Soil sample amount in microwave digestion

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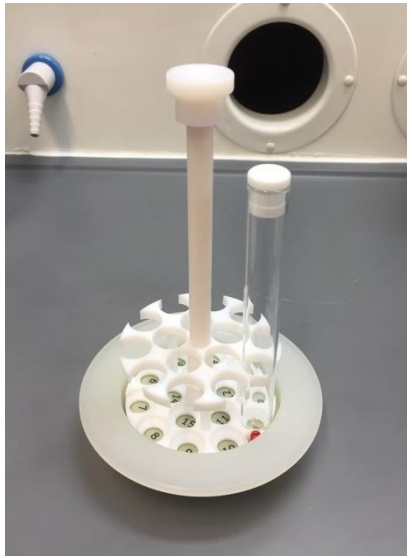
Soil sample amount in microwave digestion

- Finnish government decree 214/2007 on the assessment of soil pollution and soil decontamination
  - Action limits for ca. 50 pollutants, most of them organic
- 11 elements included
  - As, Cd, Co, Cr, Cu, Hg, Ni, Pb, Sb, V, Zn
- Drying not required, but allowed
  - SYKE freeze-dryes samples
- Sieving < 2 mm required
- Homogenisation allowed after sieving (e.g. grinding)
  - SYKE uses Fritsch ball mill, zirconia vessels

Soil sample amount in microwave digestion

- Current digestion method at SYKE (Hg not included)
  - Milestone Ultrawave
  - 15 vessels per rack
  - Disposable 15 ml glass test tubes
  - Target temperature 175°C
  - Dry sample weight 300 mg
  - 6 ml conc. HNO_3 (not Sb)
  - Sb digestion, reverse Aqua Regia
    - (4,5 ml HNO_3 + 1,5 ml HCl)
  - Final volume 15 ml
  - Dilution before ICP-MS measurement
    - (P-E Elan)



Shortcomings of current procedure

Problem	Possible solution
• Two digestions of each sample laborious	• Reverse Aqua Regia for all elements
• 15 ml vessel too small for reactive samples	• 5 vessel rack, 35 ml vessels
• Real life samples often inhomogeneous	• Increased sample amount
• Grinding laborious	• Increased sample amount -> grinding unnecessary?

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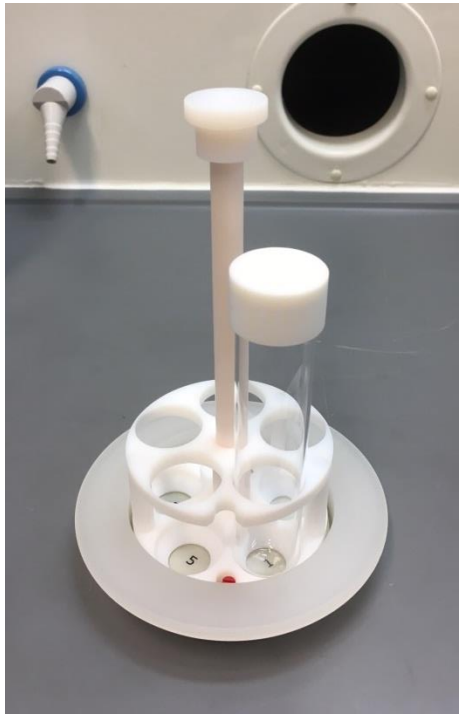
Modifications tested

- 5 samples per rack
- Disposable 35 ml glass test tubes
- Target temperature still 175°C
- Dry sample weight 500 - 1500 mg
- Reverse Aqua Regia
  - 7,5 ml HNO_3 + 2,5 ml HCl
- Final volume 30 ml
- Diluted before ICP-MS measurement
  - Thermo iCAP Q



