

University of Stuttgart
Germany



Analytische Qualitätssicherung Baden-Württemberg

Proficiency Test 8/20

- TW S10 – epichlorohydrin, acrylamide in
drinking water -

Final report

provided by
AQS Baden-Württemberg at
Institute for Sanitary Engineering, Water Quality and Solid Waste Management,
University of Stuttgart
Bandtäle 2, 70569 Stuttgart-Büsnau, Germany



Stuttgart, in January 2021

**AQS Baden-Württemberg at
Institute of Sanitary Engineering,
Water Quality and Solid Waste Management
at University of Stuttgart
Bandtäle 2
70569 Stuttgart-Büsnau
Germany
<http://www.aqsbw.de>
Tel.: +49 (0)711 / 685-65446
Fax: +49 (0)711 / 685-53769
E-Mail: info@aqsbw.de**

Responsibilities:

Scientific director:	Dr.-Ing. Michael Koch	
PT coordinator:	Dr.-Ing. Frank Baumeister	
Assistant PT coordinator	Dipl.-Biol. Biljana Marić	
Sample preparation	Gertrud Joas Cornelia Orth	
Release of the report:	Dr.-Ing. Michael Koch	26 January 2021
Version of the report	1	

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1. General

This PT was provided in the context of the AQS Baden-Württemberg drinking water PT scheme. In this round following parameter were to be determined.

- epichlorohydrin
- acrylamide

The PT was executed according to the recommendations of the German Federal Environment Agency from December 2003. These recommendations “for the execution of PTs for the measurement of chemical parameter and indicator parameter for the external quality control of drinking water laboratories” (Bundesgesundheitsblatt 46 12, 1094-1095) require, that drinking water laboratories must demonstrate their competence for all parameters they are accredited for or they want to be accredited for by a successful participation in a PT round within a cycle of 2-3 years.

The PT was executed and evaluated according to the requirements of DIN 38402-A45 and ISO/TS 20612.

2. PT design

Each participant received the following samples:

- 3 samples for the determination of epichlorohydrin in 1000-ml-glass bottles with screw cap. Preservation by cooling.
- 3 samples for the determination of acrylamide in 500-ml-glas bottles with screw cap. Preservation by cooling.

3 different concentration levels/batches were produced. All participants received the same samples.

3. Sample preparation

The samples for the determination of the above mentioned parameters were based on a real drinking water matrix.

The drinking water was filtered by using 5 µm and 1 µm filter cartridges to eliminate particles. To reduce germs, the drinking water was irradiated with ultraviolet light and pasteurised at 80°C in a stainless steel vessel overnight. During pasteurisation, the drinking water was aerated with a mixture composed of carbon dioxide and nitrogen to prevent calcium carbonate precipitation.

The drinking water was spiked with stock solutions and the concentrations covered drinking and ground water relevant ranges.

4. Sample distribution

The samples were dispatched on 23 November 2020 by express service (GoExpress).

5. Analytical methods

The participants were free to choose a suitable method, but a limit of quantification of 0,04 µg/l for epichlorohydrin and 0,05 µg/l for acrylamide was required.

The participants were informed that the samples had to be analysed in the own laboratory, with own personal and own equipment. Subcontracting of the analysis was not allowed.

The samples had to be analysed in duplicate over the complete method (sample preparation and measurement). The participants were asked to report the results in µg/l with three significant digits.

6. Submission of the results

The deadline for the submission of results was on 18 December 2020.

7. Basic principle of evaluation and assessment

The basic principle of the evaluation and assessment of the PTs from AQS Baden-Württemberg are described in the document „Evaluation of the PTs and information for the report“, which can be downloaded from www.aqsbw.de/pdf/ausw_berichte_v1_en.pdf.

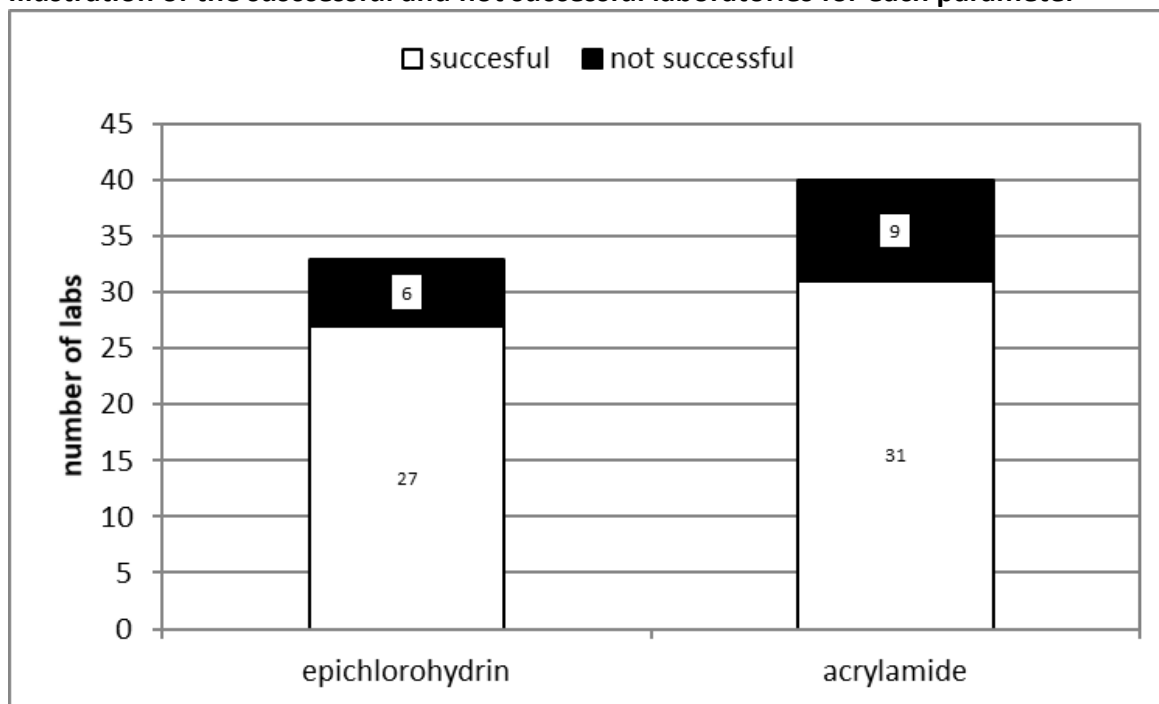
This PT was evaluated as follows:

Assigned value x_{pt}:	Consensus value (Hampel estimator)						
Standard deviation for proficiency assessment σ_{pt}:	Q method						
Upper limit of σ_{pt}:	25 %						
Lower limit of σ_{pt}:	5 %						
Assessment:	z_U -Score						
Classification of the single results:	<table style="border: none; width: 100%;"> <tr> <td style="border: none;">$z_u \leq 2,0$</td> <td style="border: none;">successful</td> </tr> <tr> <td style="border: none;">$2,0 < z_u < 3,0$</td> <td style="border: none;">questionable</td> </tr> <tr> <td style="border: none;">$z_u \geq 3,0$</td> <td style="border: none;">unsatisfactory</td> </tr> </table>	$ z_u \leq 2,0$	successful	$2,0 < z_u < 3,0$	questionable	$ z_u \geq 3,0$	unsatisfactory
$ z_u \leq 2,0$	successful						
$2,0 < z_u < 3,0$	questionable						
$ z_u \geq 3,0$	unsatisfactory						
Parameter assessment:	A parameter was assessed as successful, if more than half of the values were correctly determined (2 out of 3 values are within the tolerance limits).						

8. Evaluation

Number of participants:	47 2 laboratories did not report results
Number of reported values	218
Number of accepted values:	179 (82,1 %)

Illustration of the successful and not successful laboratories for each parameter



9. Explanation for the appendices

The explanations for the appendices can be found in the document „Evaluation of the PTs and information for the report“, which can be downloaded from www.agsbw.de/pdf/ausw_berichte_v1_en.pdf.

10. Measurement uncertainty

General:

Number of labs with valid values	45
Number of labs with valid values and reported measurement uncertainties	29 (64,4 %)
Number of valid values	218
Number of valid values with measurement uncertainties	137 (62,8 %)

Measurement uncertainties against the accreditation status

Accreditation status of the values	Number of values	Number of values with measurement uncertainty
accredited	167	116 (69,5 %)
not accredited	24	9 (37,5 %)
not specified	27	12 (44,4 %)

Interpretation of the reported measurement uncertainties:

If measurement uncertainties are underestimated values assessed as “satisfactory” in the PT ($|z_U| \leq 2$), will have a large ζ -score. $|\zeta| > 2$ means that the “own” requirements (defined in terms of estimated uncertainty) are not fulfilled.

Number of values with reported measurement uncertainty having a $z_U \leq 2,0$	114
Number of values with a magnitude of ζ-scores > 2 The own requirements of the laboratory are not fulfilled and the estimation of the measurement uncertainty is too low	24 (21,1 %)

11. Traceable reference values

The explanations about traceable reference values can be found in the document „Evaluation of the PTs and information for the report“, which can be downloaded from www.agsbw.de/pdf/ausw_berichte_v1_en.pdf

12. Internet

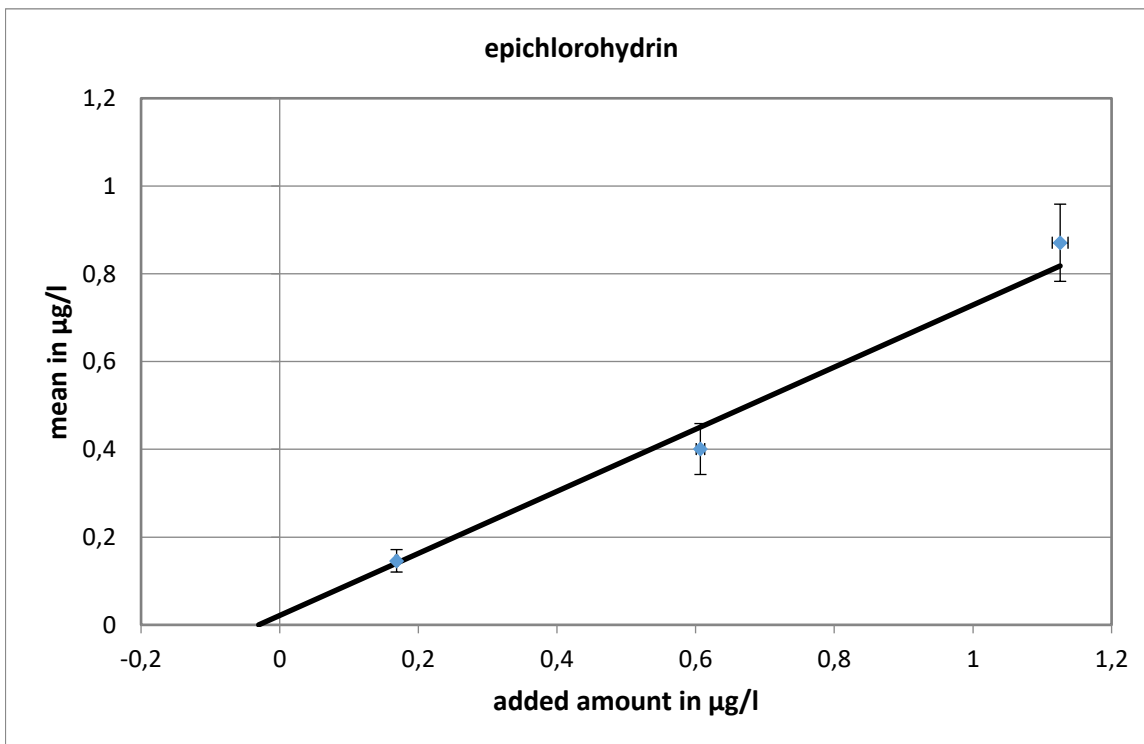
The report is available on the following webpage:

http://www.agsbw/pdf/224/report_234.pdf

epichlorohydrin

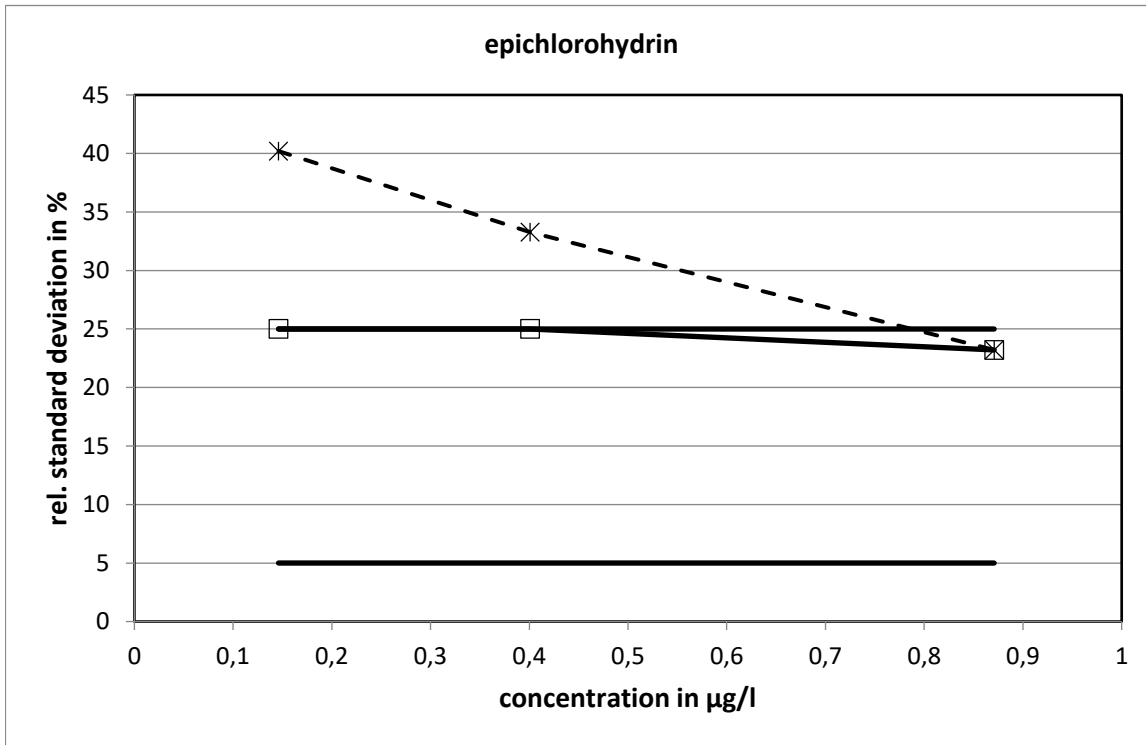
level	assigned value [µg/l]	expanded uncertainty of the assigned value [%]	standard deviation, calculated using robust statistics [µg/l]	standard deviation for proficiency assessment [µg/l]	standard deviation for proficiency assessment [%]	upper tolerance limit [µg/l]	lower tolerance limit [µg/l]	upper tolerance limit [%]	lower tolerance limit [%]	number of results	out below	out above	out [%]
1	0,1460	17,50	0,0587	0,0365	25,00	0,2307	0,0800	57,99	-45,19	33	2	4	18,2
2	0,4007	14,48	0,1334	0,1002	25,00	0,6331	0,2196	57,99	-45,19	33	5	2	21,2
3	0,8708	10,10	0,2021	0,2021	23,21	1,334	0,5033	53,19	-42,20	33	5	0	15,2
sum										99	12	6	18,2

