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Control charts for internal quality control in forensic-toxicological analyses

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Aims: General requirements for the competence of testing and calibration laboratories as a basis for accreditation can be found in the respective valid version of DIN norm EN ISO/IEC 17025 and the guidelines for quality control in forensic-toxicological analyses of the GTFCh.The guidelines of the GTFCh have been revised in 2011. Accordingly, internal quality procedures involve a control chart issued per analyte, concentration, and measuring device. For deviation of the measured value from the reference value, a maximum of ± 30% (or ± 40% at the detection limit) has been stipulated containing systematic as well as random errors. The control chart should also be checked if seven successive values increase or drop monotonously. A computer program was developed to easily adapt all these demands. **Methods:** Microsoft Excel 2010 using Visual Basic for Applications. **Results and Discussion:** The requirements of the guidelines of the GTFCh for control charts in forensic-toxicological analyses were implemented in a computer program. The following main characteristics are available from the chart: day-to-day monitoring, trend estimation, estimation of the measurement uncertainty according to the Guide to the expression of uncertainty in measurement (GUM). Therefore, precision data from control charts were combined with accuracy data derived from proficiency tests. The program was tested using a special set of test data. If applied to own data, a combined measurement uncertainty of 7.5 - 7.6% (68.2% significance) was calculated for THC concentrations ranging from 0.97 - 9.47 ng/ml. The handling of the program will additionally be demonstrated at the symposium. **Conclusion:** The software complies with the current version of the guidelines of the GTFCh and can easily be applied.

1. Introduction

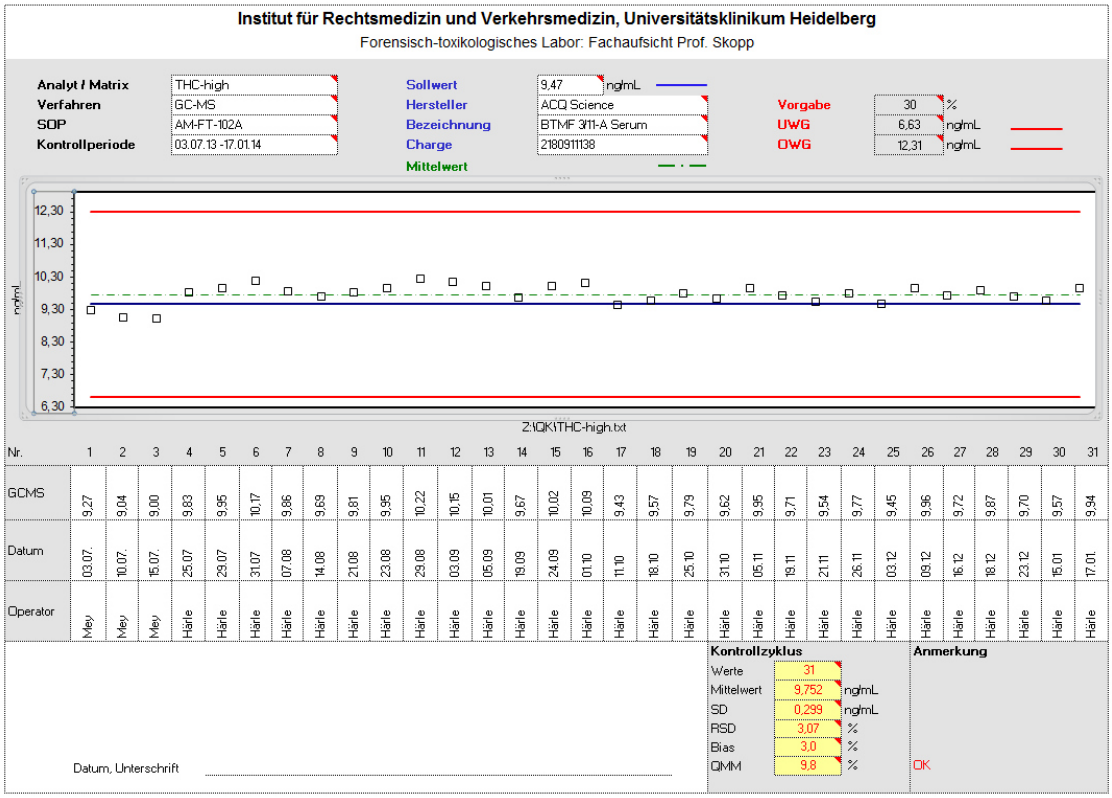
Compliance with the actual version of DIN norm EN ISO/IEC 17025 and the guidelines for quality control in forensic-toxicological analyses of the GTFCh is required for maintenance of accreditation [1-3]. The GTFCh guidelines have been revised in 2011 adopting control charts for internal quality procedures among others. Control charts allow a visual distinction between meaningful change and random variation or „noise“ in analytical assays over time. For the deviation of the measured value from the reference value a maximum of ± 30% (or ± 40% at the detection limit) is tolerable since it contains systematic as well as random errors. The analysis should also be checked if seven successive values increase or drop monotonously. To easily adapt these demands, a computer program was developed.