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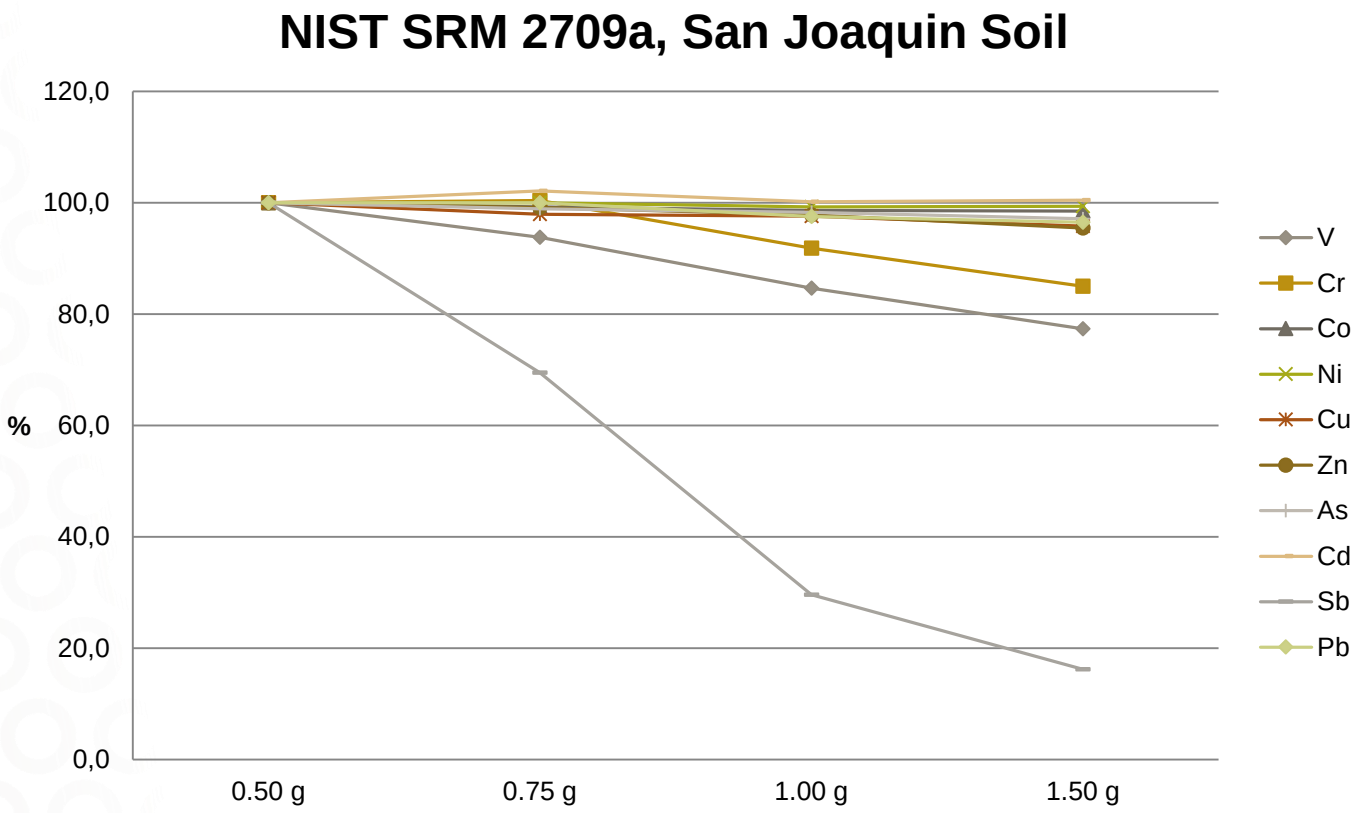
