East African Community

Final Technical Report
of the EAC – IC in Temperature Metrology

1st Interlab Comparison with Liquid-in-Glass Thermometers

Within the framework of the PTB-EAC project
“Establishment of a regional SQMT-architecture in the EAC”

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

Eingetragen unter
HRB 1468 Lünen
Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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1. INTRODUCTION

This report describes the results of the first interlab ring comparison for liquid in glass thermometer calibration capabilities from 0 to 50 deg. Cent (°C).

The participating laboratories were (names in first-/last-name):

KEBS	Nairobi, Kenya (Ref. Lab.)	Joel Kioko, Wilson Egadwa
TBS	Dar es Salaam, Tanzania	Juliua Kisamo, Edna Ndumbaro
UNBS	Kampala, Uganda	Lemeriga Yasin, Simon Rwashana,
QSAE	Addis Ababa, Ethiopia	Wondwosen Fisseha, Fistum Tesfaye

The German PTB (Mr. Stefan Wallerath & team) and the technical consultant, Mr. Reinhard Klemm (Centrocal GmbH, Werne, Germany) thank all participated laboratories and persons for their co-operation. Special thanks are directed to the reference lab (KEBS) for their organization, logistics and accomplishment in doing an interlab ring comparison for the very first time.

The EAC-IC was not started as a contest between the participating labs. No award will be presented to the "winner" because all are winners. The above given order of labs reflects the order of cycling the artifact(s) rather than any other relevancy.

The results are presented in an open form rather than to make them anonymous. Any of the participants will get all informations from any other lab.

During a workshop, held at QSAE in Addis Ababa/Ethiopia, from April 7th to April 11th, 2008 the results were presented and discussed. The German PTB as well as the consultant thank QSAE for organization and hospitality.

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2. ABSTRACT

The EAC-IC was done within a reasonable amount of time. The returned liquid-in-glass thermometers were in a good condition. No break occurred and stability of the artifacts was achieved. The recalibration at the original lab in Germany produced expected results without any surprise.

The results given by the labs were in the form of calibration certificates. The formal layout was acceptable. No lab added a detailed uncertainty budget – as originally requested.

An example or guide for a detailed uncertainty budget was developed by the consultant and sent to the labs. By return detailed budgets were made available in an acceptable form. Guide and individual budgets are given as appendices to this report.

The final elaboration, done by the consultant, is added to this report in both tabulary and graphical form. The report is written in " Word® ", graphics and tables in " Excel® " and the PDF - documents are made by using " Acrobat Professional ® ".

No lab achieved at all temperatures an E_n – value of 1,0 or below. Each lab showed results which are partly not consistently within the queue of the related measurements.

The workshop held in Addis Ababa is the "toolkit" for the following interlab comparison circles with electrical thermometers.

3. RESULTS

3.1 Overview

The liquid-in-glass (LIG) thermometers were calibrated by a German DKD - lab named "Hessische Eichdirektion Darmstadt" – further abbreviated as HED – before and after EAC-IC. The thermometers have a mark for the immersion depth (76 mm). Due to the fact, that all labs in the EAC-IC have calibrated the artifacts at full immersion, the final calibration at HED was performed in both depths – 76 mm and full.
KEBS as the leading lab for the EAC-IC did the first calibration and stated the routines for the calibrations in the consecutive labs TBS, UNBS and QSAE. Unfortunately, KEBS did no calibration at the end of the circle to estimate stability and integrity of the artifact.

To have a clear understanding and give a good support to the labs for further interlab comparisons all results are referenced to KEBS as well as to HED in 76 mm and full immersion depth.