Recovery and matrix content

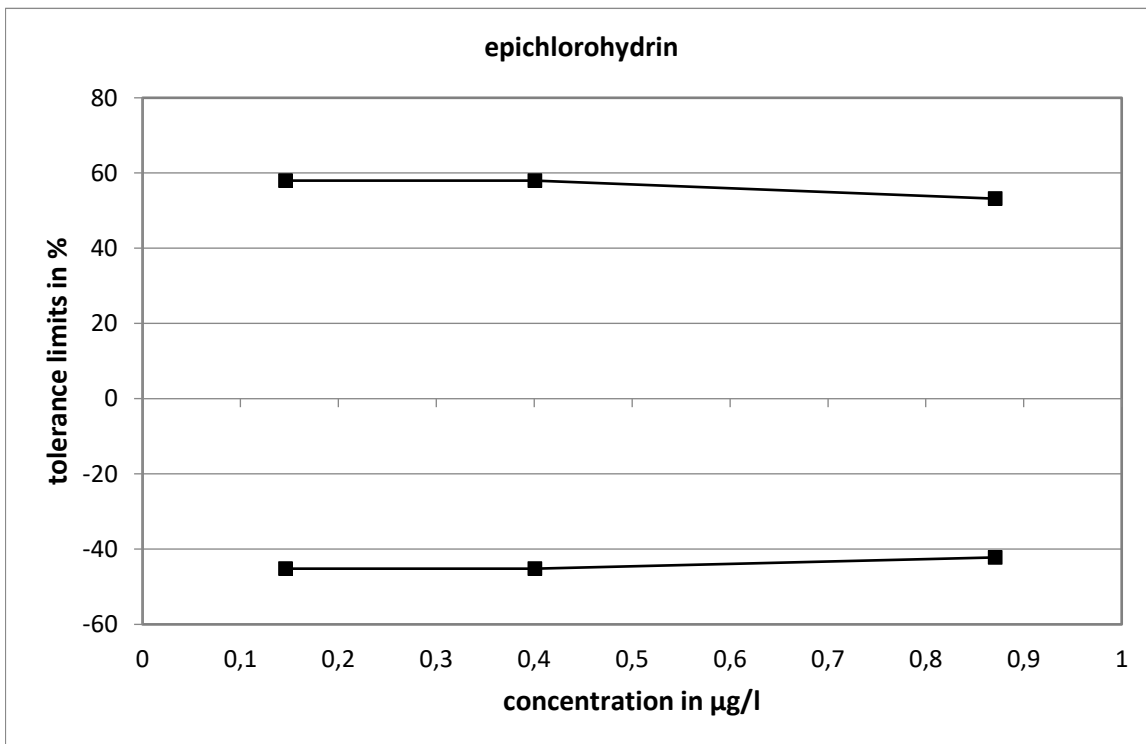


slope of the regression: 0,708; recovery rate: 70,8 %
 neg. x-axis intercept = matrix content: 0,0303 µg/l
 expanded uncertainty of the matrix content: 0,0303 µg/l = 100 %

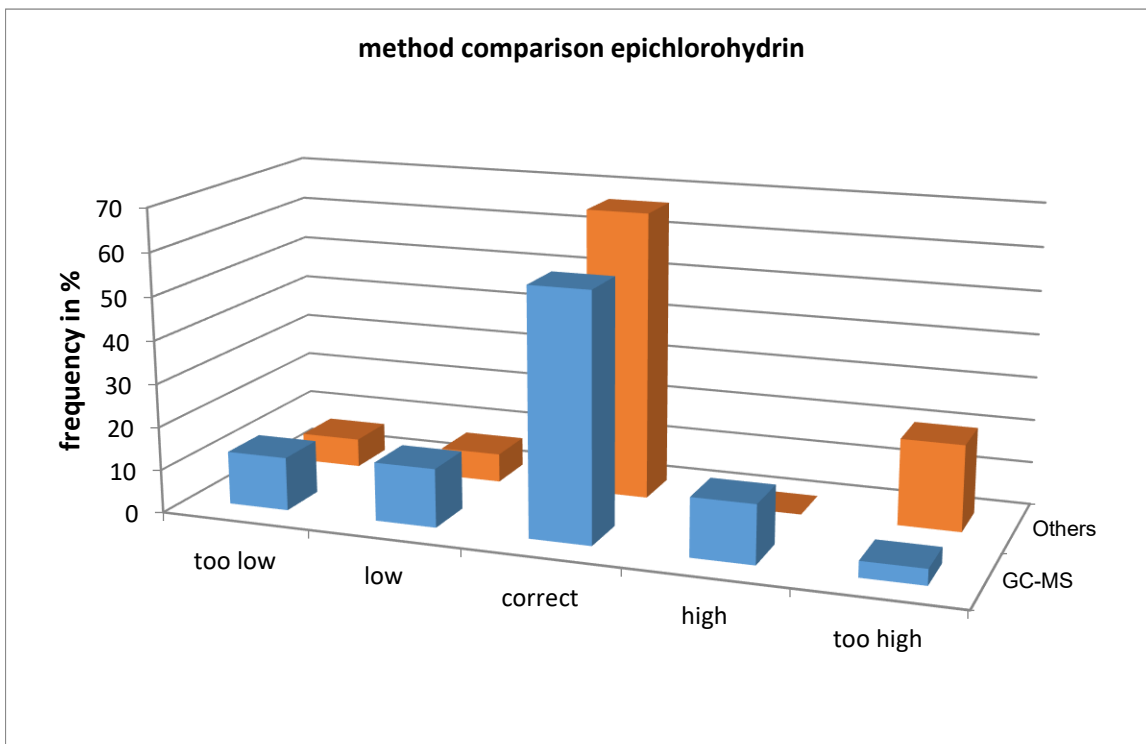
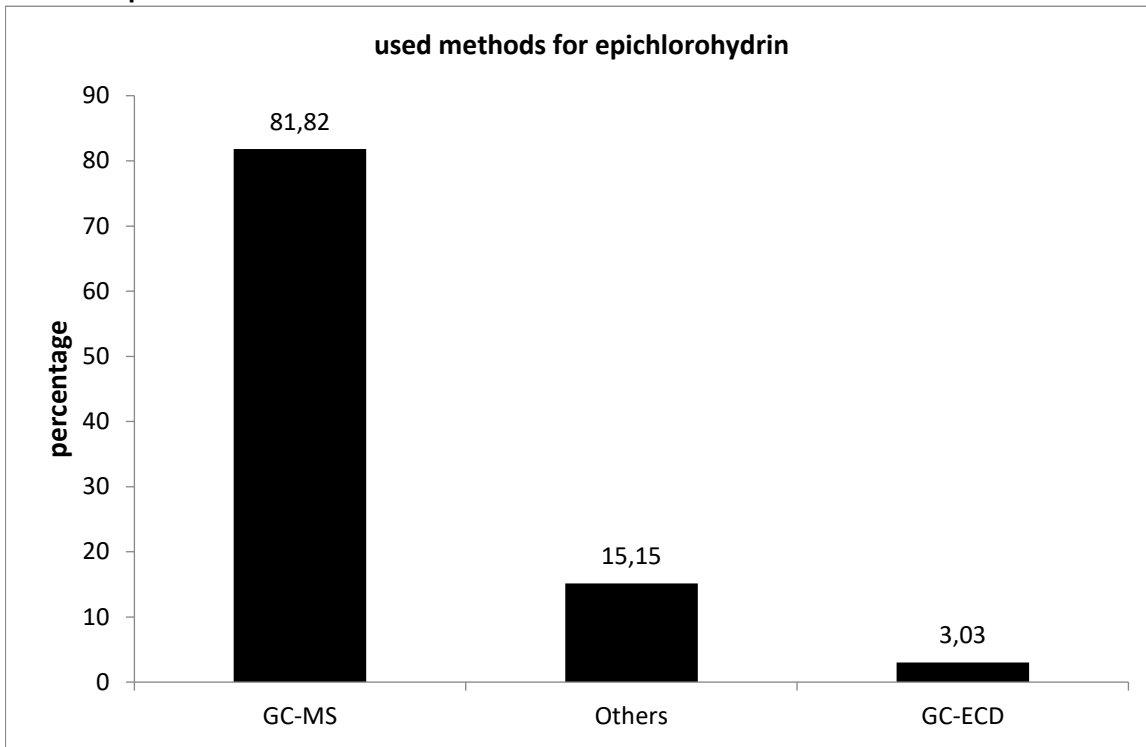
Relative standard deviation and tolerance limits



The relative standard deviations calculated with the Q-method reached the upper limit with two concentration levels.



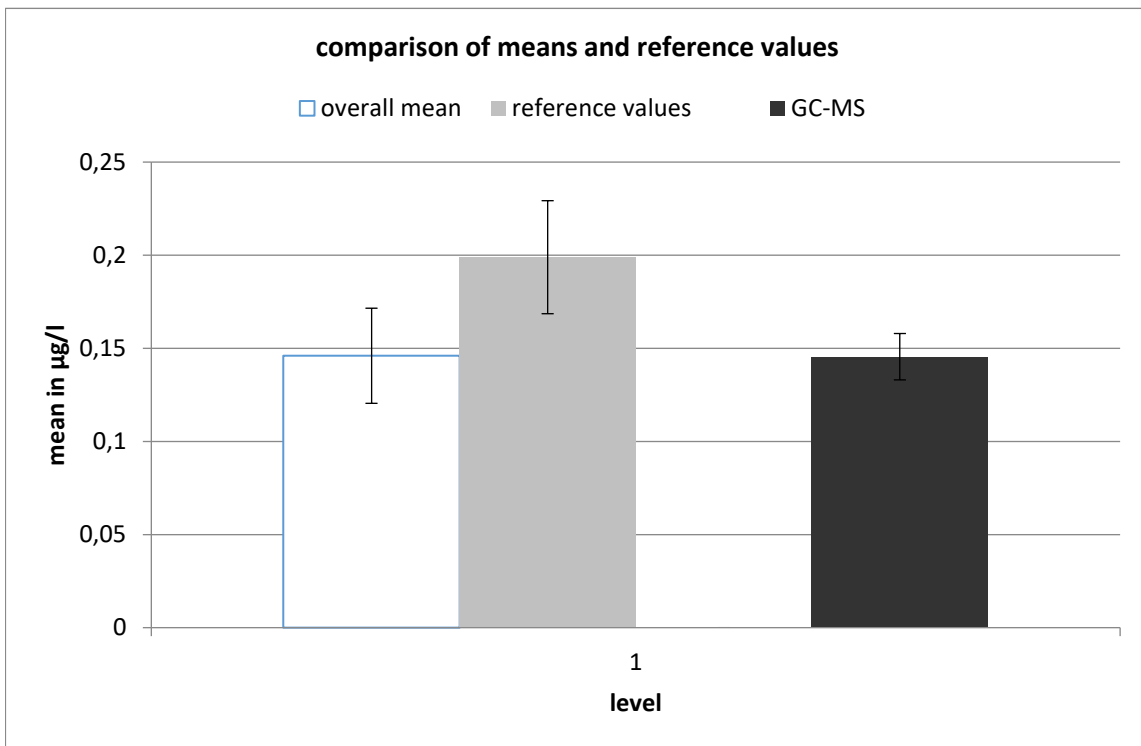
Method specific evaluation

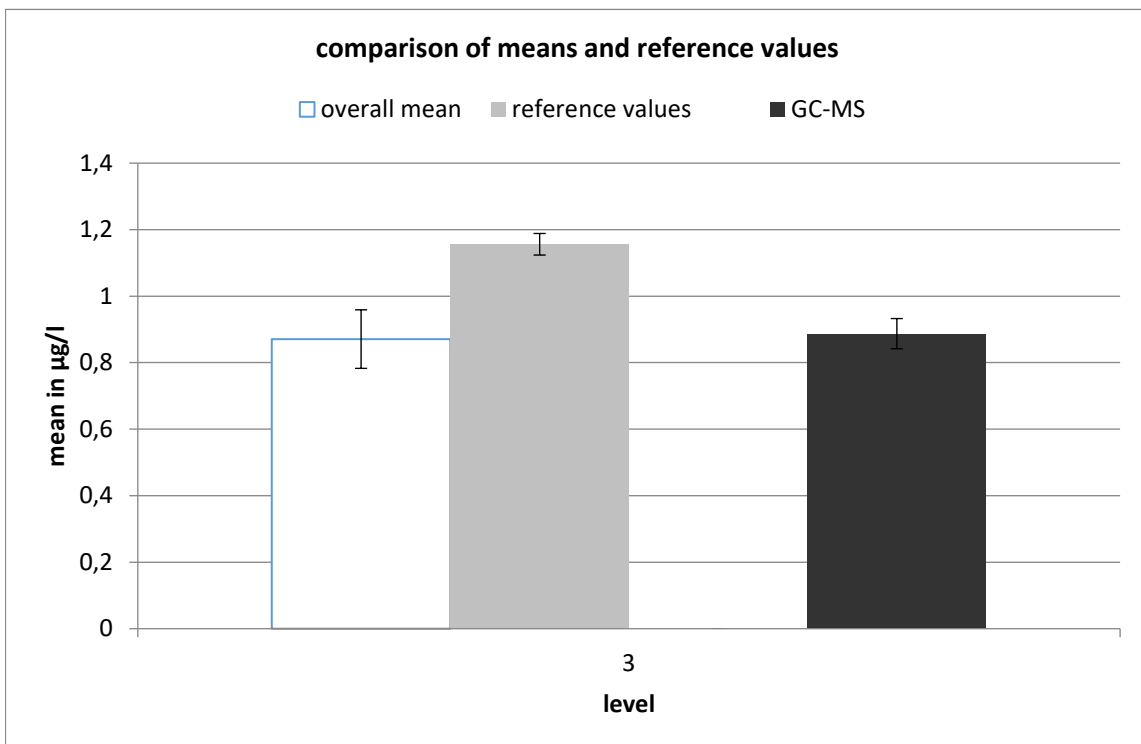
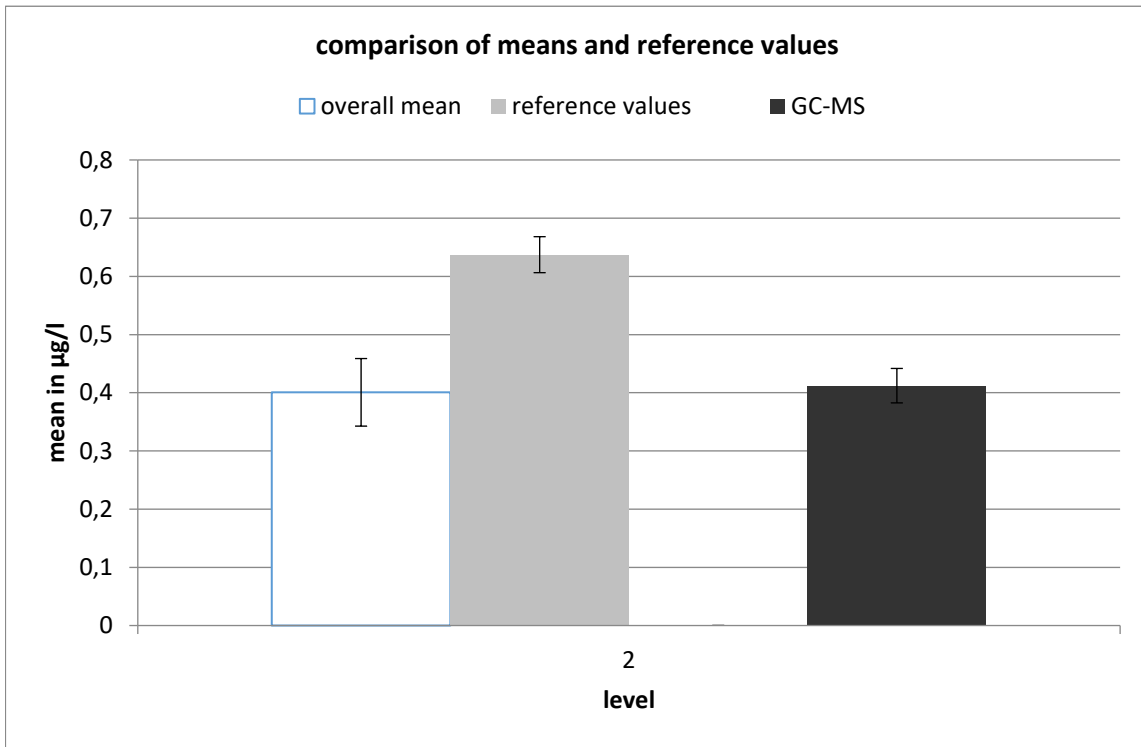


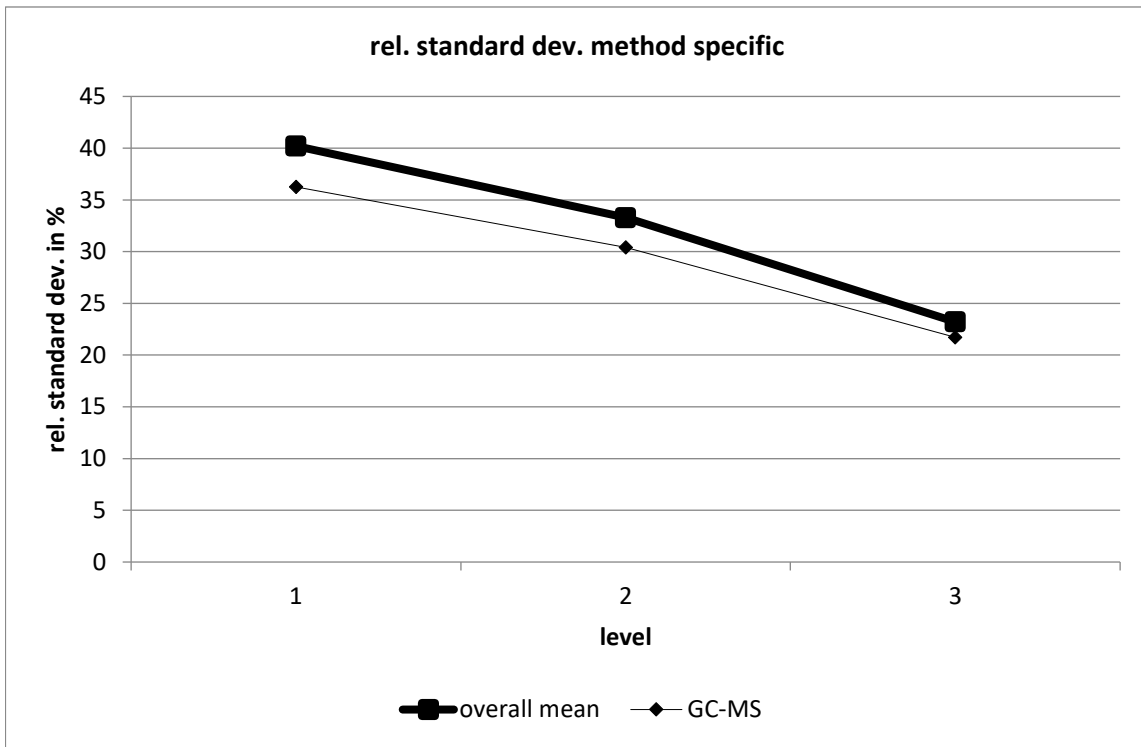
The differences between the methods were not significant.