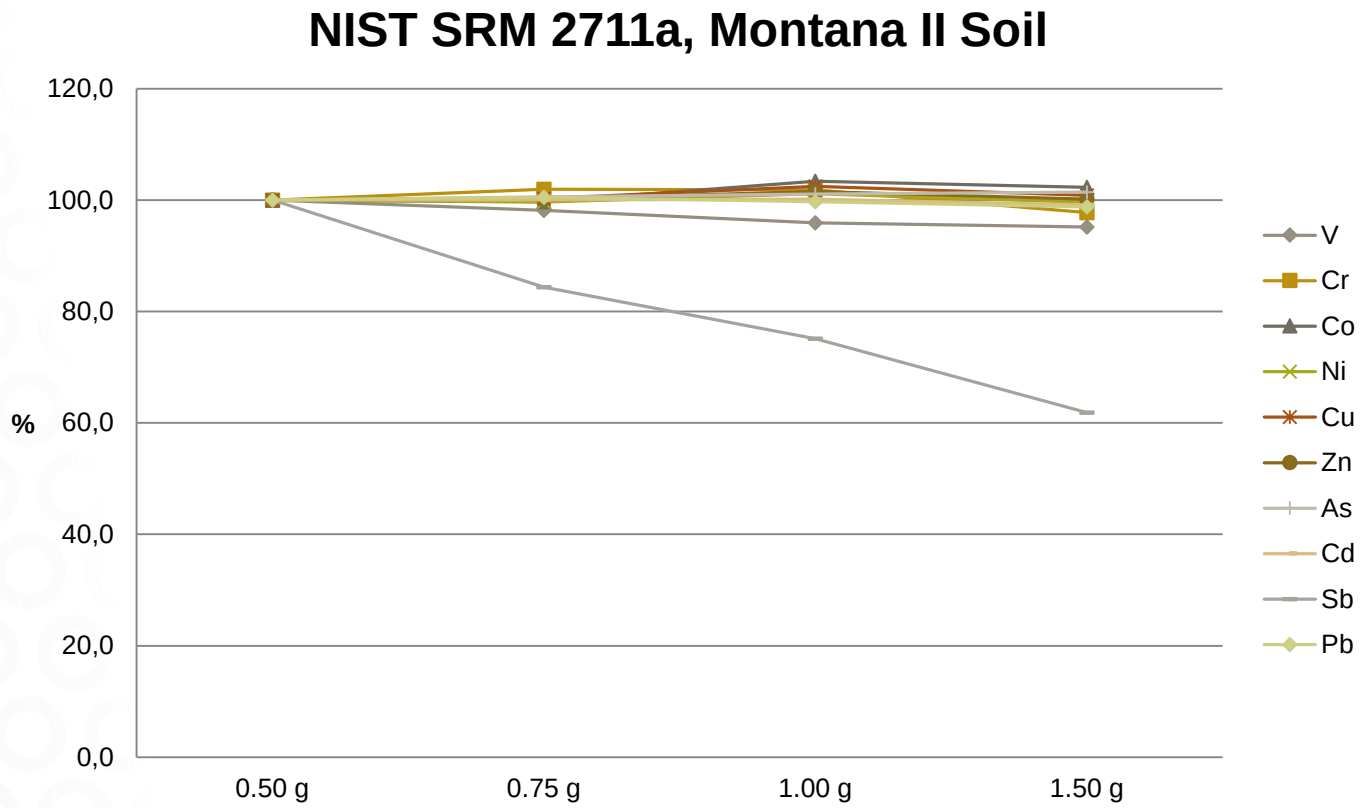
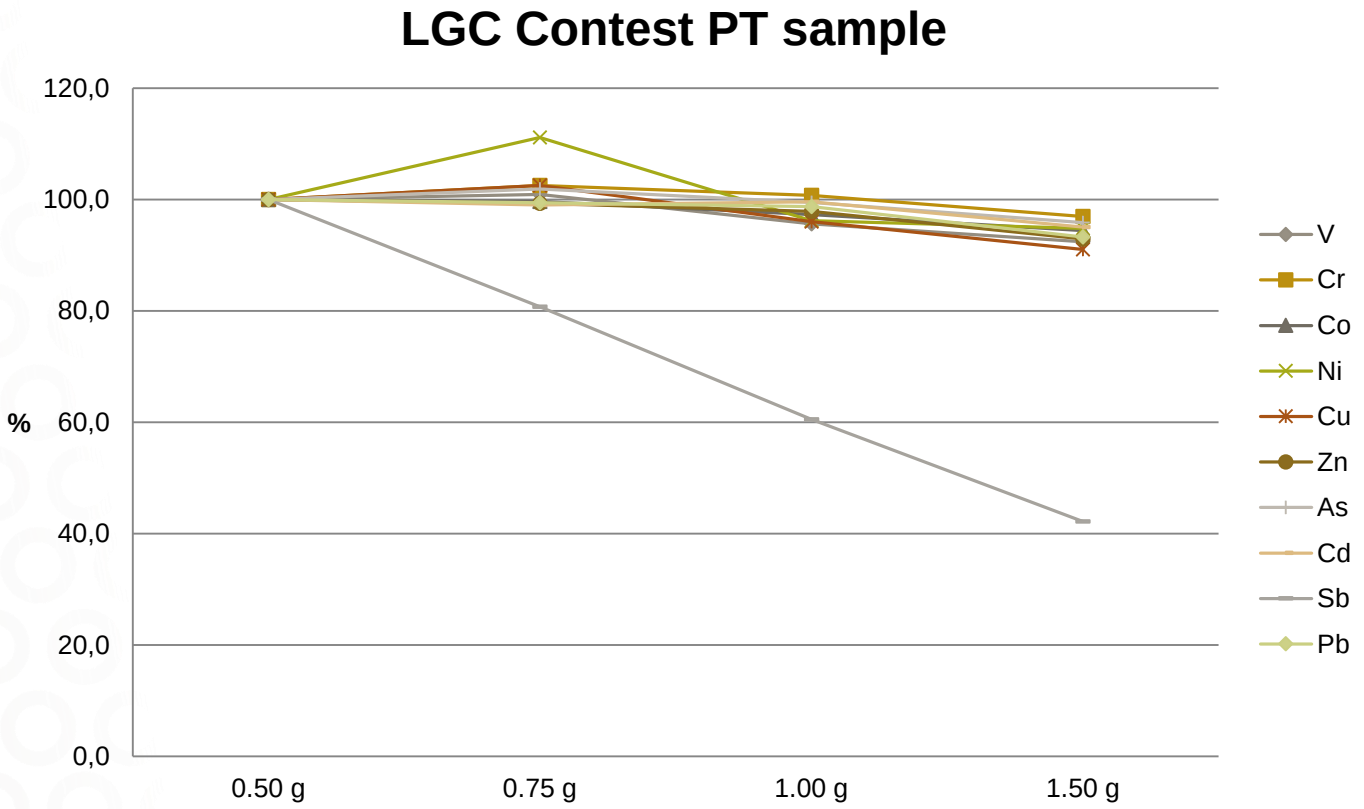
Results