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Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
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Fax: +49 (0) 23 89 - 409 - 80
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3.2 Table 1: Overview of results

Lab Name	Immersion	Target Temp. in °C	Actual temp, in ˚C	Indicated Temp in ˚C	Corr ˚C	Uncert.(k=2) in mK	LV-RV in ˚C	En KEBS - Lab	LV-HED in ˚C 76 mm	En HED - Lab	LV-HED total imm. in ˚C	En HED - Lab	
KEBS	TOTAL	0	0,039	0,000	0,039	0,060	0,000	0,00	0,059	0,88	0,039	0,62	Below BMC !!!
	TOTAL	20	20,143	20,040	0,103	0,060	0,000	0,00	0,113	1,68	0,083	1,31	Below BMC !!!
	TOTAL	30	29,986	30,000	-0,014	0,060	0,000	0,00	-0,034	-0,51	0,036	0,57	Below BMC !!!
	TOTAL	40	40,015	40,200	-0,185	0,060	0,000	0,00	-0,235	-3,50	-0,075	-1,19	Below BMC !!!
	TOTAL	50	49,854	50,100	-0,246	0,060	0,000	0,00	-0,266	-3,97	0,004	0,06	Below BMC !!!
UNBS	TOTAL	0	0,000	-0,050	0,050	0,160	0,010	0,06	0,070	0,43	0,050	0,31	
	TOTAL	20	20,000	19,920	0,080	0,160	-0,020	-0,13	0,090	0,55	0,060	0,37	
	TOTAL	30	30,100	29,910	0,190	0,160	0,200	1,19	0,170	1,04	0,240	1,49	
	TOTAL	40	39,950	39,900	0,050	0,160	0,240	1,38	0,000	0,00	0,160	0,99	
	TOTAL	50	50,100	49,900	0,200	0,160	0,450	2,61	0,180	1,11	0,450	2,79	
TBS	TOTAL	0	0,000	0,070	-0,070	0,100	-0,109	-0,93	-0,050	-0,48	-0,070	-0,69	
	TOTAL	20	20,000	19,990	0,010	0,100	-0,093	-0,80	0,020	0,19	-0,010	-0,10	
	TOTAL	30	30,000	30,100	-0,100	0,100	-0,086	-0,74	-0,120	-1,15	-0,050	-0,49	
	TOTAL	40	40,000	40,210	-0,210	0,100	-0,025	-0,21	-0,260	-2,49	-0,100	-0,98	
	TOTAL	50	50,000	50,220	-0,220	0,100	0,026	0,22	-0,240	-2,30	0,030	0,29	
QSAE	TOTAL	0	0,001	-0,050	0,051	0,025	0,012	0,18	0,071	1,82	0,051	1,59	
	TOTAL	20	20,103	20,071	0,032	0,025	-0,071	-1,09	0,042	1,08	0,012	0,37	
	TOTAL	30	30,045	30,087	-0,042	0,026	-0,028	-0,43	-0,062	-1,56	0,008	0,24	
	TOTAL	40	40,059	40,156	-0,097	0,028	0,088	1,33	-0,147	-3,58	0,013	0,38	Below BMC !!!
	TOTAL	50	50,073	50,340	-0,267	0,028	-0,021	-0,32	-0,287	-6,99	-0,017	-0,49	Below BMC !!!

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Temperatur

CENTROCAL GmbH
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Fax: +49 (0) 23 89 - 409 - 80
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Stadtsparkasse Werne
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HED 76 mm	76 mm	0	-0,020	-0,020	0,030	0,059	HED-KEBS		
	76 mm	20	0,020	-0,010	0,030	0,113	HED-KEBS		
	76 mm	30	0,010	0,020	0,030	-0,034	HED-KEBS		
	76 mm	40	0,040	0,050	0,030	-0,235	HED-KEBS		
	76 mm	50	0,020	0,020	0,030	-0,266	HED-KEBS		
HED TOTAL	TOTAL	0	---	0,000	0,020			0,039	HED-KEBS
	TOTAL	20	---	0,020	0,020			0,083	HED-KEBS
	TOTAL	30	---	-0,050	0,020			0,036	HED-KEBS
	TOTAL	40	---	-0,110	0,020			-0,075	HED-KEBS
	TOTAL	50	---	-0,250	0,020			0,004	HED-KEBS

LV = Lab-Value
UNBS, TBS, QSAE, KEBS

RV = Reference Value
KEBS or HED

HED = Hess. Eichdirektion
German Ref. Lab.