Comparison of means and reference values

level	mean [µg/l]	exp. uncertainty [µg/l]	exp. uncertainty [%]	reference value [µg/l]	exp. uncertainty [µg/l]	exp. uncertainty [%]
1	0,1460	0,0255	17,5	0,1989	0,0303	15,3
2	0,4007	0,0580	14,5	0,6372	0,0309	4,9
3	0,8708	0,0880	10,1	1,1560	0,0324	2,8





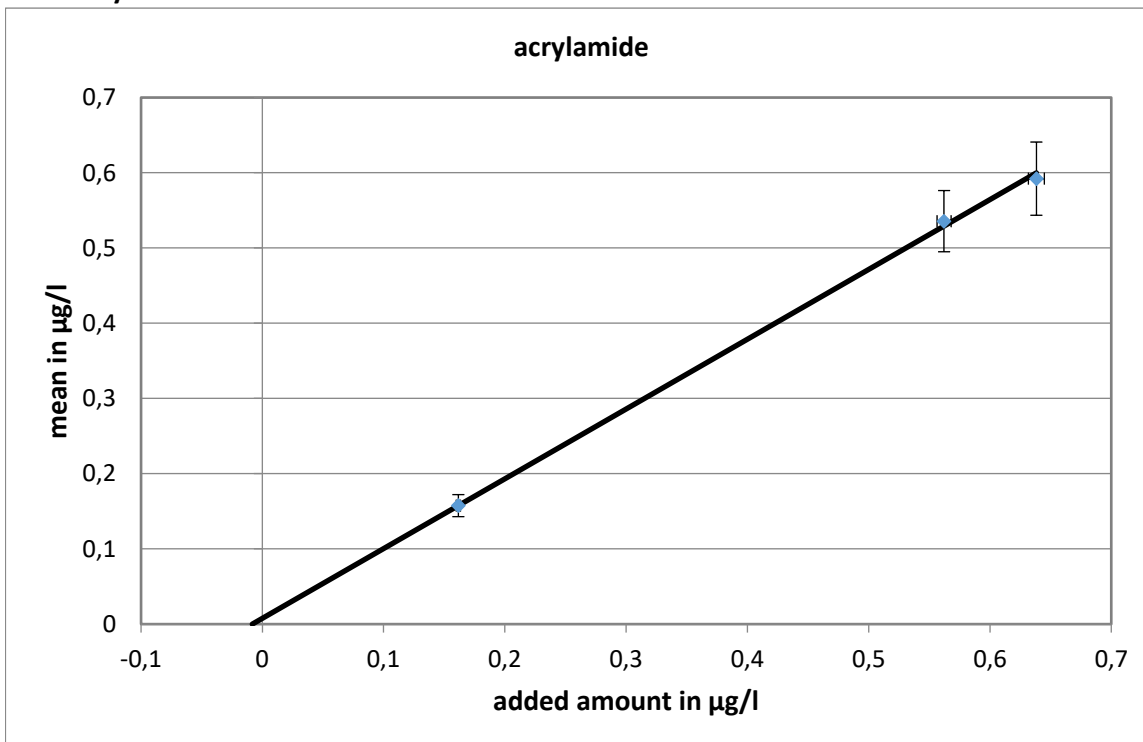


GC-MS									
level	robust mean [$\mu\text{g/l}$]	exp. unc. of the mean [$\mu\text{g/l}$]	exp. unc. of the mean [%]	robust standard deviation [$\mu\text{g/l}$]	robust standard deviation [%]	number of results	out below	out above	out [%]
1	0,145	0,012	8,564	0,053	36,25	28	1	2	10,71
2	0,412	0,03	7,187	0,125	30,42	28	3	1	14,29
3	0,887	0,046	5,131	0,193	21,72	28	6	0	21,43

acrylamide

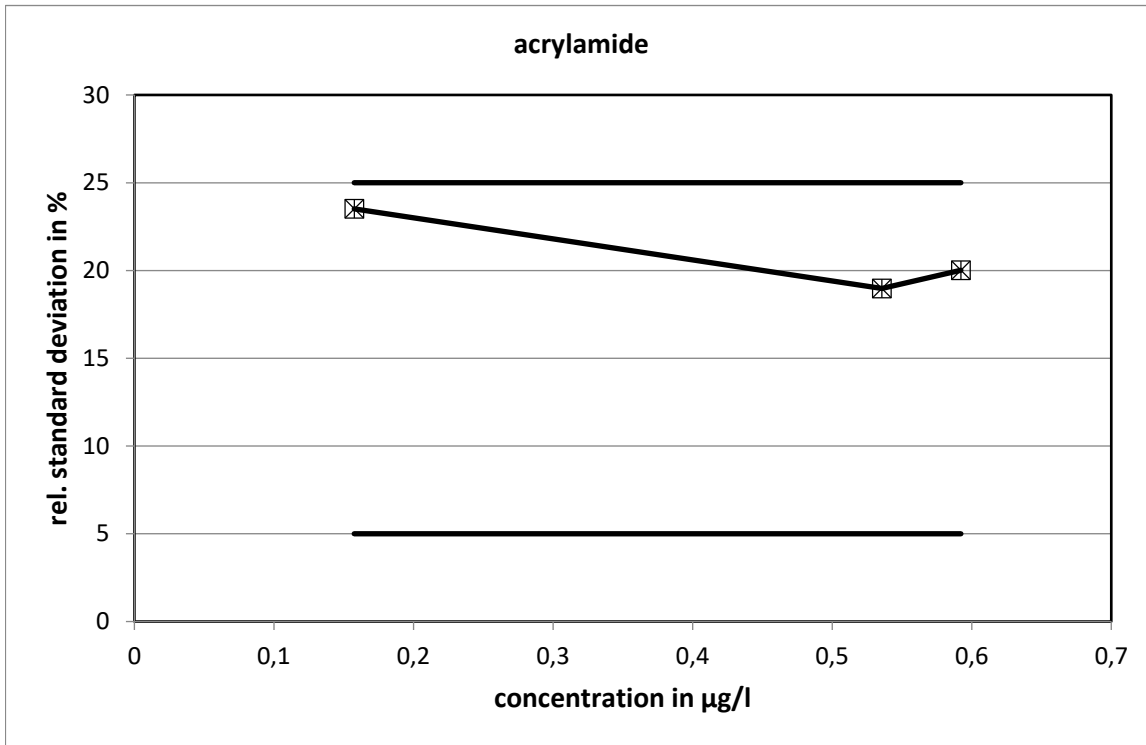
level	assigned value [µg/l]	expanded uncertainty of the assigned value [%]	standard deviation, calculated using robust statistics [µg/l]	standard deviation for proficiency assessment [µg/l]	standard deviation for proficiency assessment [%]	upper tolerance limit [µg/l]	lower tolerance limit [µg/l]	upper tolerance limit [%]	lower tolerance limit [%]	number of results	out below	out above	out [%]
1	0,1575	9,29	0,0370	0,0370	23,51	0,2426	0,0903	53,98	-42,70	40	3	2	12,5
2	0,5357	7,60	0,1017	0,1017	18,98	0,7622	0,3482	42,29	-35,00	39	6	1	17,9
3	0,5922	8,22	0,1185	0,1185	20,01	0,8580	0,3745	44,88	-36,77	37	8	1	22,5
sum										116	17	4	18,1

Recovery and matrix content

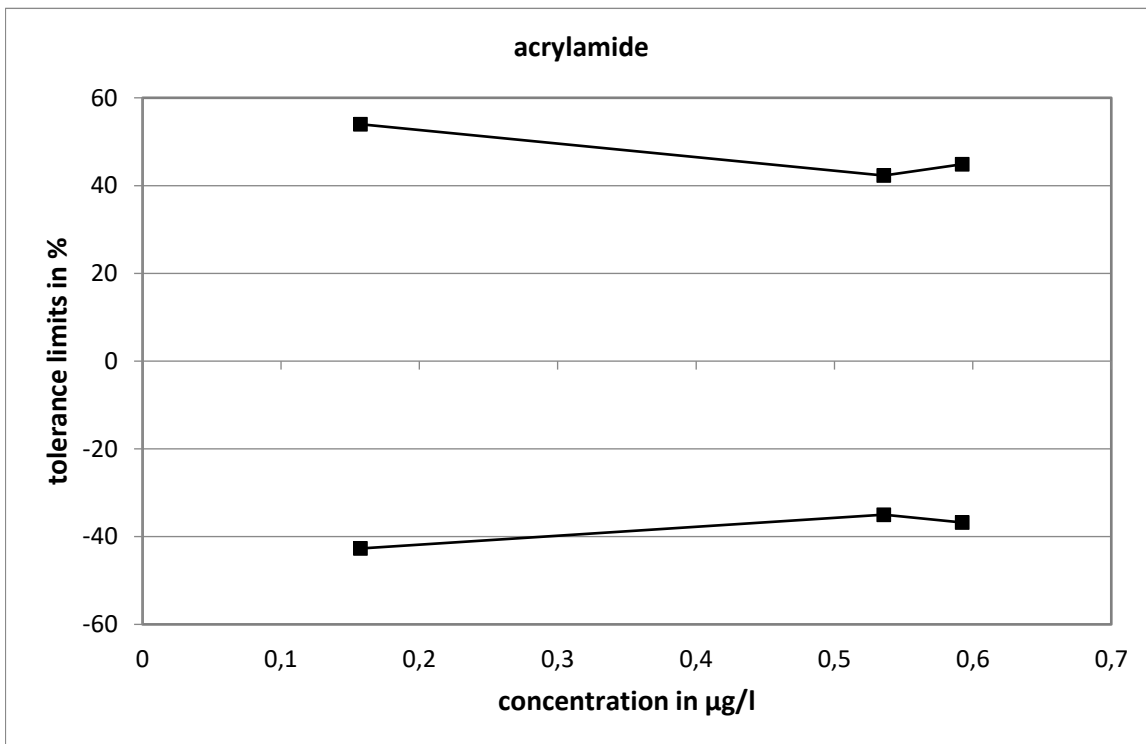


slope of the regression: 0,928; recovery rate: 92,8 %
 neg. x-axis intercept = matrix content: 0,0083 µg/l
 expanded uncertainty of the matrix content: 0,0083 µg/l = 100 %

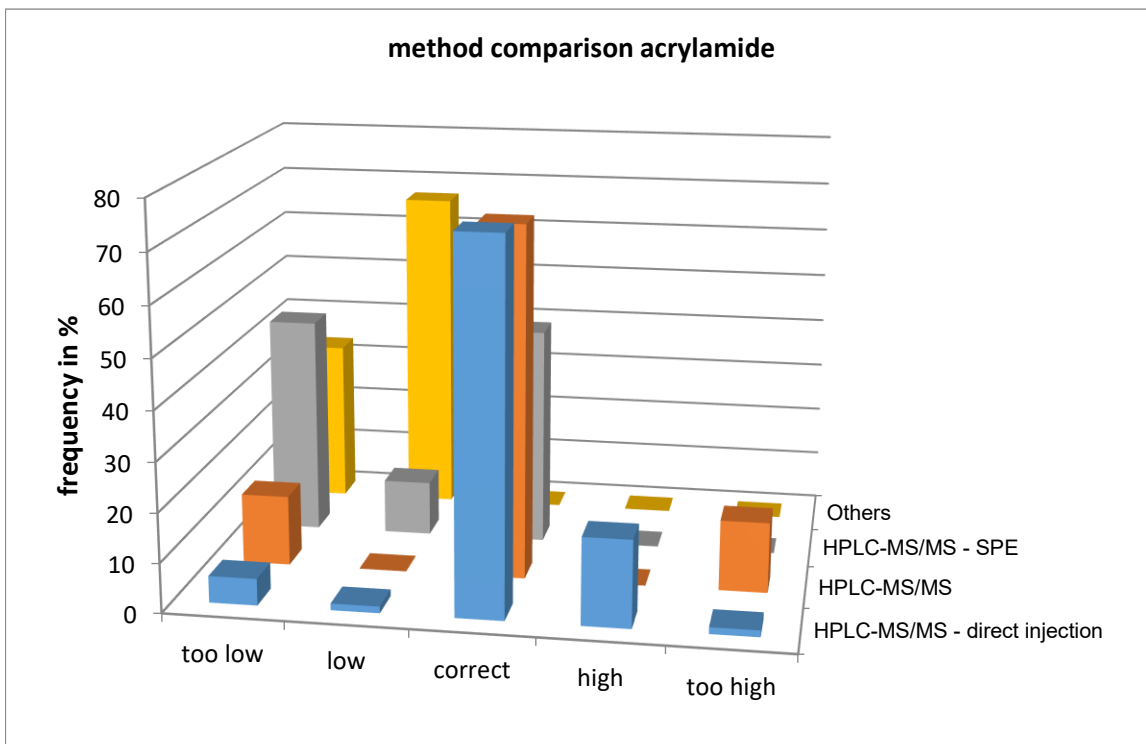
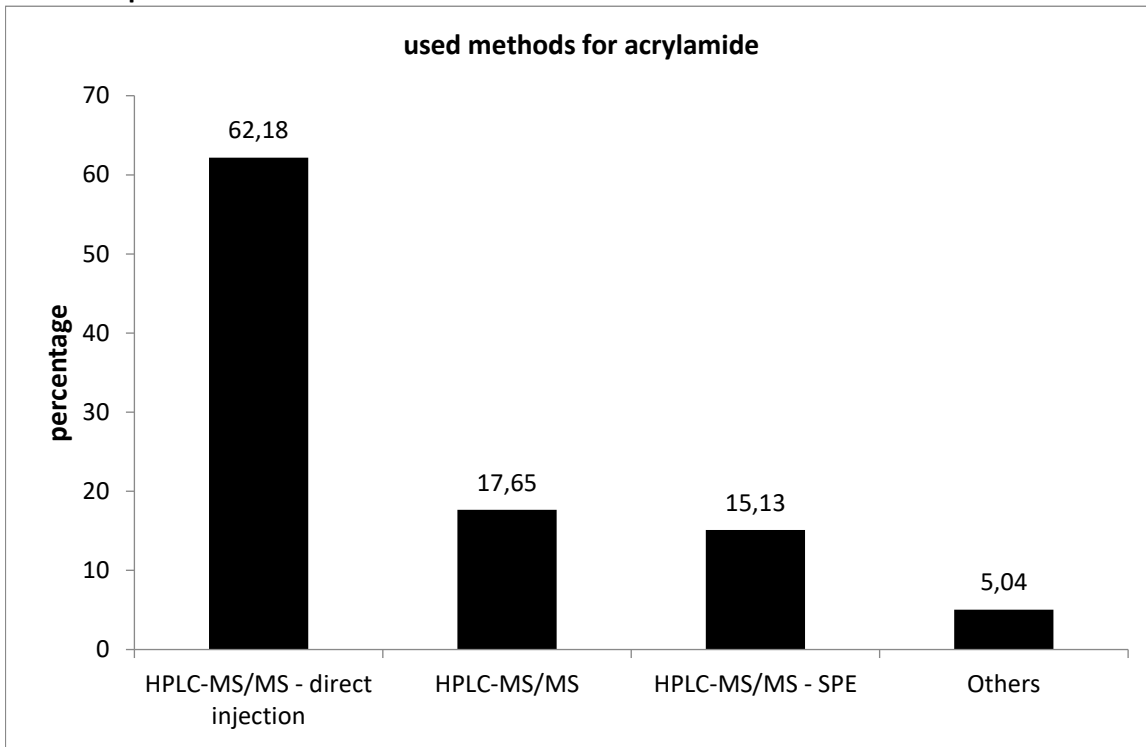
Relative standard deviation and tolerance limits



The relative standard deviations calculated with the Q-method did not reach the limits.