NIST SRM 2709a, San Joaquin Soil
120,0
100,0
80,0
60,0
40,0
20,0
0,0
%
0.50 g
0.75 g
1.00 g
1.50 g
V
Cr
Co
Ni
Cu
Zn
As
Cd
Sb
Pb

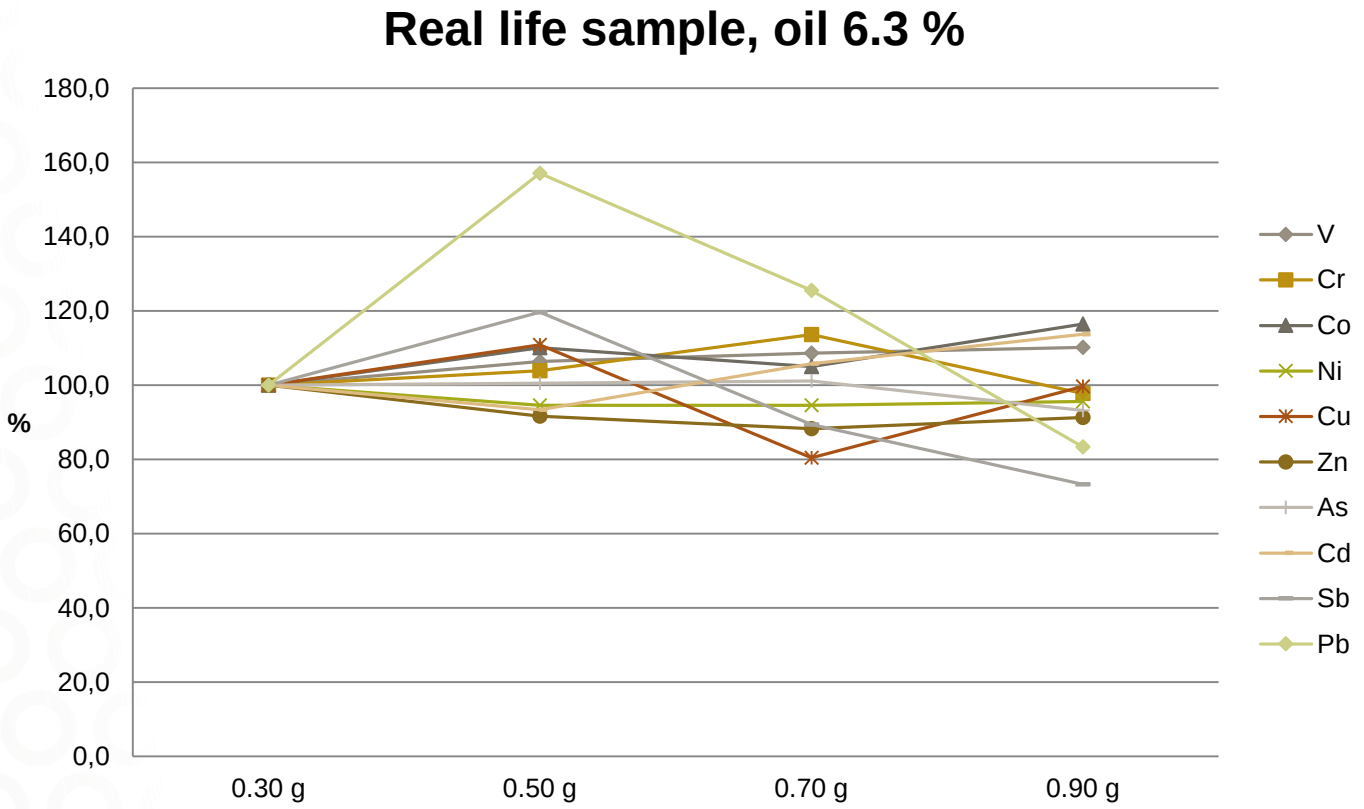
Results


NIST SRM 2711a, Montana II Soil
120,0
100,0
80,0
60,0
40,0
20,0
0,0
%
0.50 g
0.75 g
1.00 g
1.50 g
V
Cr
Co
Ni
Cu
Zn
As
Cd
Sb
Pb

Results


LGC Contest PT sample
120,0
100,0
80,0
%
60,0
40,0
20,0
0,0
0.50 g
0.75 g
1.00 g
1.50 g
V
Cr
Co
Ni
Cu
Zn
As
Cd
Sb
Pb

Results


Real life sample, oil 6.3 %
180,0
160,0
140,0
120,0
100,0
80,0
60,0
40,0
20,0
0,0
%
0.30 g
0.50 g
0.70 g
0.90 g
V
Cr
Co
Ni
Cu
Zn
As
Cd
Sb
Pb

Conclusions

- Increase of sample amount limited due to poor Sb recovery
  - Increase from 300 mg to 500-600 mg possible with larger vessels and higher acid volume
- Some problems with V and Cr as well
- Grinding cannot be left out due to inadequate homogeneity of real life samples
- 5 vessel rack with larger test tubes works very well with reactive samples

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Future method development

- Validation and accreditation of reverse Aqua Regia digestion with large vessels
- Inclusion of Hg
  - Preliminary SRM results show excellent agreement with certified values
- Inclusion of sediments and sludges
- Inclusion of other elements not yet tested
- Digestion programme ramp and hold time optimisation
- Validation and accreditation of Thermo iCAP Q ICP-MS for soil, sediment and sludge analysis

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Thank you!

Results table

Results mg/kg												
Sample List		Sample weight	V	Cr	Co	Ni	Cu	Zn	As	Cd	Sb	Pb
NIST2709a 0.500	SRM	0.50 g	82,74	99,01	12,82	79,72	32,45	101,73	10,49	0,387	0,682	14,60