2. Methods

To easily test these demands, a computer program was developed using Microsoft Excel 2010 and Visual Basic for Applications.

3. Results and Discussion

The requirements of the guidelines of the GTFCh for control charts in forensic-toxicological analyses were implemented in a computer program.



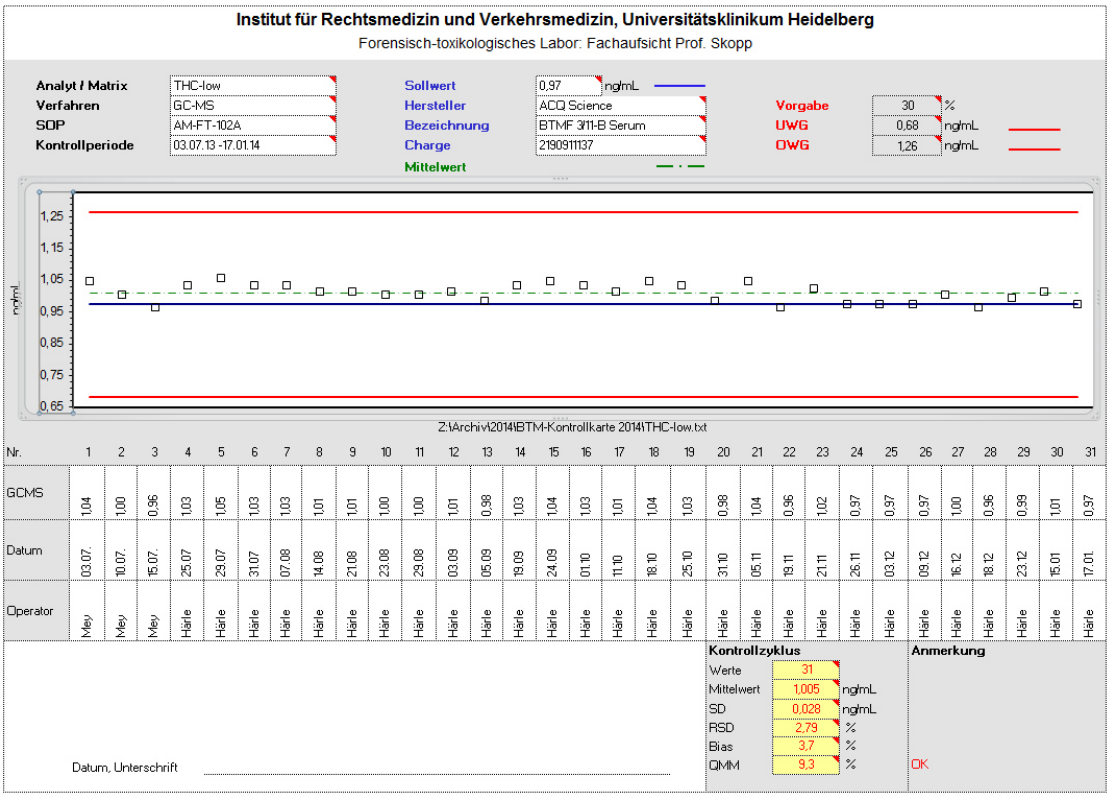