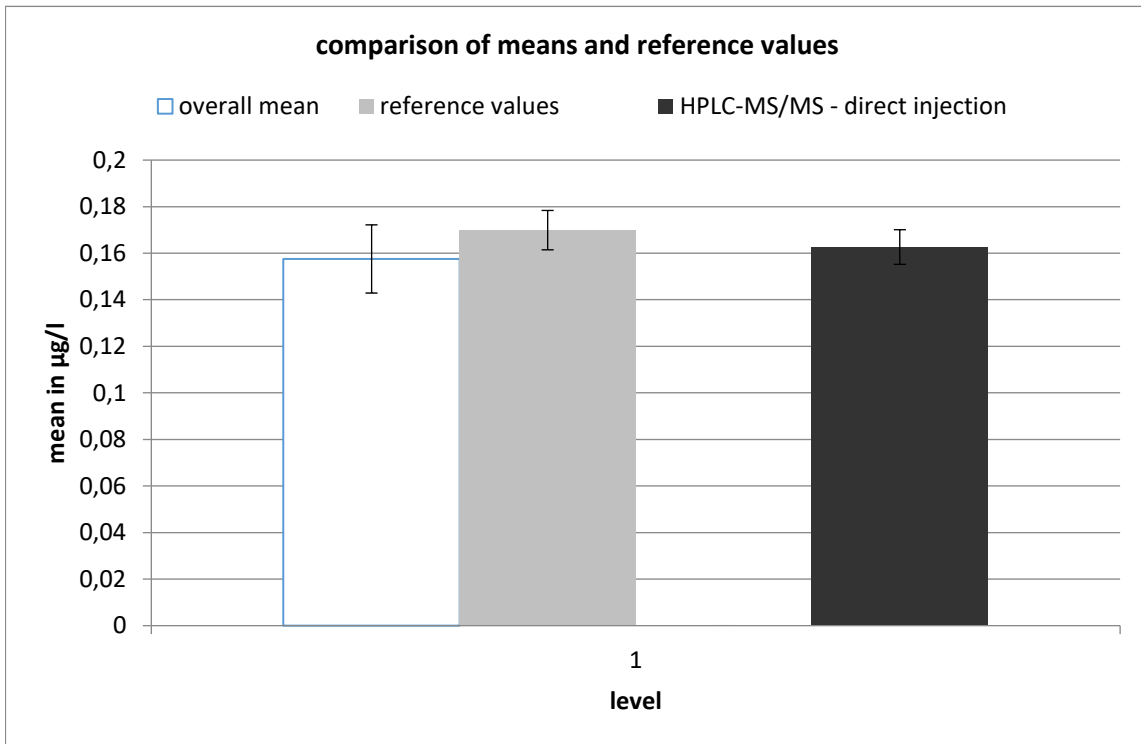
Method specific evaluation

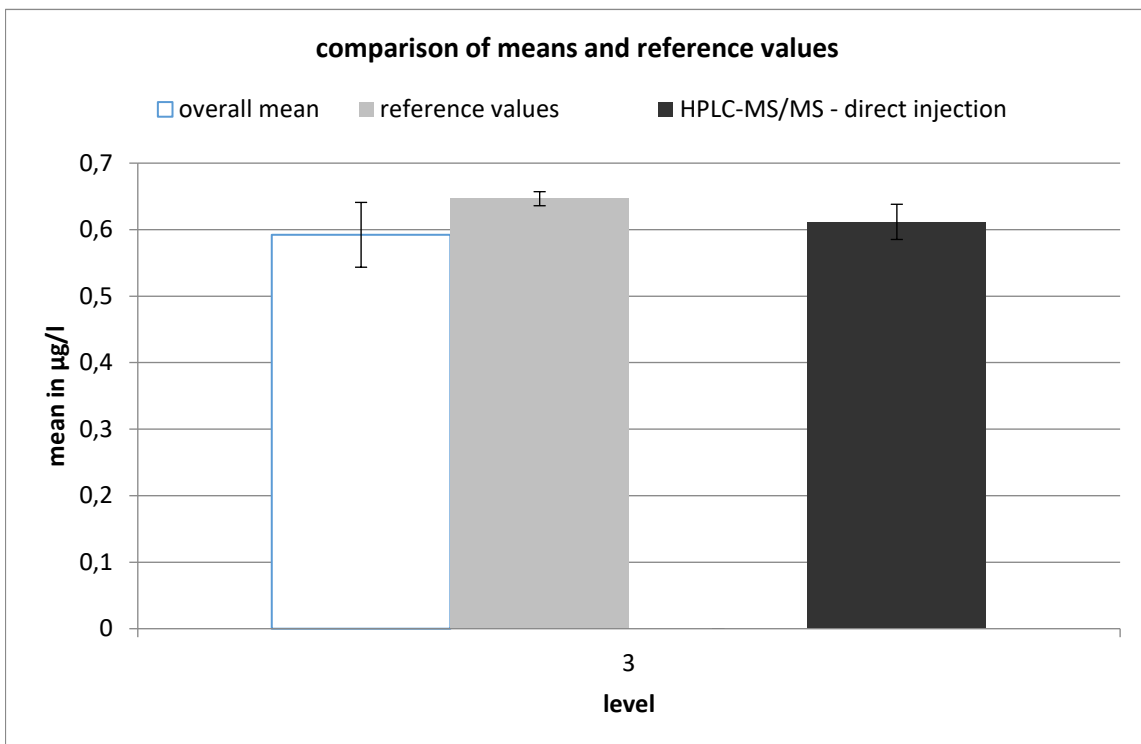
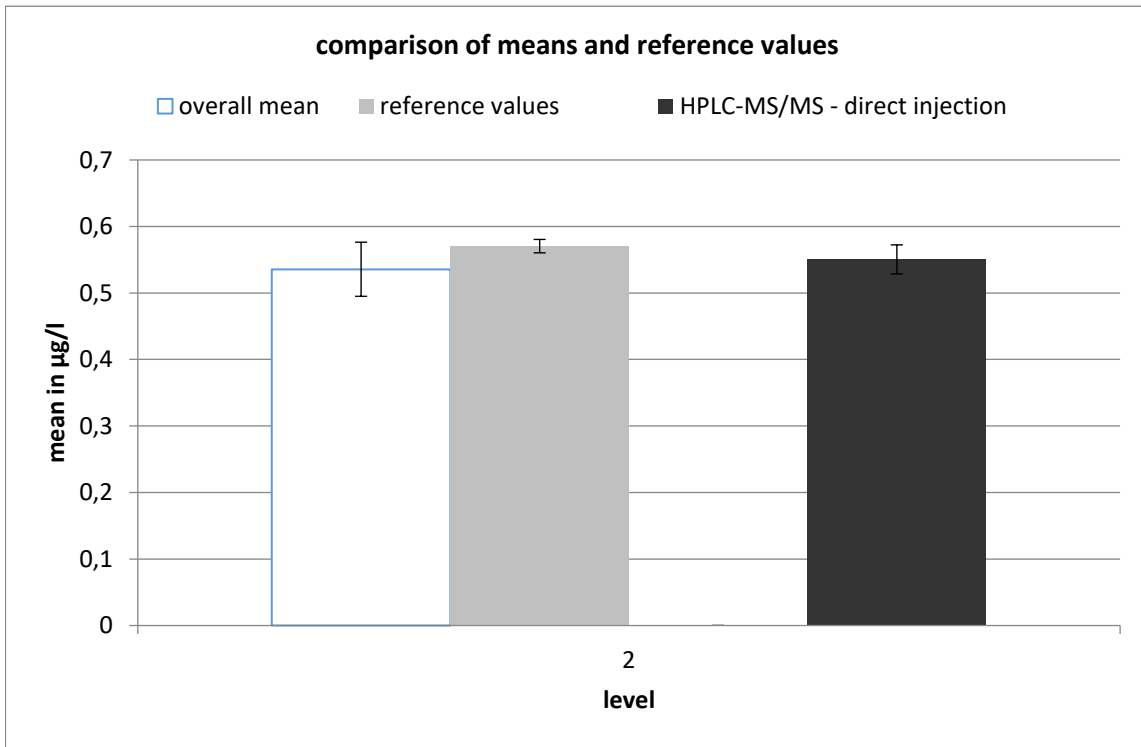


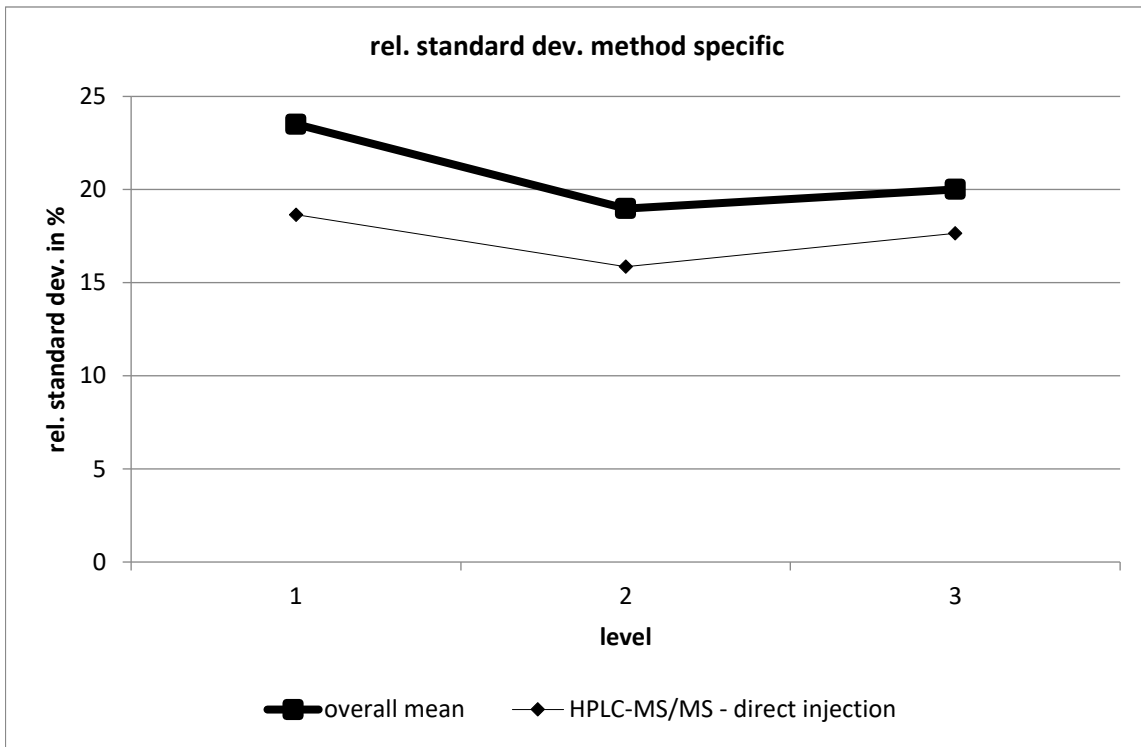
The values determined with HPLC-MS/MS-direct injection showed the closest statistical distribution.

Comparison of means and reference values

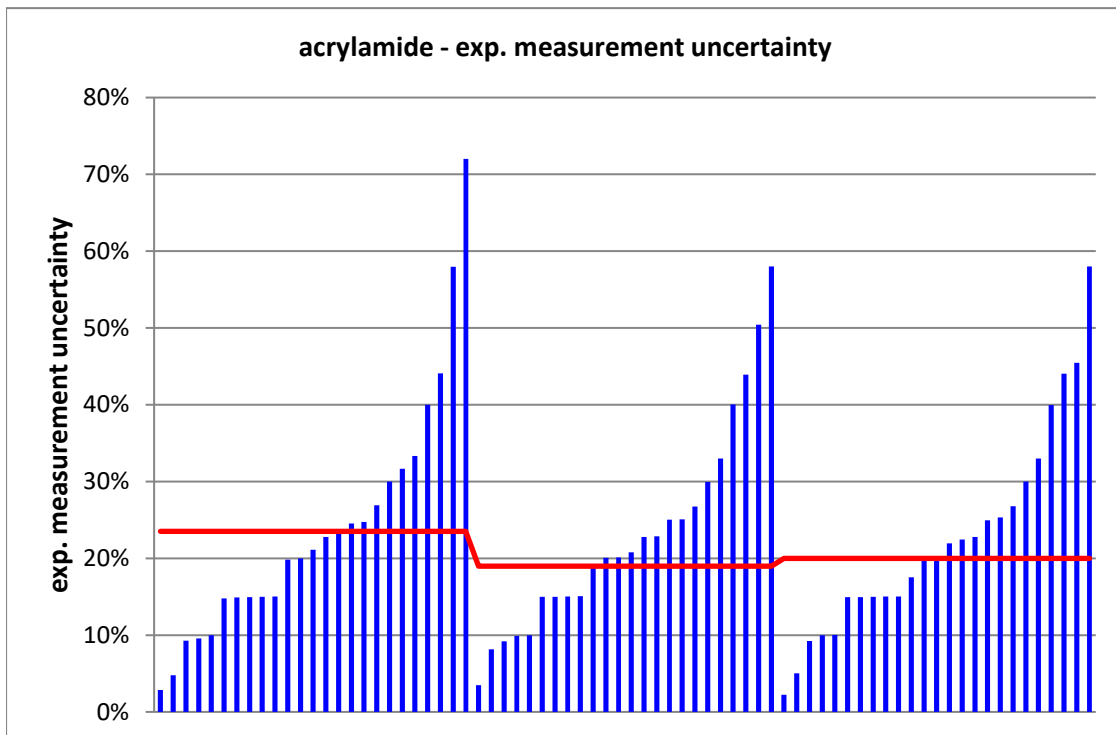
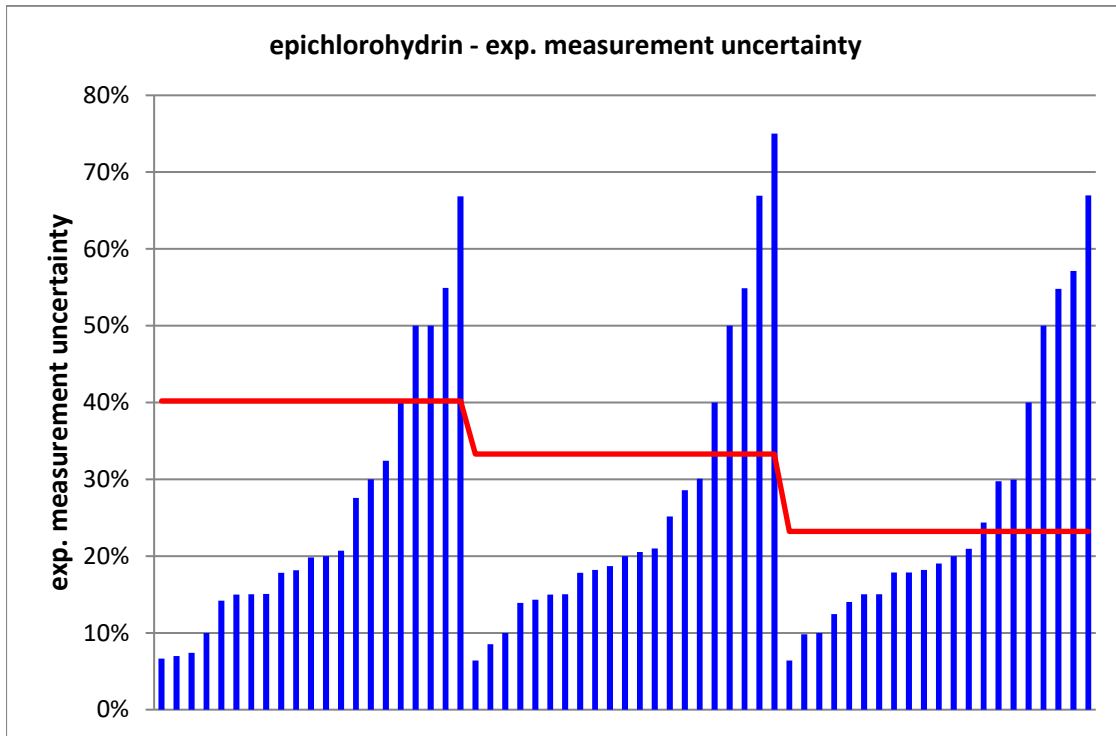
level	mean [µg/l]	exp. uncertainty [µg/l]	exp. uncertainty [%]	reference value [µg/l]	exp. uncertainty [µg/l]	exp. uncertainty [%]
1	0,1575	0,0146	9,3	0,1699	0,0085	5,0
2	0,5357	0,0407	7,6	0,5704	0,0101	1,8
3	0,5922	0,0487	8,2	0,6465	0,0106	1,6