NIST2709a 0.750	SRM	0.75 g	77,61	99,42	12,72	79,74	31,77	100,86	10,38	0,395	0,474	14,60
NIST2709a 1.000	SRM	1.00 g	70,04	90,94	12,64	79,12	31,67	99,48	10,31	0,387	0,202	14,24
NIST2709a 1.500	SRM	1.50 g	64,03	84,16	12,63	79,20	31,09	97,10	10,19	0,389	0,111	14,09
NIST2710a 0.500	SRM	0.50 g	61,24	36,14	9,46	21,36	143,2	435,9	114,7	58,47	17,28	1520
NIST2710a 0.750	SRM	0.75 g	60,12	36,86	9,43	21,29	143,6	436,7	115,3	58,52	14,58	1528
NIST2710a 1.000	SRM	1.00 g	58,75	36,81	9,78	21,62	146,8	442,8	115,9	58,58	12,99	1516
NIST2710a 1.500	SRM	1.50 g	58,30	35,34	9,68	21,29	144,4	436,9	116,3	57,97	10,70	1502
LGC Aquacheck 0.500	PT sample	0.50 g	25,11	77,22	4,02	19,72	5,97	9,93	1,14	0,014	0,053	5,07
LGC Aquacheck 0.750	PT sample	0.75 g	25,26	77,28	3,99	19,41	5,87	10,04	1,14	0,009	0,041	4,82
LGC Aquacheck 1.000	PT sample	1.00 g	26,41	79,05	4,07	19,83	6,02	9,84	1,13	0,008	0,041	4,75
LGC Aquacheck 1.500	PT sample	1.50 g	24,70	76,04	3,97	19,18	5,73	9,42	0,98	0,008	0,025	4,45
1416-00187-01 0.300	Oily sample	0.30 g	13,03	283,9	25,95	708,6	1647	1344	479,5	2,07	497,9	8559
1416-00187-01 0.500	Oily sample	0.50 g	13,86	295,1	28,57	669,9	1825	1231	482,1	1,93	595,7	13443
1416-00187-01 0.700	Oily sample	0.70 g	14,15	322,6	27,26	670,4	1324	1186	484,7	2,19	445,0	10744
1416-00187-01 0.900	Oily sample	0.90 g	14,35	277,6	30,24	677,6	1641	1227	447,0	2,35	364,7	7139
LGC Contest 0.500	PT sample	0.50 g	74,27	916,5	20,88	38,62	90,64	200,1	76,48	0,633	11,07	313,2
LGC Contest 0.750	PT sample	0.75 g	74,96	939,4	20,81	42,94	92,93	198,7	77,92	0,627	8,94	311,4
LGC Contest 1.000	PT sample	1.00 g	71,01	923,1	20,32	37,17	87,06	195,8	76,08	0,630	6,70	309,3
LGC Contest 1.500	PT sample	1.50 g	68,63	888,6	19,71	36,61	82,49	186,0	73,33	0,602	4,67	292,2