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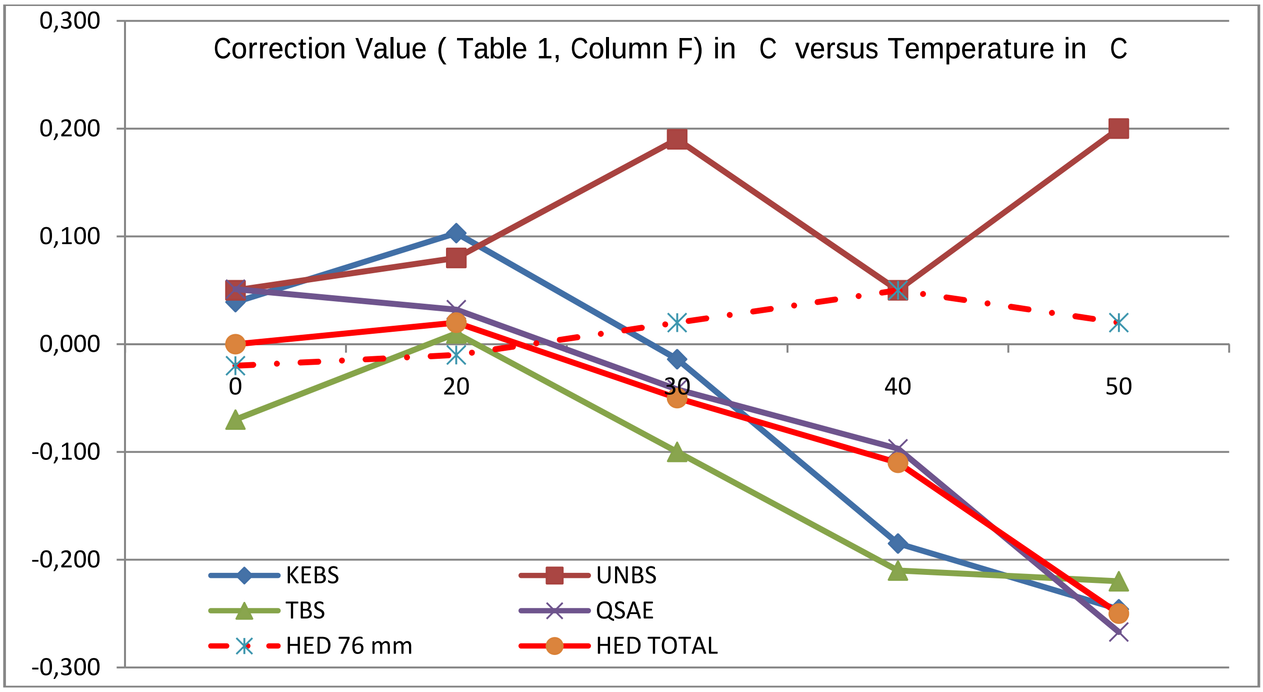
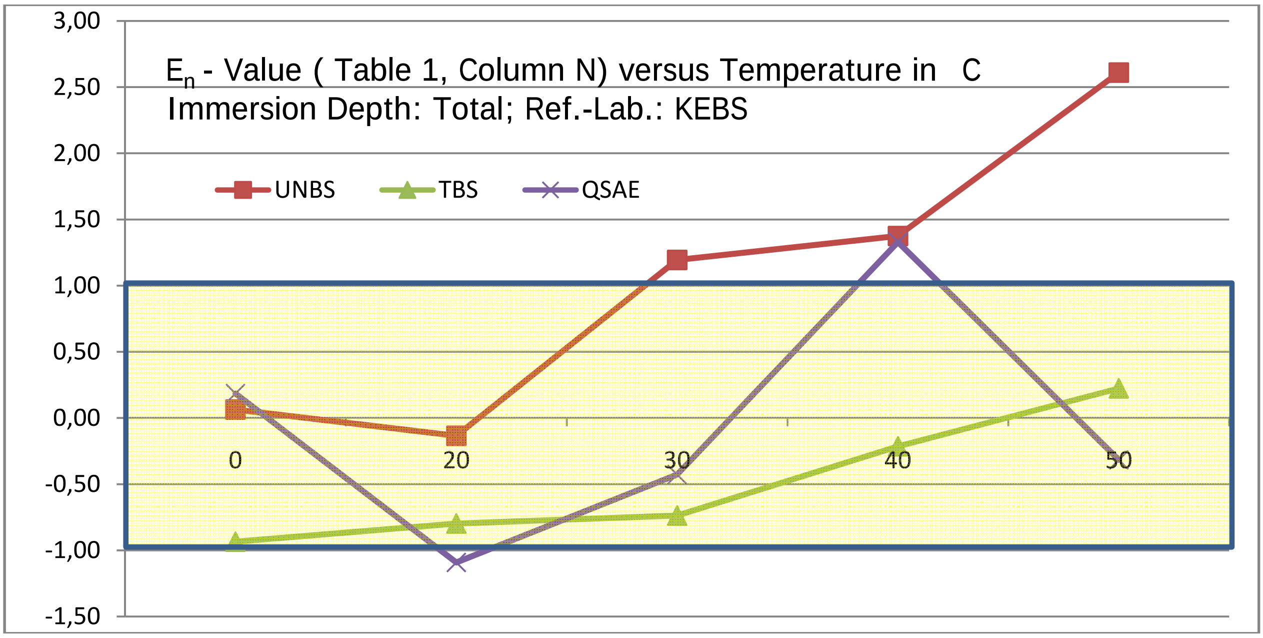
3.3 Table 2: Graphical display of correction values versus temperature