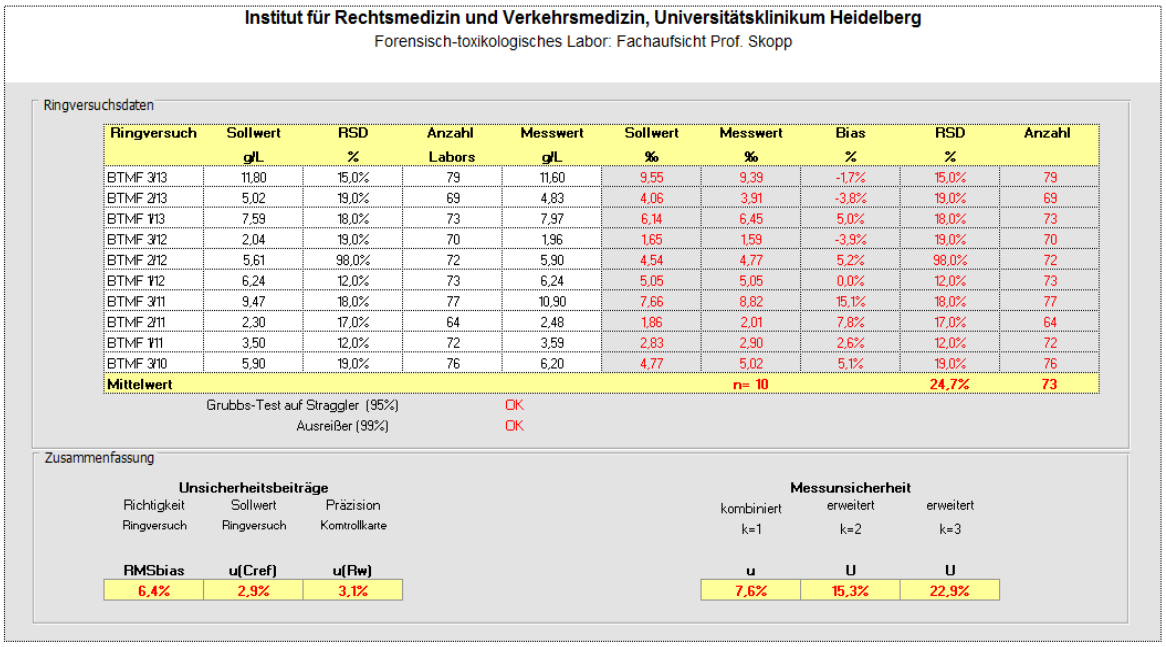


Fig. 1 and 2. Control Charts for BTMF 3/11-A and BTMF 3/11-B.

The following main characteristics are available from the chart: day-to-day monitoring, trend estimation, estimation of the measurement uncertainty according to the Guide to the Expression of Uncertainty in Measurement [1] (Figures 1 and 2).



