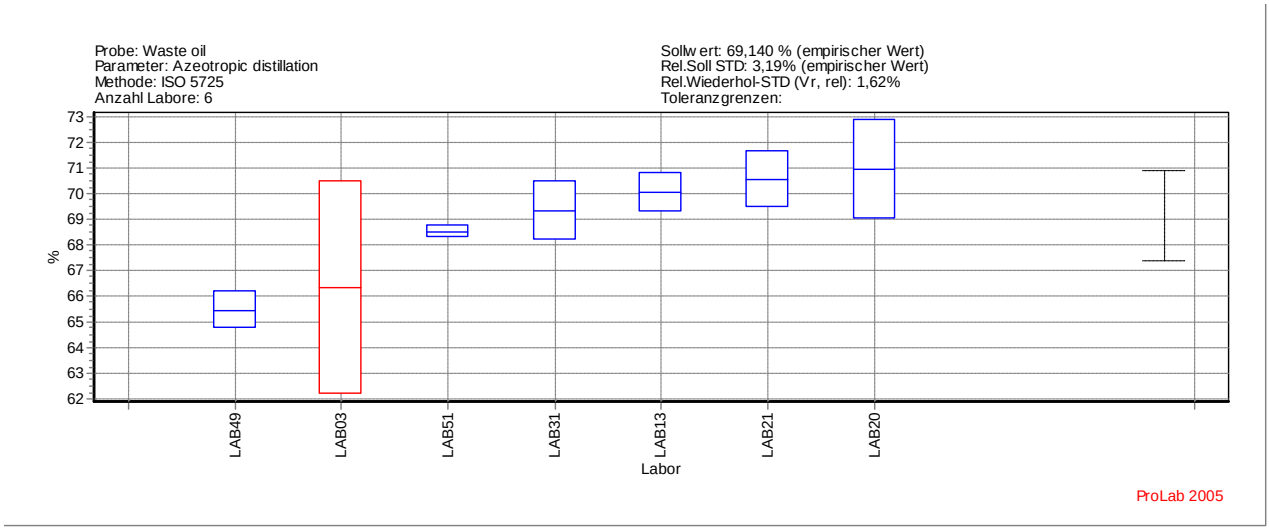
Azeotropic Distillation



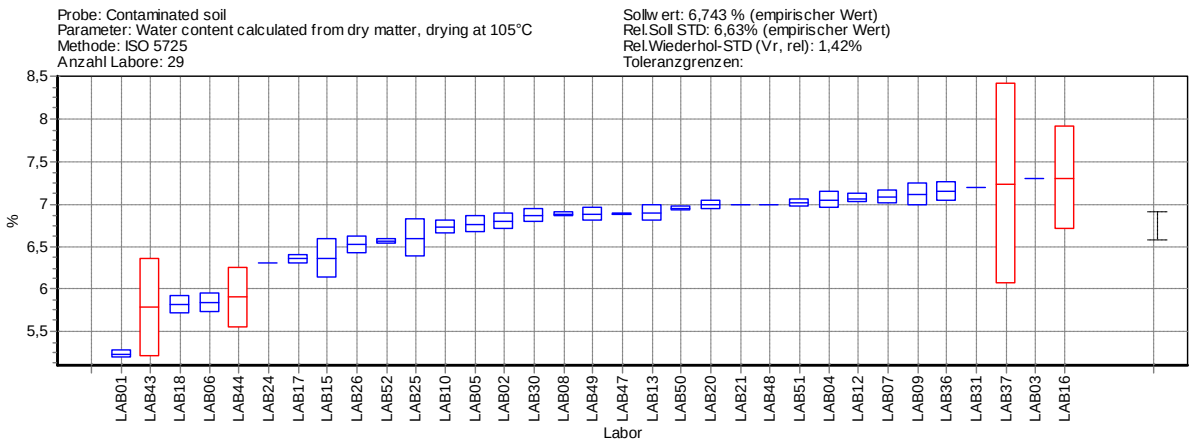


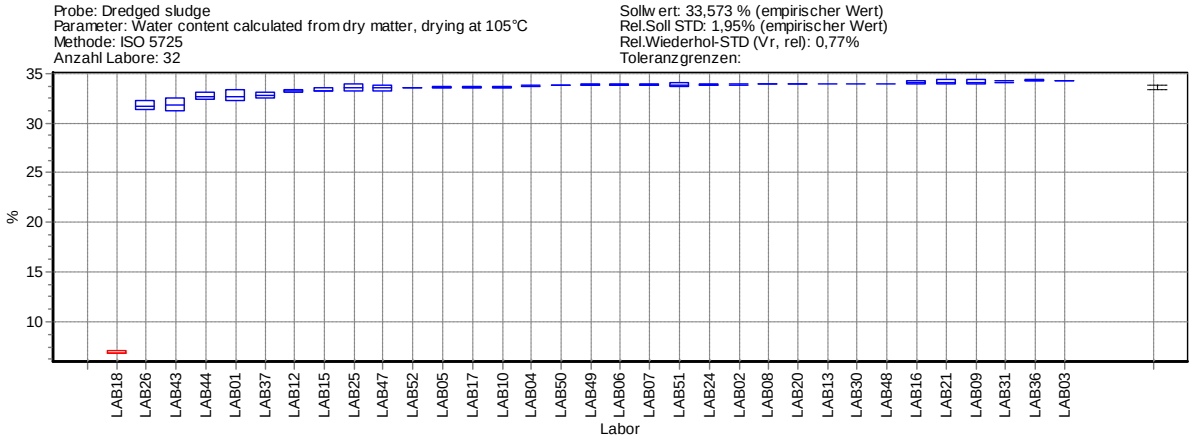


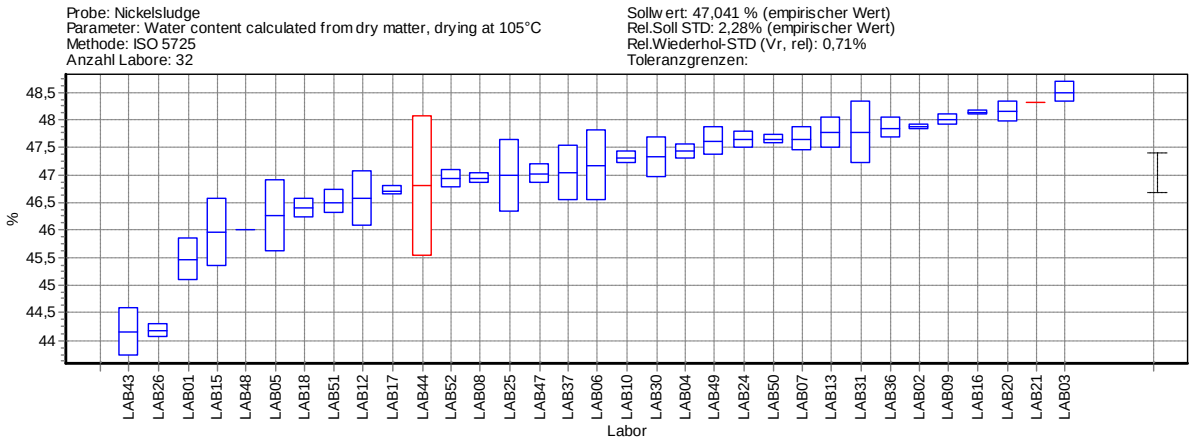

Probe: Nickelsludge
Parameter: Azeotropic distillation
Methode: ISO 5725
Anzahl Labore: 7
Sollwert: 43,208 % (empirischer Wert)
Rel.Soll STD: 6,31% (empirischer Wert)
Rel.Wiederhol-STD (Vr, rel): 3,14%
Toleranzgrenzen:
%
47
46
45
44
43
42
41
40
39
38
37
LAB13
LAB03
LAB49
LAB21
LAB31
LAB51
LAB20
Labor
ProLab 2005


Probe: Waste oil
Parameter: Azeotropic distillation
Methode: ISO 5725
Anzahl Labore: 6
Sollwert: 69,140 % (empirischer Wert)
Rel.Soll STD: 3,19% (empirischer Wert)
Rel.Wiederhol-STD (Vr, rel): 1,62%
Toleranzgrenzen:
%
73
72
71
70
69
68
67
66
65
64
63
62
LAB49
LAB03
LAB51
LAB31
LAB13
LAB21
LAB20
Labor
ProLab 2005