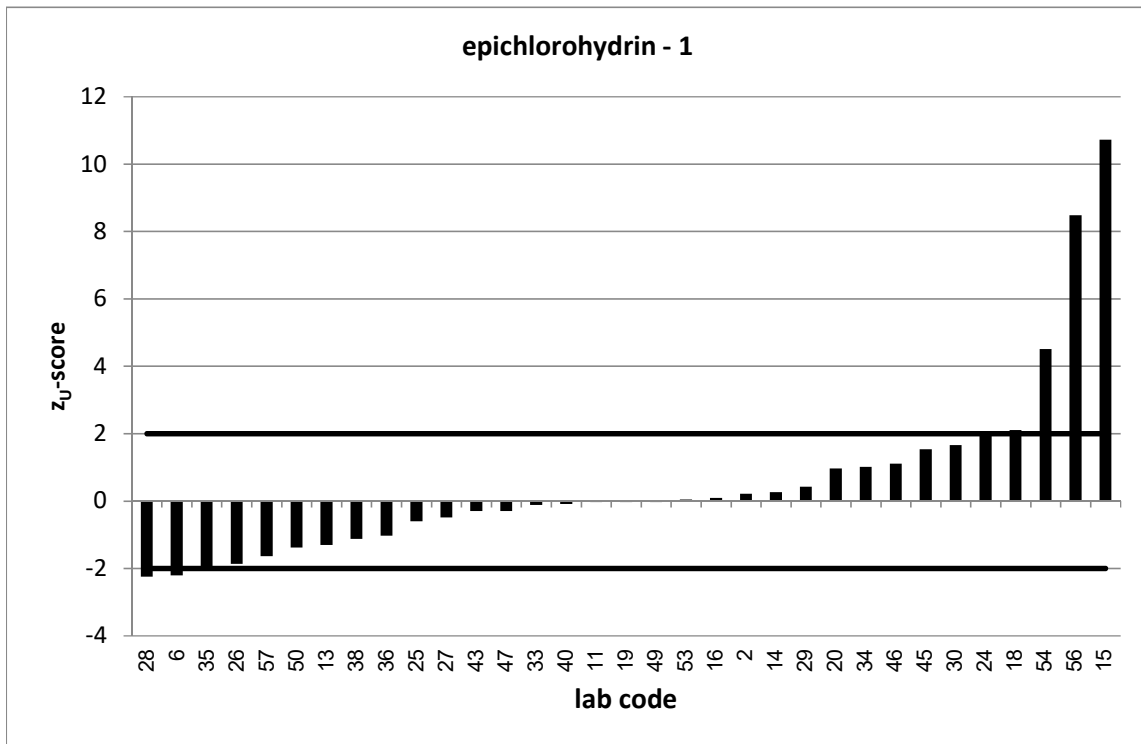
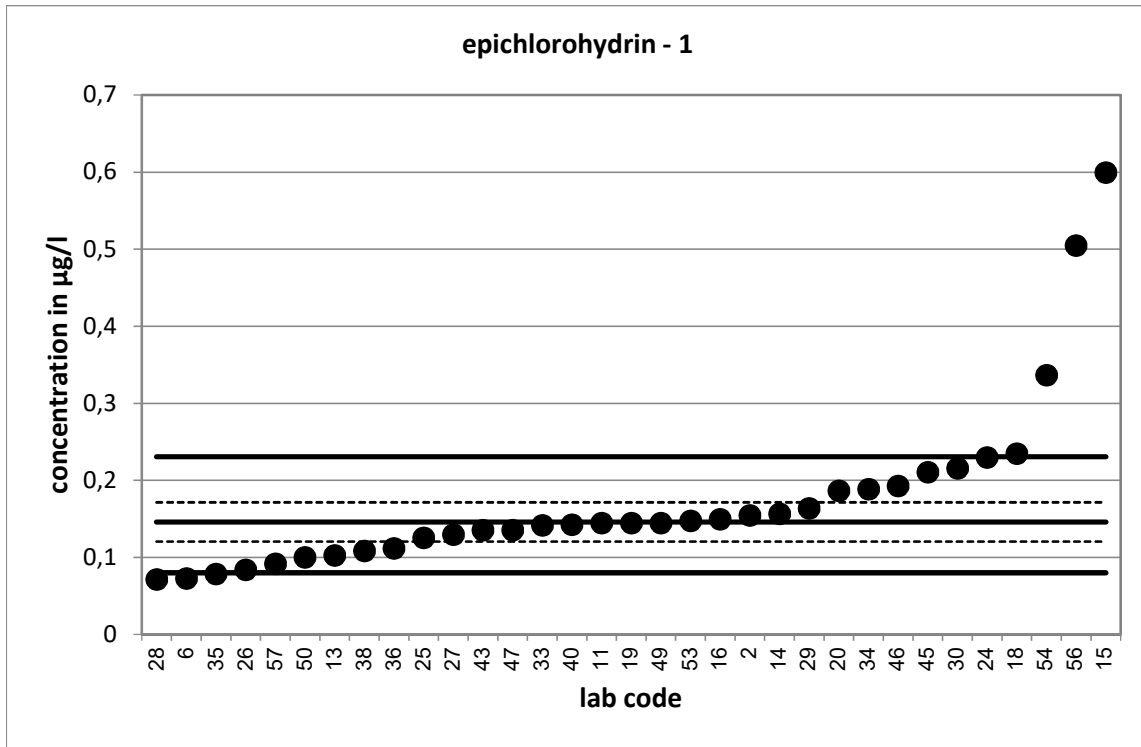
HPLC-MS/MS - direct injection									
level	robust mean [$\mu\text{g/l}$]	exp. unc. of the mean [$\mu\text{g/l}$]	exp. unc. of the mean [%]	robust standard deviation [$\mu\text{g/l}$]	robust standard deviation [%]	number of results	out below	out above	out [%]
1	0,163	0,007	4,573	0,03	18,65	26	1	1	7,692
2	0,55	0,022	3,964	0,087	15,86	25	2	1	12
3	0,612	0,026	4,328	0,108	17,66	26	2	1	11,54

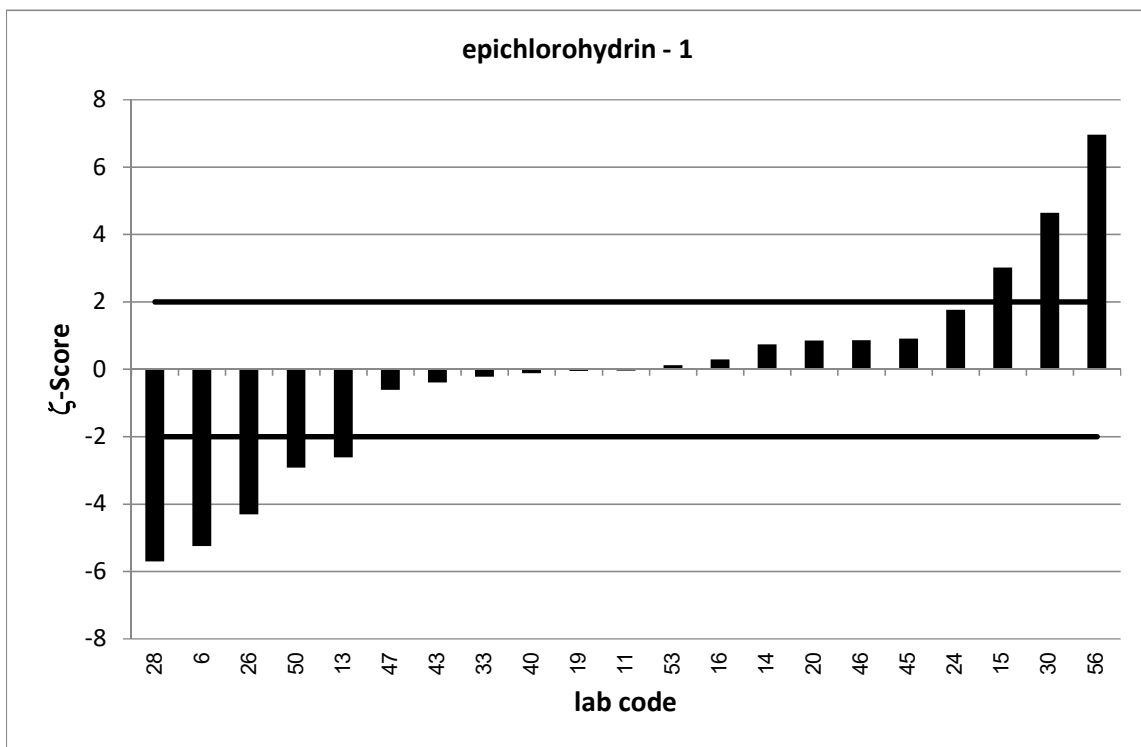
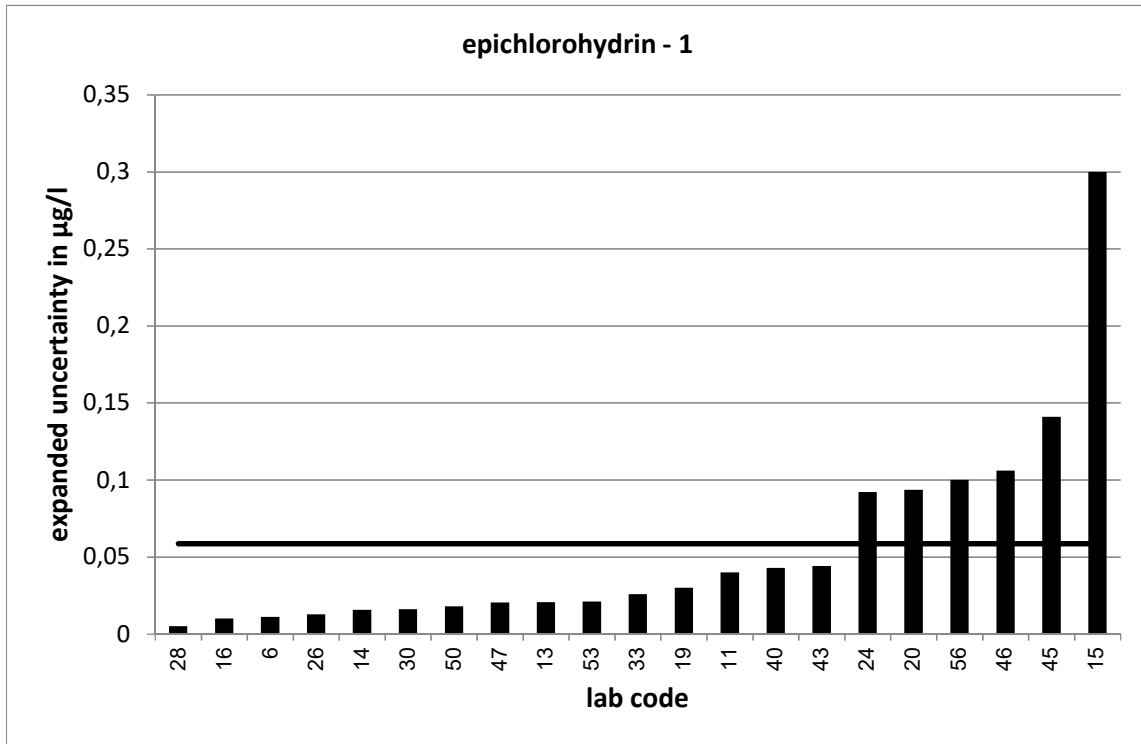


PT 8/20 - TW S10		epichlorohydrin - 1			
assigned value [$\mu\text{g/l}$]*		0,146 \pm 0,0255			
upper tolerance limit [$\mu\text{g/l}$]		0,2307			
lower tolerance limit [$\mu\text{g/l}$]		0,08003			
lab code	result [$\mu\text{g/l}$]	\pm	ζ -score	z_U -score	assessm.**
2	0,155			0,2	s
6	0,073	0,011	-5,2	-2,2	q
11	0,145	0,04	0,0	0,0	s
13	0,103	0,021	-2,6	-1,3	s
14	0,157	0,016	0,7	0,3	s
15	0,6	0,3	3,0	10,7	u
16	0,15	0,01	0,3	0,1	s
18	0,235			2,1	q
19	0,145	0,03	-0,1	0,0	s
20	0,187	0,094	0,8	1,0	s
24	0,23	0,092	1,8	2,0	s
25	0,126			-0,6	s
26	0,0845	0,013	-4,3	-1,9	s
27	0,13			-0,5	s
28	0,0718	0,005	-5,7	-2,2	q
29	0,164			0,4	s
30	0,216	0,016	4,6	1,7	s
33	0,142	0,026	-0,2	-0,1	s
34	0,189			1,0	s
35	0,079			-2,0	s
36	0,112			-1,0	s
38	0,109			-1,1	s
40	0,143	0,043	-0,1	-0,1	s
43	0,136	0,044	-0,4	-0,3	s
45	0,211	0,141	0,9	1,5	s
46	0,193	0,106	0,9	1,1	s
47	0,136	0,02	-0,6	-0,3	s
49	0,145			0,0	s
50	0,1005	0,018	-2,9	-1,4	s
53	0,148	0,021	0,1	0,0	s
54	0,337			4,5	u
56	0,505	0,1	7,0	8,5	u
57	0,092			-1,6	s

* The stated uncertainty of the assigned value is the expanded uncertainty with a coverage factor $k=2$ corresponding to a confidence level of about 95%

** s = satisfactory, q = questionable, u = unsatisfactory

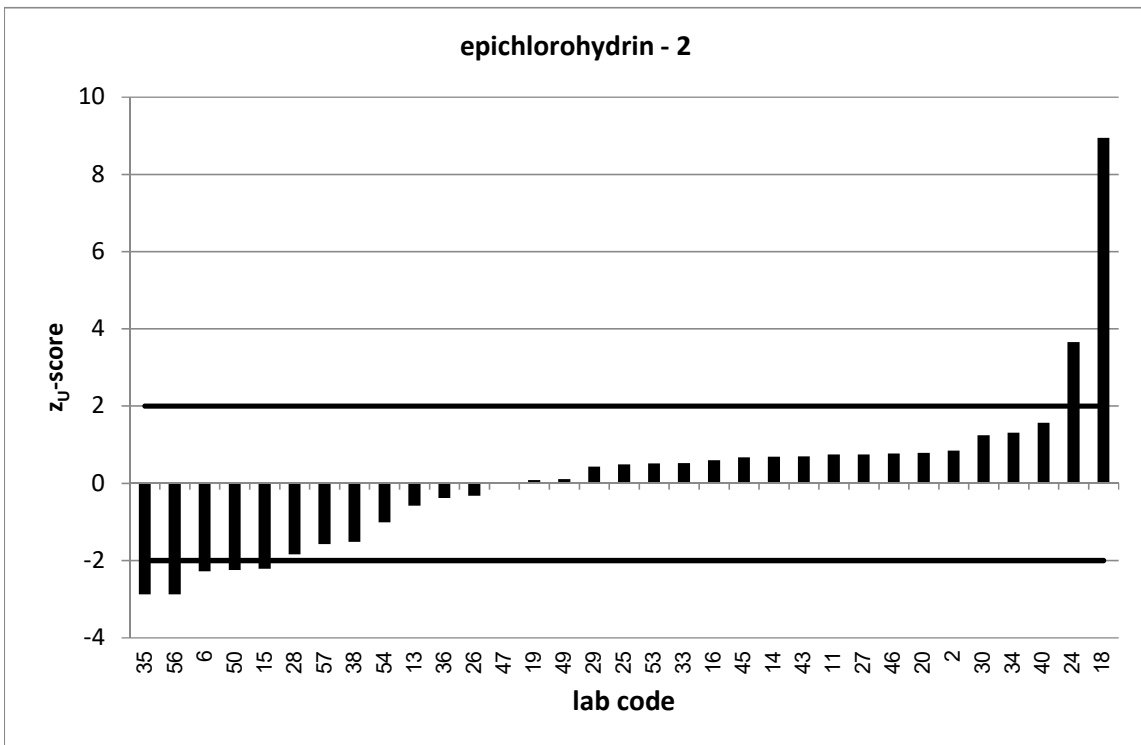
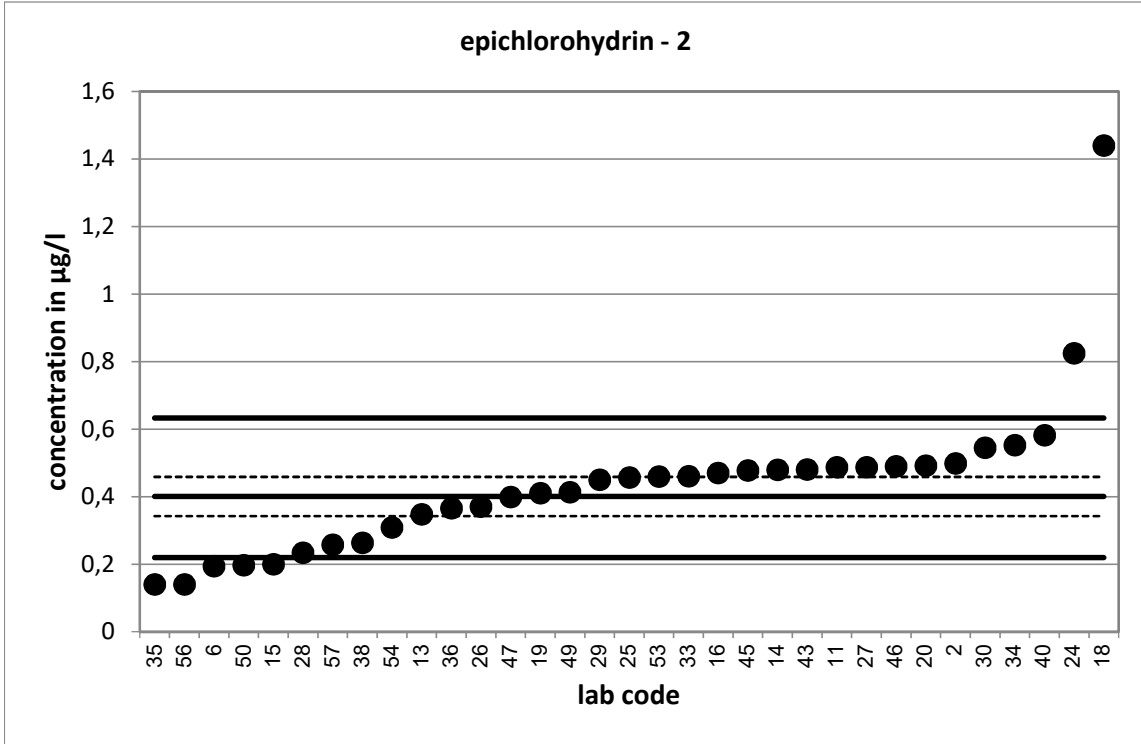