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Results table

Results relative to smallest sample amount (%)												
Sample List		Sample weight	V	Cr	Co	Ni	Cu	Zn	As	Cd	Sb	Pb
NIST2709a 0.500	SRM	0.50 g	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0
NIST2709a 0.750	SRM	0.75 g	93,8	100,4	99,3	100,0	97,9	99,1	98,9	102,1	69,5	100,0
NIST2709a 1.000	SRM	1.00 g	84,6	91,8	98,6	99,3	97,6	97,8	98,3	100,2	29,6	97,5
NIST2709a 1.500	SRM	1.50 g	77,4	85,0	98,5	99,4	95,8	95,5	97,1	100,5	16,2	96,5
NIST2710a 0.500	SRM	0.50 g	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0
NIST2710a 0.750	SRM	0.75 g	98,2	102,0	99,7	99,7	100,3	100,2	100,6	100,1	84,4	100,5
NIST2710a 1.000	SRM	1.00 g	95,9	101,9	103,4	101,2	102,5	101,6	101,1	100,2	75,1	99,7
NIST2710a 1.500	SRM	1.50 g	95,2	97,8	102,3	99,6	100,9	100,2	101,5	99,1	61,9	98,8
LGC Aquacheck 0.500	PT sample	0.50 g	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0
LGC Aquacheck 0.750	PT sample	0.75 g	100,6	100,1	99,4	98,4	98,2	101,2	99,7	62,6	77,7	94,9
LGC Aquacheck 1.000	PT sample	1.00 g	105,2	102,4	101,4	100,6	100,8	99,1	99,4	53,7	77,5	93,7
LGC Aquacheck 1.500	PT sample	1.50 g	98,4	98,5	98,8	97,2	96,0	94,9	85,7	54,5	47,5	87,7
1416-00187-01 0.300	Oily sample	0.30 g	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0
1416-00187-01 0.500	Oily sample	0.50 g	106,4	103,9	110,1	94,5	110,8	91,6	100,5	93,4	119,6	157,1
1416-00187-01 0.700	Oily sample	0.70 g	108,6	113,6	105,0	94,6	80,4	88,2	101,1	105,8	89,4	125,5
1416-00187-01 0.900	Oily sample	0.90 g	110,1	97,8	116,5	95,6	99,7	91,3	93,2	113,7	73,3	83,4
LGC Contest 0.500	PT sample	0.50 g	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0	100,0
LGC Contest 0.750	PT sample	0.75 g	100,9	102,5	99,7	111,2	102,5	99,3	101,9	99,0	80,7	99,4
LGC Contest 1.000	PT sample	1.00 g	95,6	100,7	97,3	96,2	96,0	97,9	99,5	99,6	60,5	98,8
LGC Contest 1.500	PT sample	1.50 g	92,4	97,0	94,4	94,8	91,0	93,0	95,9	95,1	42,2	93,3

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