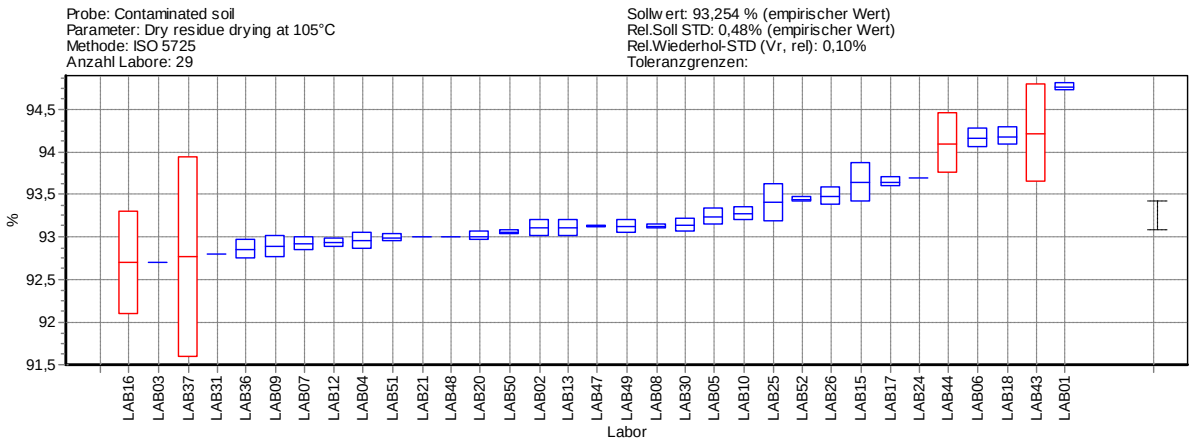
Water content calculated from dry matter (drying at 105 °C)

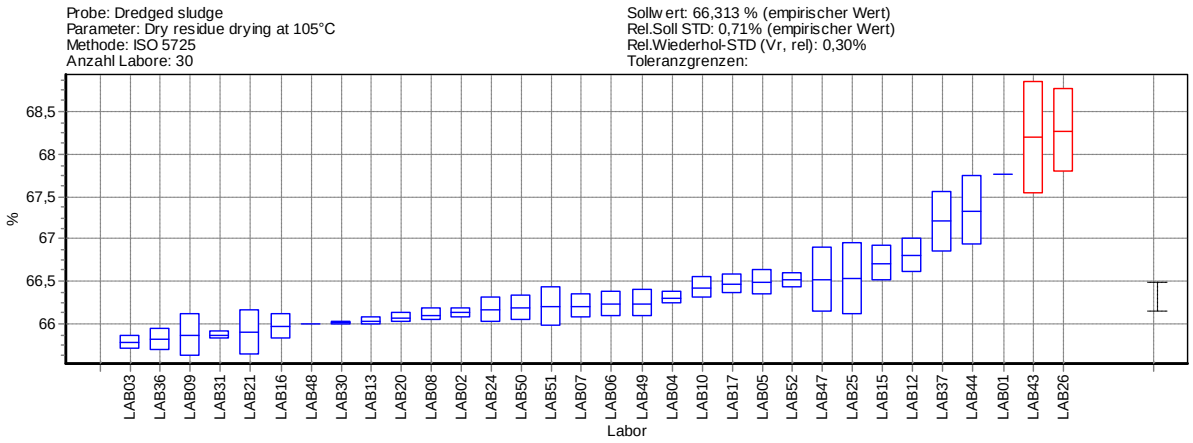


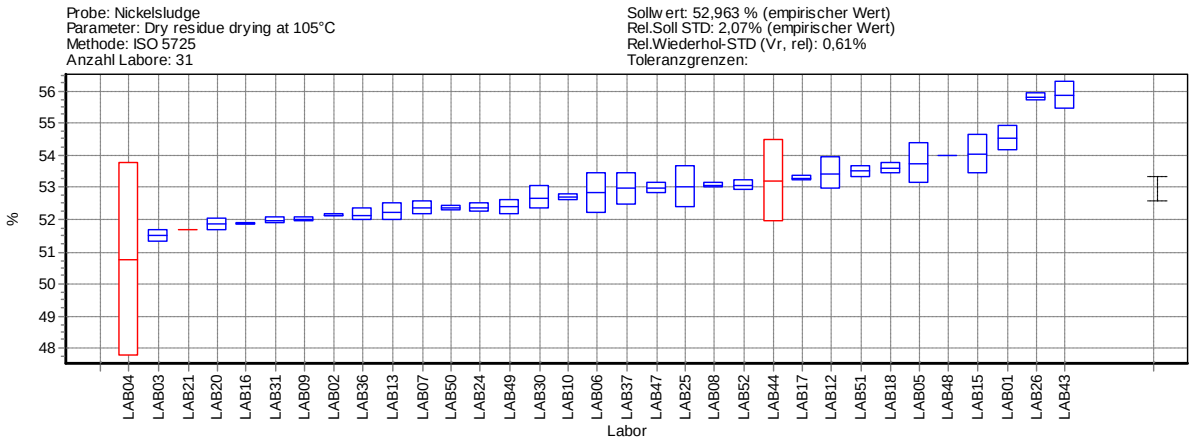




Drying residue an 105 °C







Loss on ignition of dry matter