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Forensisch-toxikologisches Labor: Fachaufsicht Prof. Skopp

Ringversuchsdaten

Ringversuch	Sollwert g/L	RSD %	Anzahl Labors	Messwert g/L	Sollwert ‰	Messwert ‰	Bias %	RSD %	Anzahl
BTMF 3\13	11,80	15,0%	79	11,60	9,55	9,39	-1,7%	15,0%	79
BTMF 2\13	5,02	19,0%	69	4,83	4,06	3,91	-3,8%	19,0%	69
BTMF 1\13	7,59	18,0%	73	7,97	6,14	6,45	5,0%	18,0%	73
BTMF 3\12	2,04	19,0%	70	1,96	1,65	1,59	-3,9%	19,0%	70
BTMF 2\12	5,61	98,0%	72	5,90	4,54	4,77	5,2%	98,0%	72
BTMF 1\12	6,24	12,0%	73	6,24	5,05	5,05	0,0%	12,0%	73
BTMF 3\11	9,47	18,0%	77	10,90	7,66	8,82	15,1%	18,0%	77
BTMF 2\11	2,30	17,0%	64	2,48	1,86	2,01	7,8%	17,0%	64
BTMF 1\11	3,50	12,0%	72	3,59	2,83	2,90	2,6%	12,0%	72
BTMF 3\10	5,90	19,0%	76	6,20	4,77	5,02	5,1%	19,0%	76
Mittelwert						**n= 10**		**24,7%**	**73**

Grubbs-Test auf Straggler (95%) OK
Ausreißer (99%) OK

Zusammenfassung

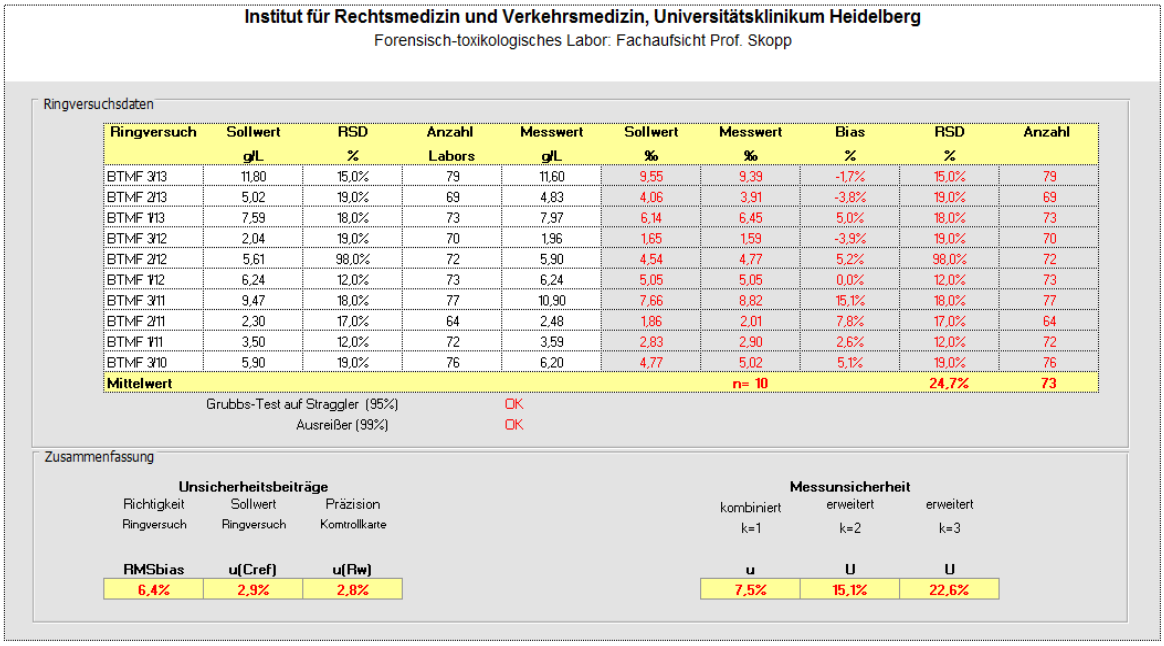
Unsicherheitsbeiträge

Richtigkeit Ringversuch	Sollwert Ringversuch	Präzision Komtrollkarte
RMSbias	**u(Cref)**	**u(Rw)**
6,4%	**2,9%**	**3,1%**

Messunsicherheit

kombiniert k=1	erweitert k=2	erweitert k=3
u	**U**	**U**
7,6%	**15,3%**	**22,9%**

Fig. 3. Combined measurement uncertainty for BTMF 3/11-A.