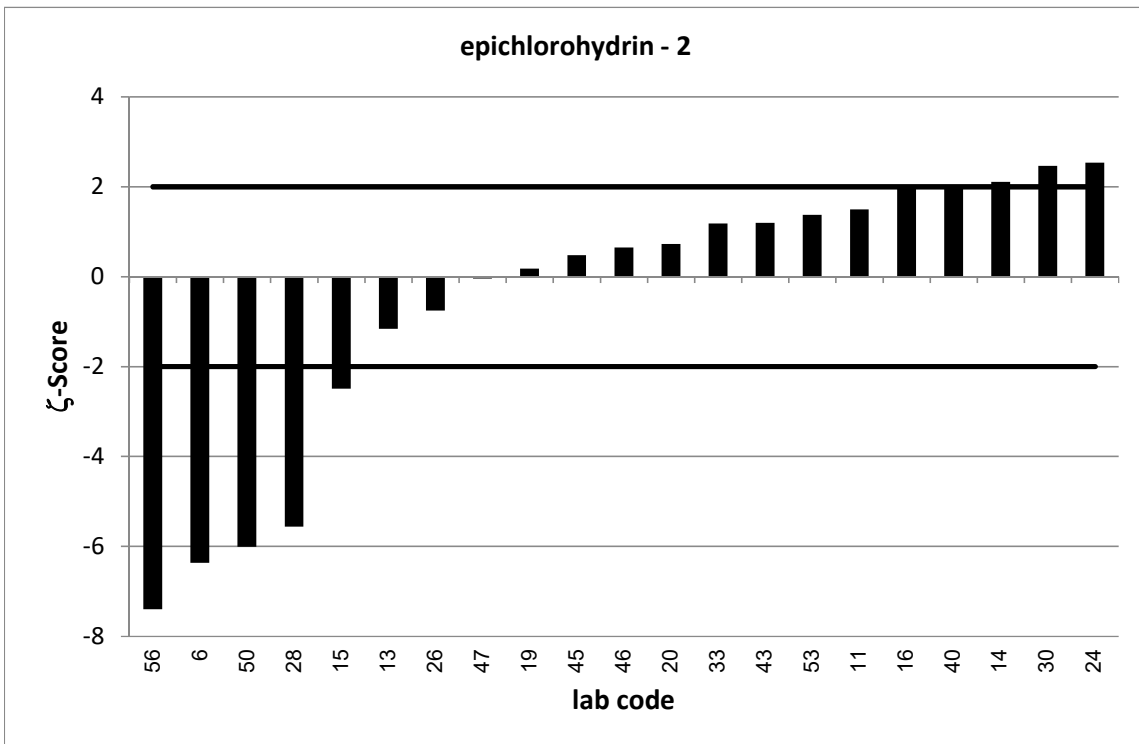
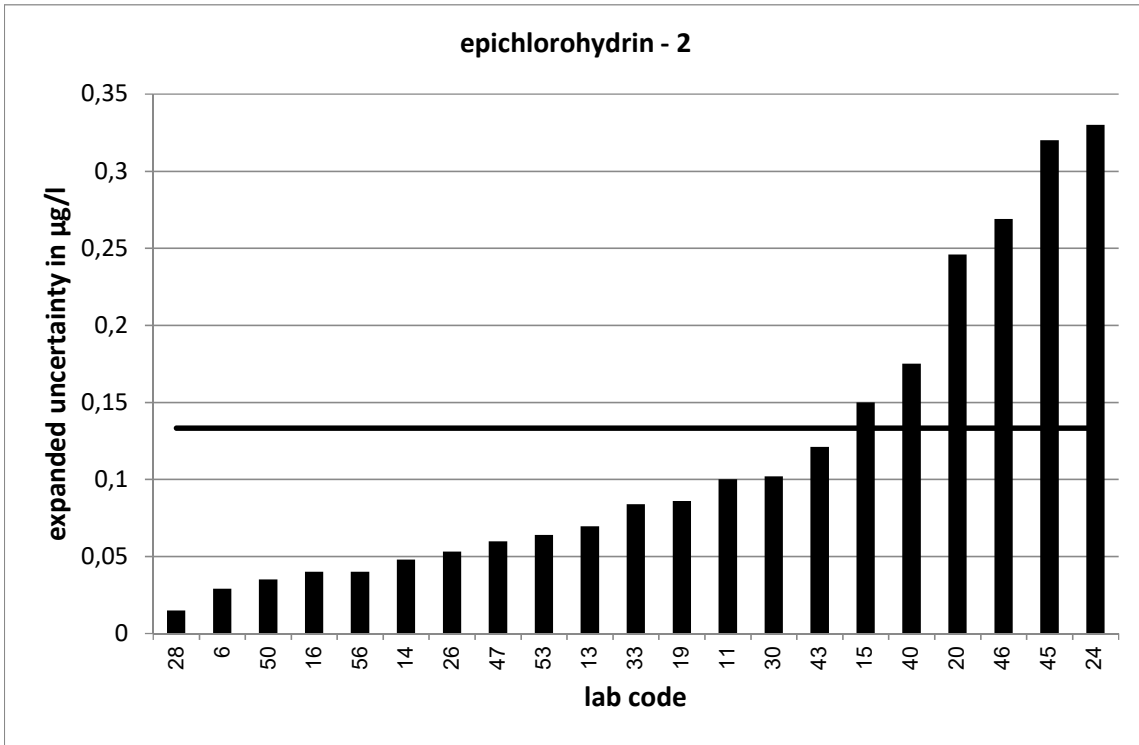


PT 8/20 - TW S10		epichlorohydrin - 2			
assigned value [$\mu\text{g/l}$]*		0,4007 \pm 0,058			
upper tolerance limit [$\mu\text{g/l}$]		0,6331			
lower tolerance limit [$\mu\text{g/l}$]		0,2196			
lab code	result [$\mu\text{g/l}$]	\pm	ζ -score	z_U -score	assessm.**
2	0,499			0,8	s
6	0,194	0,029	-6,4	-2,3	q
11	0,487	0,1	1,5	0,7	s
13	0,348	0,07	-1,2	-0,6	s
14	0,48	0,048	2,1	0,7	s
15	0,2	0,15	-2,5	-2,2	q
16	0,47	0,04	2,0	0,6	s
18	1,44			8,9	u
19	0,41	0,086	0,2	0,1	s
20	0,492	0,246	0,7	0,8	s
24	0,825	0,33	2,5	3,7	u
25	0,457			0,5	s
26	0,371	0,053	-0,8	-0,3	s
27	0,487			0,7	s
28	0,234	0,015	-5,6	-1,8	s
29	0,45			0,4	s
30	0,545	0,102	2,5	1,2	s
33	0,461	0,084	1,2	0,5	s
34	0,553			1,3	s
35	0,14			-2,9	q
36	0,366			-0,4	s
38	0,263			-1,5	s
40	0,582	0,175	2,0	1,6	s
43	0,481	0,121	1,2	0,7	s
45	0,478	0,32	0,5	0,7	s
46	0,49	0,269	0,6	0,8	s
47	0,399	0,06	0,0	0,0	s
49	0,413			0,1	s
50	0,1969	0,035	-6,0	-2,3	q
53	0,46	0,064	1,4	0,5	s
54	0,309			-1,0	s
56	0,14	0,04	-7,4	-2,9	q
57	0,258			-1,6	s

* The stated uncertainty of the assigned value is the expanded uncertainty with a coverage factor $k=2$ corresponding to a confidence level of about 95%

** s = satisfactory, q = questionable, u = unsatisfactory

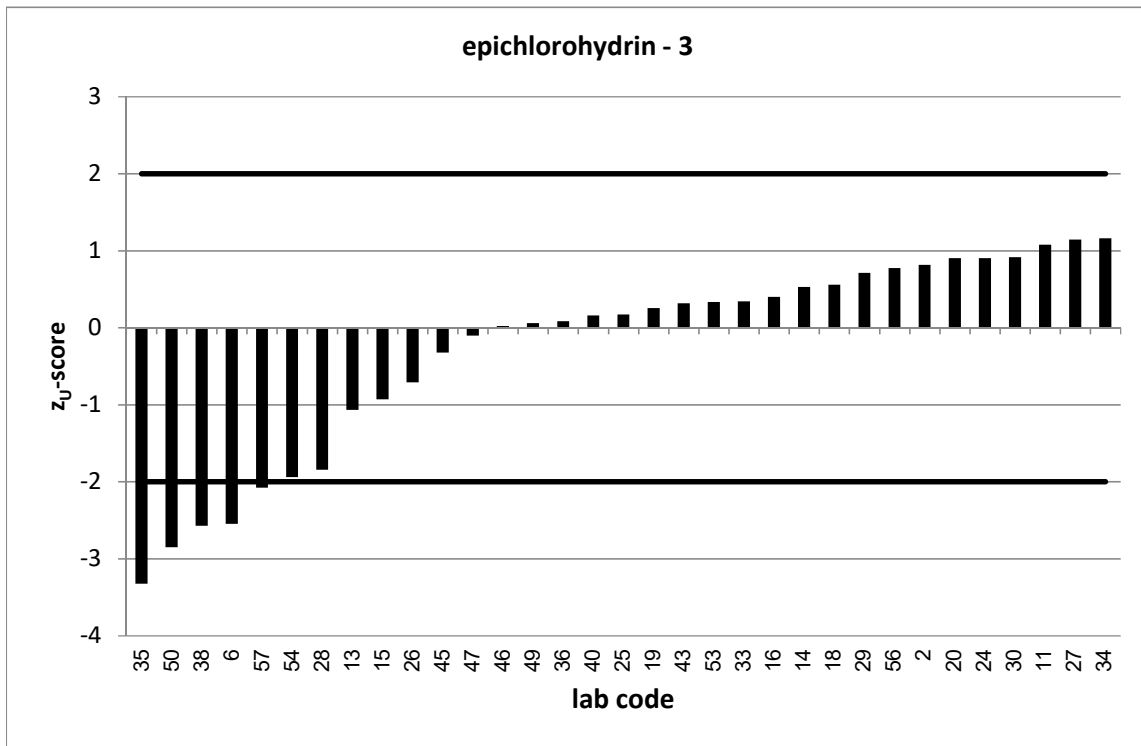
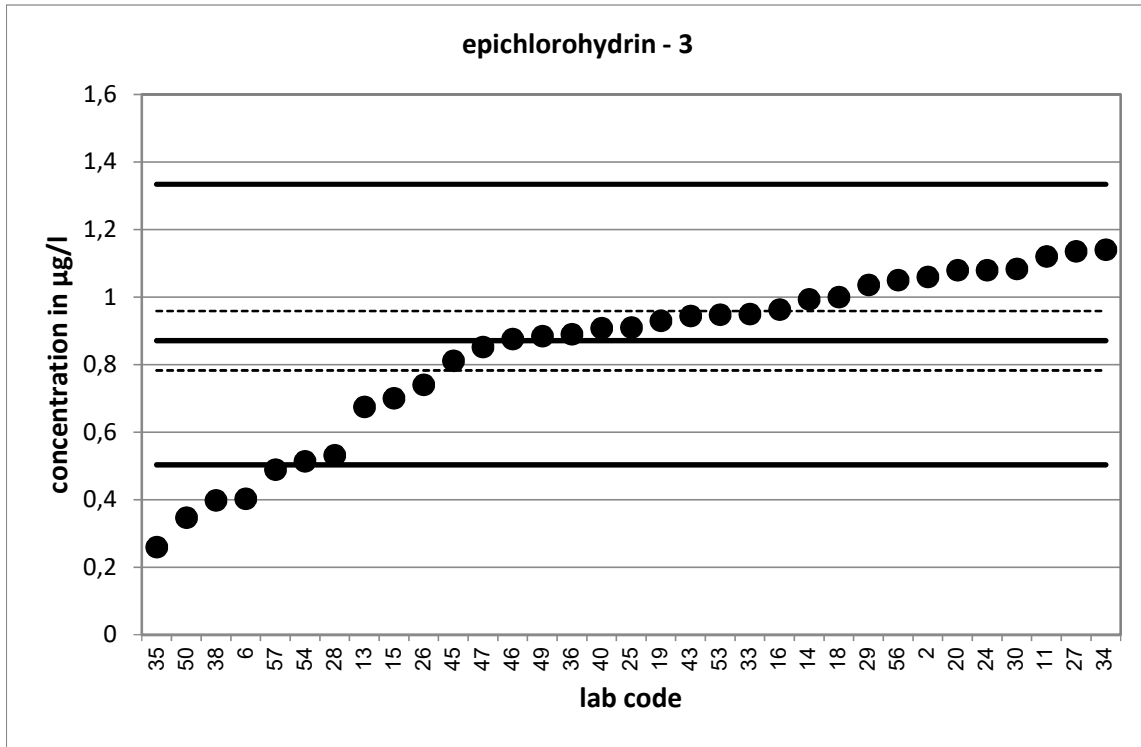