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Ringversuchsdaten

Ringversuch	Sollwert g/L	RSD %	Anzahl Labors	Messwert g/L	Sollwert ‰	Messwert ‰	Bias %	RSD %	Anzahl
BTMF 3\13	11,80	15,0%	79	11,60	9,55	9,39	-1,7%	15,0%	79
BTMF 2\13	5,02	19,0%	69	4,83	4,06	3,91	-3,8%	19,0%	69
BTMF 1\13	7,59	18,0%	73	7,97	6,14	6,45	5,0%	18,0%	73
BTMF 3\12	2,04	19,0%	70	1,96	1,65	1,59	-3,9%	19,0%	70
BTMF 2\12	5,61	98,0%	72	5,90	4,54	4,77	5,2%	98,0%	72
BTMF 1\12	6,24	12,0%	73	6,24	5,05	5,05	0,0%	12,0%	73
BTMF 3\11	9,47	18,0%	77	10,90	7,66	8,82	15,1%	18,0%	77
BTMF 2\11	2,30	17,0%	64	2,48	1,86	2,01	7,8%	17,0%	64
BTMF 1\11	3,50	12,0%	72	3,59	2,83	2,90	2,6%	12,0%	72
BTMF 3\10	5,90	19,0%	76	6,20	4,77	5,02	5,1%	19,0%	76
Mittelwert						**n= 10**		**24,7%**	**73**

Grubbs-Test auf Straggler (95%) OK
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Zusammenfassung

Unsicherheitsbeiträge

Richtigkeit Ringversuch	Sollwert Ringversuch	Präzision Komtrollkarte
RMSbias	**u(Cref)**	**u(Rw)**
6,4%	**2,9%**	**2,8%**

Messunsicherheit

kombiniert k=1	erweitert k=2	erweitert k=3
u	**U**	**U**
7,5%	**15,1%**	**22,6%**

Fig. 4. Combined measurement uncertainty for BTMF 3/11-B.

Therefore, precision data from control charts were combined with accuracy data derived from proficiency tests. If applied to these data, a combined measurement uncertainty of 7.5 to 7.6% (68.2% significance) was calculated for THC serum concentrations at 9.47 and 0.97 ng/mL (Figures 3 and 4, Table 1).

For non-forensic samples, the root mean square of the error of measurement (QMM) is also specified [4]. The handling of the program was demonstrated at the symposium. For members, a download will be available on the GTFCh-homepage (www.gtfch.org).

Tab. 1. Estimation of the combined measurement uncertainty (MU) using precision data from control charts (n=31) and accuracy data derived from proficiency tests (n=10). The root mean square of the error of measurement (QMM) is also specified.

Quality control sample	BTMF 3/11-A Serum	BTMF 3/11-B Serum
Reference value	9,47 ng/mL	0,97 ng/mL
Mean value	9,75 ng/mL	1,00 ng/mL
Bias	3,0 %	3,7 %
QMM		
k=1	4,3 %	4,6 %
k=2	6,9 %	6,8 %
k=3	9,8 %	9,3 %
MU		
k=1	7,6 %	7,5 %
k=2	15,3 %	15,1 %
k=3	22,9 %	22,6 %

4. Conclusion

The software complies with the current version of the guidelines of the GTFCh and can easily be applied.

5. References

[1] Guide to the Expression of Uncertainty in Measurement (Geneva, Switzerland: International Organisation for Standardisation), ISBN 92-67-10188-9, 1995.
[2] Nordtest Technical Report 537, Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories, 2003.
[3] Richtlinien der GTFCh zur Qualitätssicherung bei forensisch-toxikologischen Untersuchungen. Toxichem Krimtech 2009;76:42-176.
[4] Macdonald Rainer. J Lab Med 2006;30:111–117.