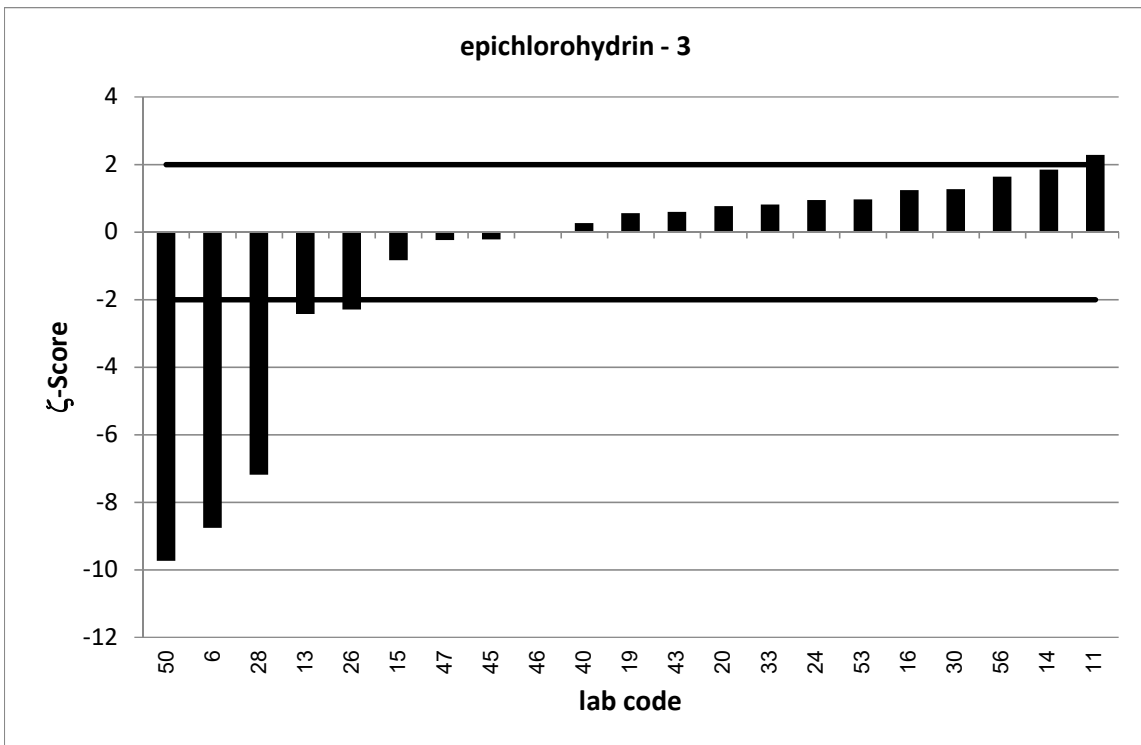
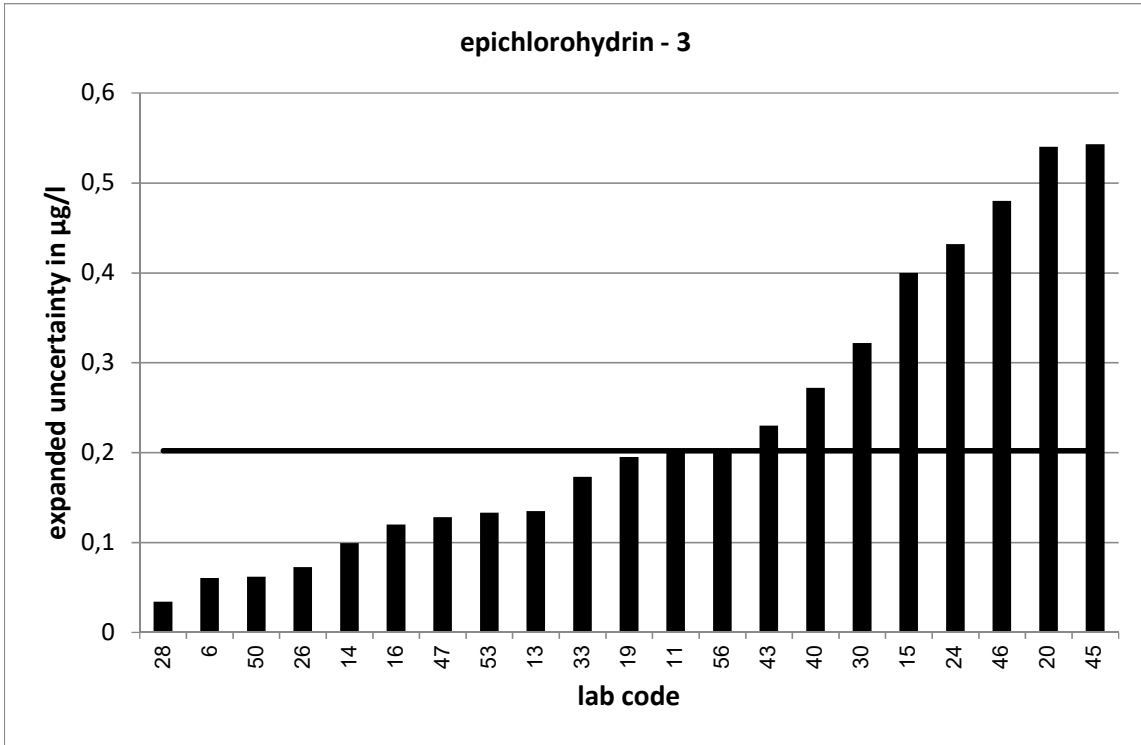


PT 8/20 - TW S10		epichlorohydrin - 3			
assigned value [$\mu\text{g/l}$]*		0,8708 \pm 0,088			
upper tolerance limit [$\mu\text{g/l}$]		1,334			
lower tolerance limit [$\mu\text{g/l}$]		0,5033			
lab code	result [$\mu\text{g/l}$]	\pm	ζ -score	z_U -score	assessm.**
2	1,06			0,8	s
6	0,403	0,061	-8,8	-2,5	q
11	1,12	0,2	2,3	1,1	s
13	0,675	0,135	-2,4	-1,1	s
14	0,993	0,099	1,8	0,5	s
15	0,7	0,4	-0,8	-0,9	s
16	0,963	0,12	1,2	0,4	s
18	1			0,6	s
19	0,93	0,195	0,6	0,3	s
20	1,08	0,54	0,8	0,9	s
24	1,08	0,432	0,9	0,9	s
25	0,91			0,2	s
26	0,74	0,073	-2,3	-0,7	s
27	1,136			1,1	s
28	0,532	0,034	-7,2	-1,8	s
29	1,036			0,7	s
30	1,083	0,322	1,3	0,9	s
33	0,95	0,173	0,8	0,3	s
34	1,14			1,2	s
35	0,26			-3,3	u
36	0,89			0,1	s
38	0,398			-2,6	q
40	0,908	0,272	0,3	0,2	s
43	0,944	0,23	0,6	0,3	s
45	0,811	0,543	-0,2	-0,3	s
46	0,876	0,48	0,0	0,0	s
47	0,852	0,128	-0,2	-0,1	s
49	0,884			0,1	s
50	0,3469	0,062	-9,7	-2,9	q
53	0,948	0,133	1,0	0,3	s
54	0,514			-1,9	s
56	1,05	0,2	1,6	0,8	s
57	0,489			-2,1	q

* The stated uncertainty of the assigned value is the expanded uncertainty with a coverage factor $k=2$ corresponding to a confidence level of about 95%

** s = satisfactory, q = questionable, u = unsatisfactory

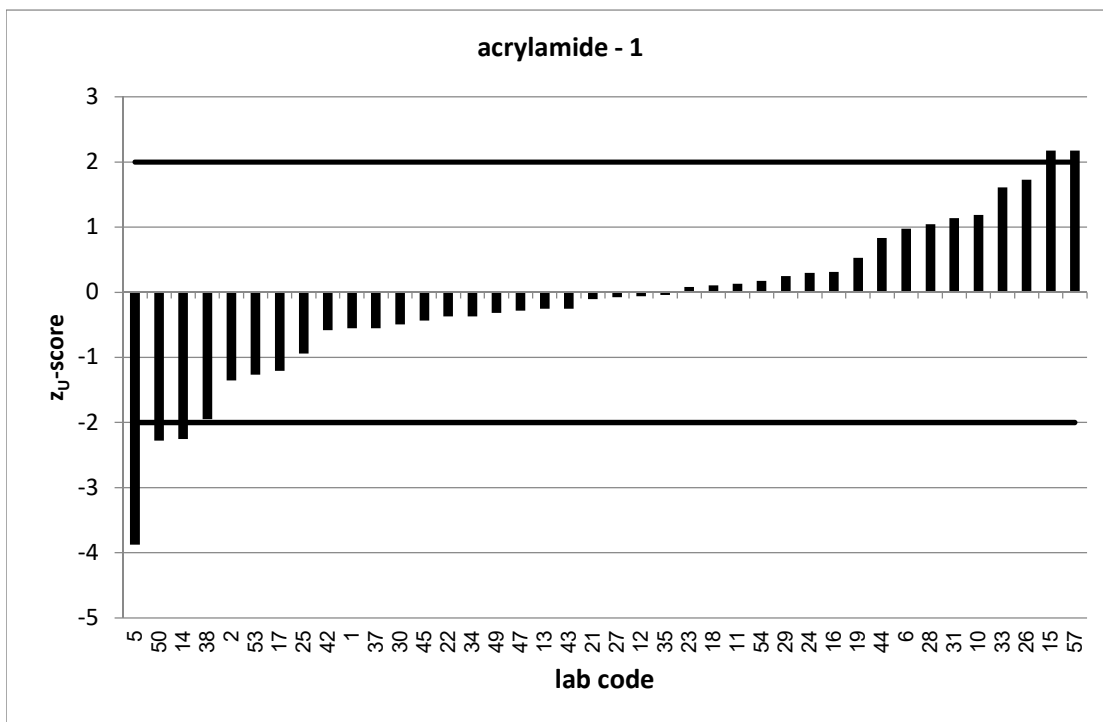
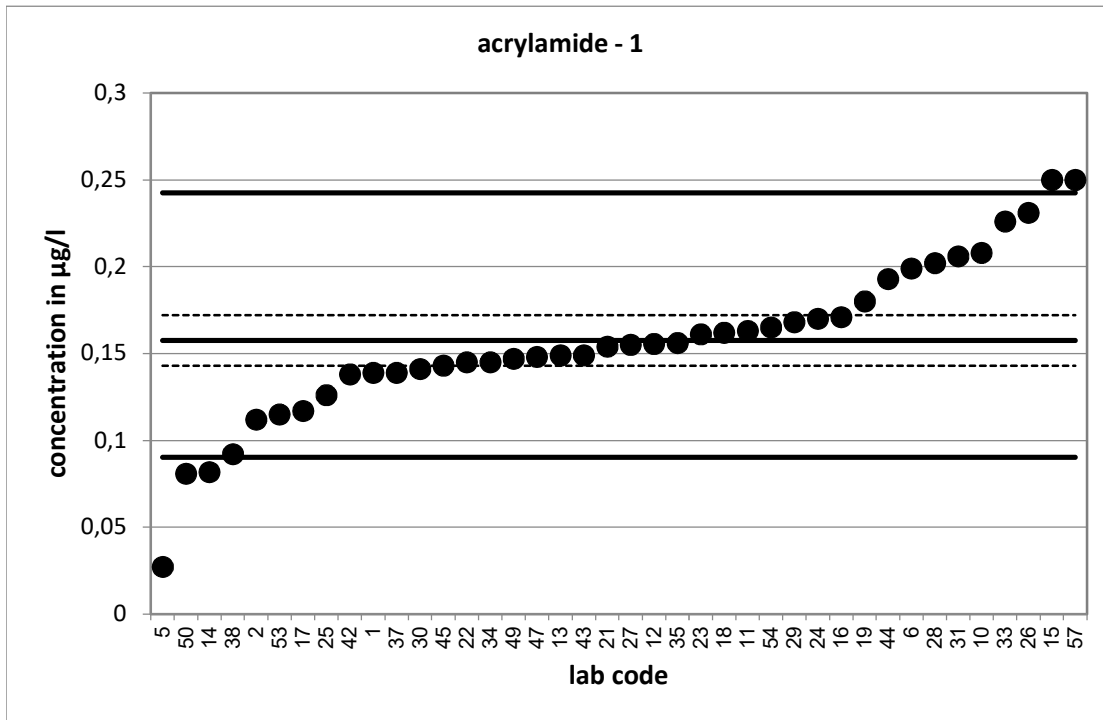


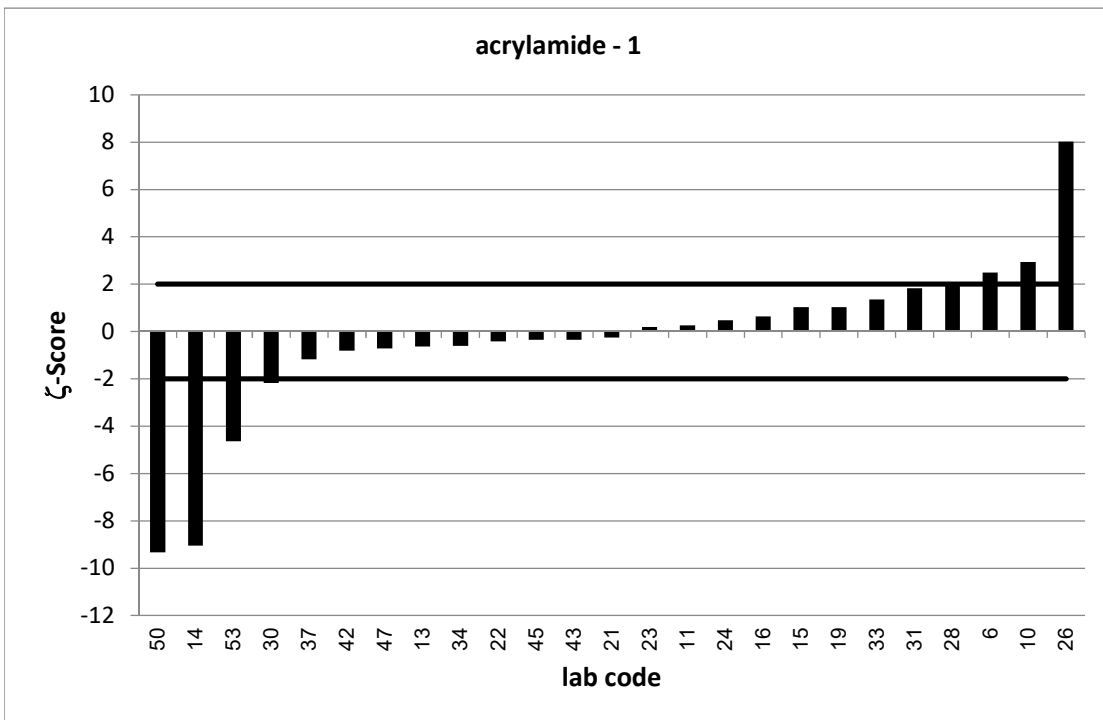
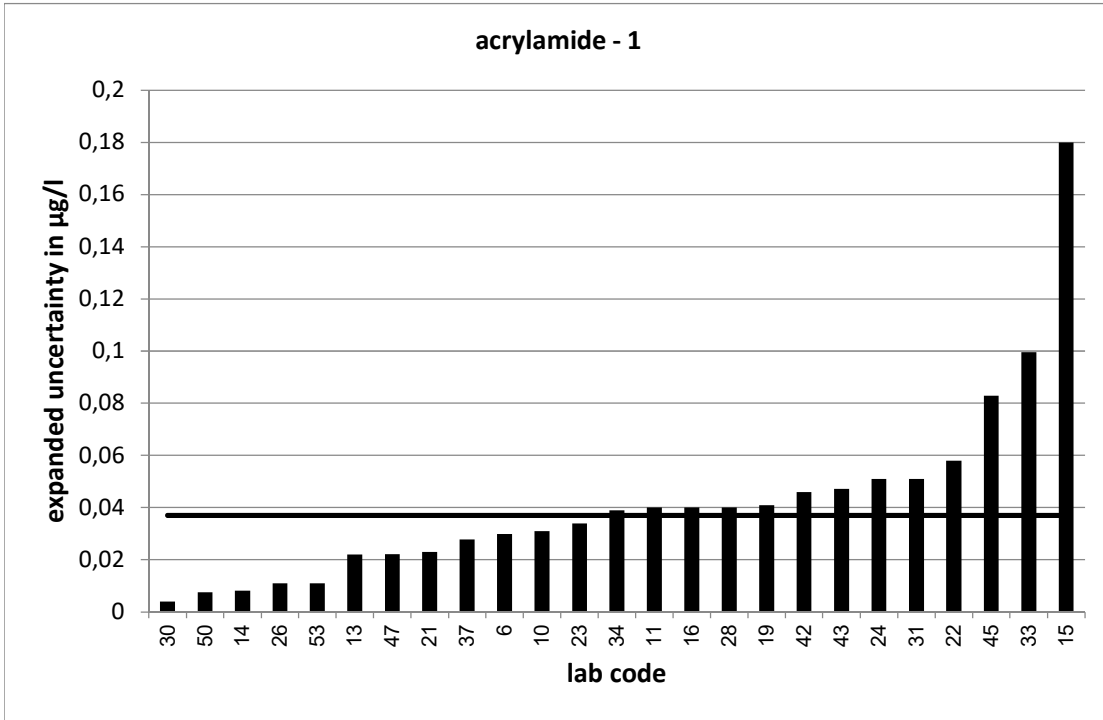


PT 8/20 - TW S10		acrylamide - 1			
assigned value [$\mu\text{g/l}$]*		0,1575 \pm 0,0146			
upper tolerance limit [$\mu\text{g/l}$]		0,2426			
lower tolerance limit [$\mu\text{g/l}$]		0,09026			
lab code	result [$\mu\text{g/l}$]	\pm	ζ -score	z_U -score	assessm.**
1	0,139			-0,6	s
2	0,112			-1,4	s
5	0,0271			-3,9	u
6	0,199	0,03	2,5	1,0	s
10	0,208	0,031	2,9	1,2	s
11	0,163	0,04	0,3	0,1	s
12	0,1555			-0,1	s
13	0,149	0,022	-0,6	-0,3	s
14	0,0817	0,008	-9,0	-2,3	q
15	0,25	0,18	1,0	2,2	q
16	0,171	0,04	0,6	0,3	s
17	0,117			-1,2	s
18	0,162			0,1	s
19	0,18	0,041	1,0	0,5	s
21	0,154	0,023	-0,3	-0,1	s
22	0,145	0,058	-0,4	-0,4	s
23	0,161	0,034	0,2	0,1	s
24	0,17	0,051	0,5	0,3	s
25	0,126			-0,9	s
26	0,231	0,011	8,0	1,7	s
27	0,155			-0,1	s
28	0,202	0,04	2,1	1,0	s
29	0,168			0,2	s
30	0,141	0,004	-2,2	-0,5	s
31	0,206	0,051	1,8	1,1	s
33	0,226	0,1	1,4	1,6	s
34	0,145	0,039	-0,6	-0,4	s
35	0,156			0,0	s
37	0,139	0,028	-1,2	-0,6	s
38	0,092			-1,9	s
42	0,138	0,046	-0,8	-0,6	s
43	0,149	0,047	-0,3	-0,3	s
44	0,193			0,8	s
45	0,143	0,083	-0,3	-0,4	s
47	0,148	0,022	-0,7	-0,3	s
49	0,147			-0,3	s
50	0,0808	0,008	-9,3	-2,3	q
53	0,115	0,011	-4,6	-1,3	s
54	0,165			0,2	s
57	0,25			2,2	q

* The stated uncertainty of the assigned value is the expanded uncertainty with a coverage factor $k=2$ corresponding to a confidence level of about 95%

** s = satisfactory, q = questionable, u = unsatisfactory

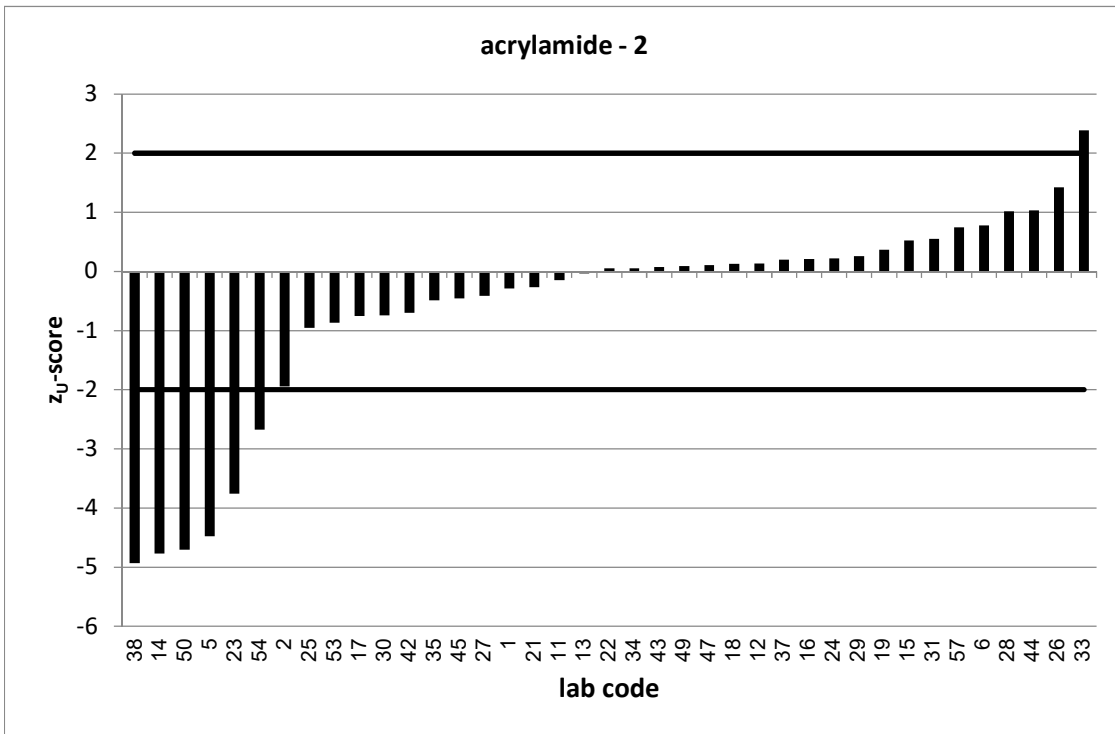
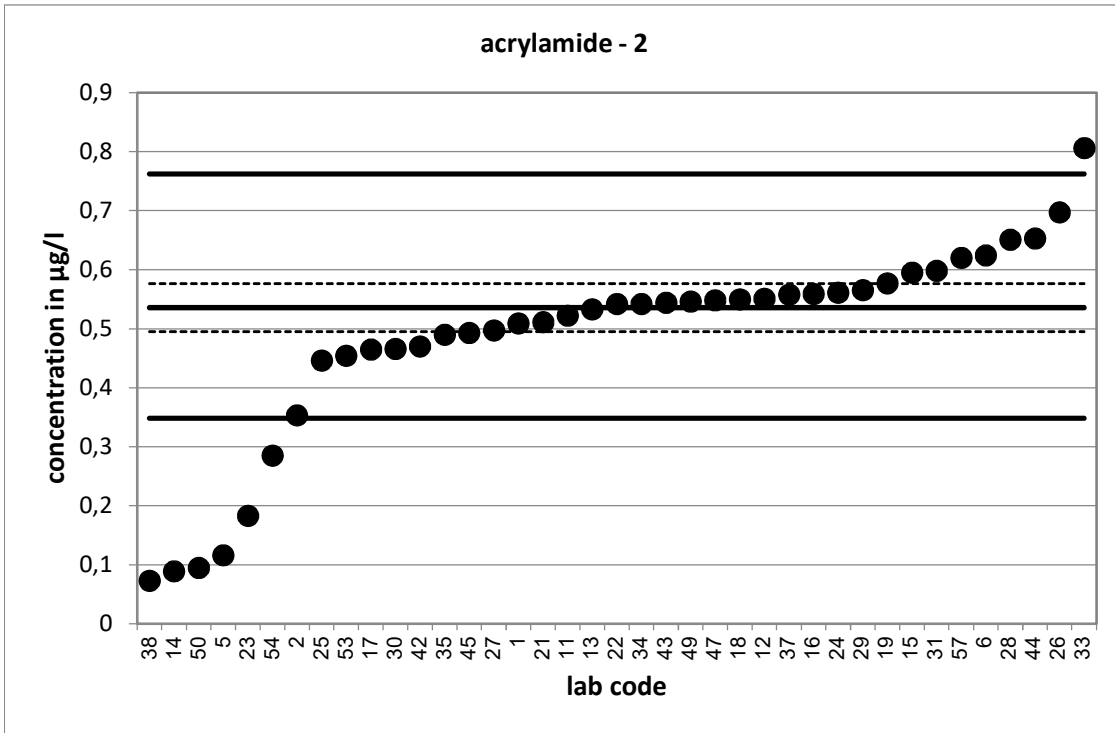


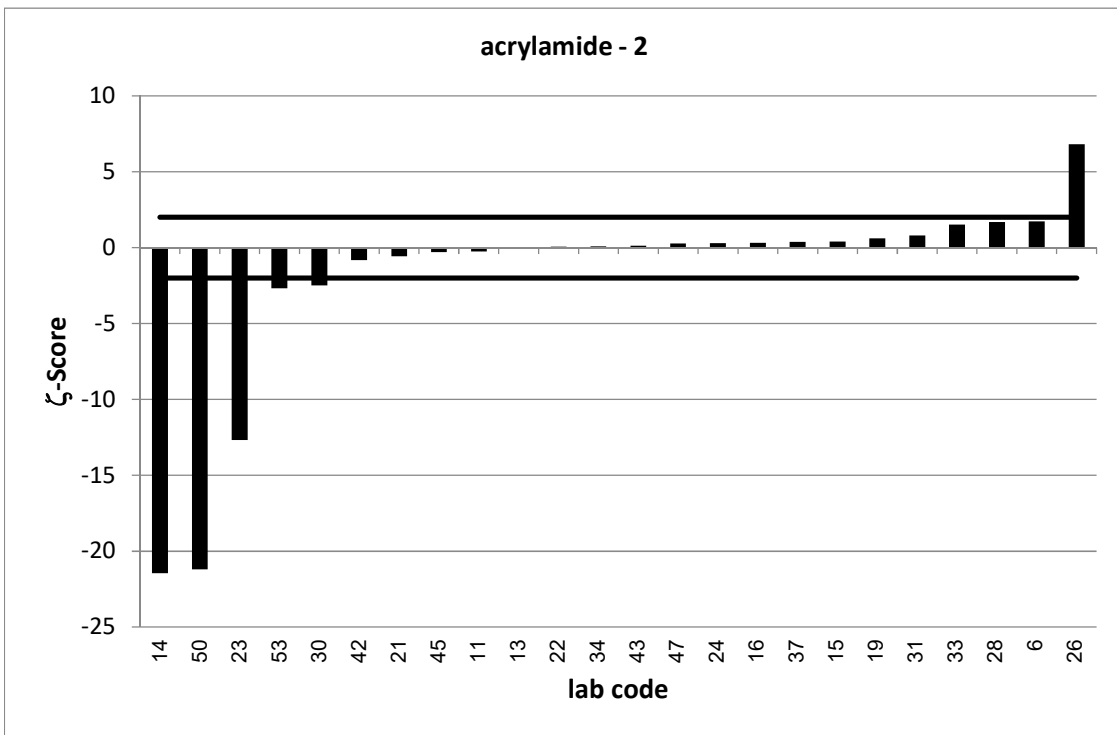
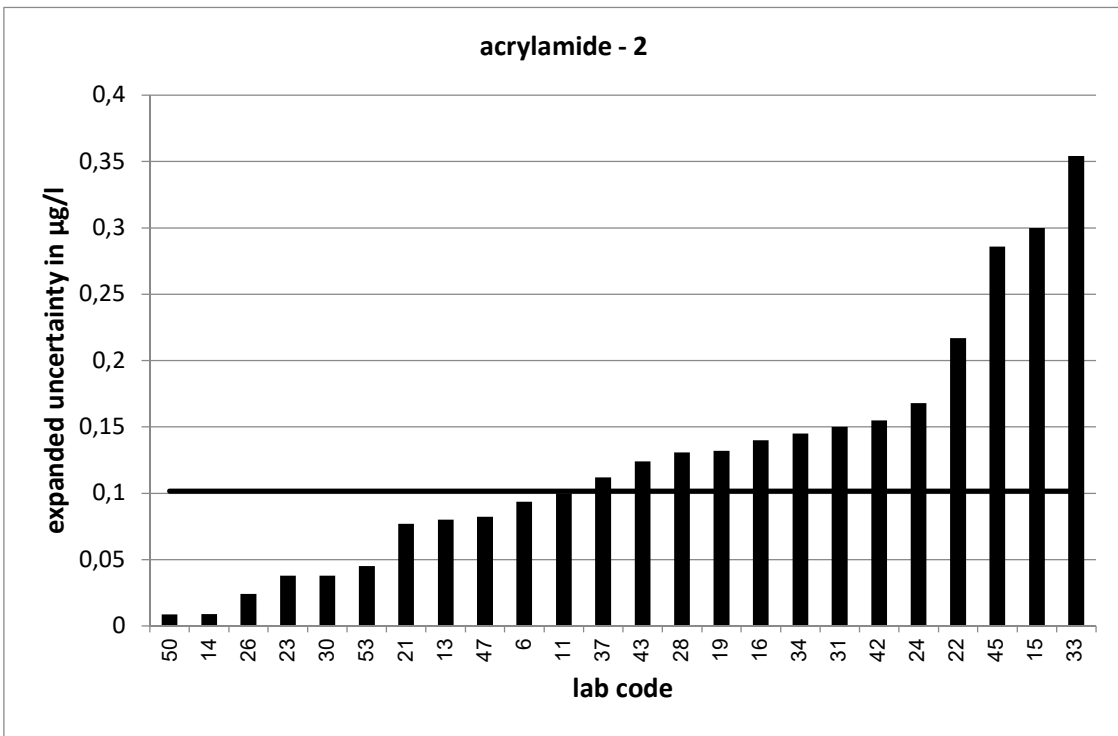


PT 8/20 - TW S10		acrylamide - 2			
assigned value [$\mu\text{g/l}$]*		0,5357 \pm 0,0407			
upper tolerance limit [$\mu\text{g/l}$]		0,7622			
lower tolerance limit [$\mu\text{g/l}$]		0,3482			
lab code	result [$\mu\text{g/l}$]	\pm	ζ -score	z_U -score	assessm.**
1	0,509			-0,3	s
2	0,353			-1,9	s
5	0,116			-4,5	u
6	0,624	0,094	1,7	0,8	s
11	0,522	0,1	-0,3	-0,1	s
12	0,5507			0,1	s
13	0,533	0,08	-0,1	0,0	s
14	0,0888	0,009	-21,5	-4,8	u
15	0,595	0,3	0,4	0,5	s
16	0,559	0,14	0,3	0,2	s
17	0,465			-0,8	s
18	0,55			0,1	s
19	0,577	0,132	0,6	0,4	s
21	0,511	0,077	-0,6	-0,3	s
22	0,542	0,217	0,1	0,1	s
23	0,183	0,038	-12,7	-3,8	u
24	0,561	0,168	0,3	0,2	s
25	0,446			-1,0	s
26	0,697	0,024	6,8	1,4	s
27	0,497			-0,4	s
28	0,651	0,131	1,7	1,0	s
29	0,565			0,3	s
30	0,466	0,038	-2,5	-0,7	s
31	0,598	0,15	0,8	0,6	s
33	0,806	0,354	1,5	2,4	q
34	0,542	0,145	0,1	0,1	s
35	0,49			-0,5	s
37	0,558	0,112	0,4	0,2	s
38	0,073			-4,9	u
42	0,47	0,155	-0,8	-0,7	s
43	0,544	0,124	0,1	0,1	s
44	0,653			1,0	s
45	0,493	0,286	-0,3	-0,5	s
47	0,548	0,082	0,3	0,1	s
49	0,546			0,1	s
50	0,0946	0,009	-21,2	-4,7	u
53	0,454	0,045	-2,7	-0,9	s
54	0,285			-2,7	q
57	0,62			0,7	s

* The stated uncertainty of the assigned value is the expanded uncertainty with a coverage factor $k=2$ corresponding to a confidence level of about 95%

** s = satisfactory, q = questionable, u = unsatisfactory





PT 8/20 - TW S10		acrylamide - 3			
assigned value [$\mu\text{g/l}$]*		0,5922 \pm 0,0487			
upper tolerance limit [$\mu\text{g/l}$]		0,858			
lower tolerance limit [$\mu\text{g/l}$]		0,3745			
lab code	result [$\mu\text{g/l}$]	\pm	ζ -score	z_U -score	assessm.**
1	0,607			0,1	s
2	0,406			-1,7	s
5	0,0447			-5,0	u
6	0,732	0,11	2,3	1,1	s
10	0,348	0,052	-6,9	-2,2	q
11	0,628	0,11	0,6	0,3	s
12	0,627			0,3	s
13	0,595	0,089	0,1	0,0	s
14	0,069	0,007	-21,3	-4,8	u
15	0,88	0,4	1,4	2,2	q
16	0,624	0,14	0,4	0,2	s
17	0,456			-1,3	s
18	0,64			0,4	s
19	0,65	0,148	0,7	0,4	s
21	0,621	0,093	0,5	0,2	s
22	0,613	0,245	0,2	0,2	s
23	0,0501	0,011	-21,7	-5,0	u
24	0,647	0,194	0,5	0,4	s
25	0,491			-0,9	s
26	0,807	0,018	8,3	1,6	s
27	0,541			-0,5	s
28	0,736	0,148	1,8	1,1	s
29	0,645			0,4	s
30	0,536	0,027	-2,0	-0,5	s
31	0,693	0,173	1,1	0,8	s
33	0,854	0,376	1,4	2,0	s
34	0,586	0,157	-0,1	-0,1	s
35	0,557			-0,3	s
37	0,651	0,13	0,8	0,4	s
38	0,265			-3,0	u
42	0,509	0,168	-1,0	-0,8	s
43	0,608	0,154	0,2	0,1	s
44	0,727			1,0	s
45	0,574	0,333	-0,1	-0,2	s
47	0,619	0,093	0,5	0,2	s
49	0,627			0,3	s
50	0,3176	0,029	-9,7	-2,5	q
53	0,499	0,05	-2,7	-0,9	s
54	0,366			-2,1	q
57	0,16			-4,0	u

* The stated uncertainty of the assigned value is the expanded uncertainty with a coverage factor $k=2$ corresponding to a confidence level of about 95%

** s = satisfactory, q = questionable, u = unsatisfactory