3.4 Table 3: Graphical display of E_n vs temp., ref.-lab.: KEBS



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Lohstraße 2, D-59368 Werne
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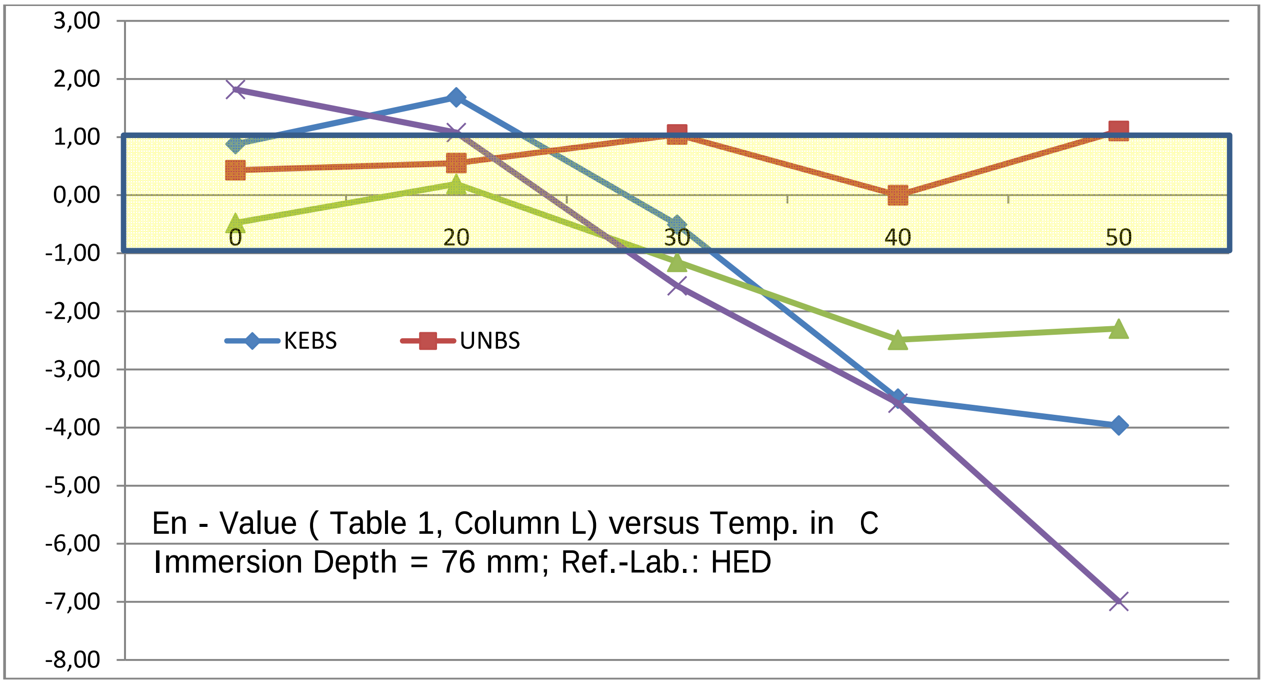
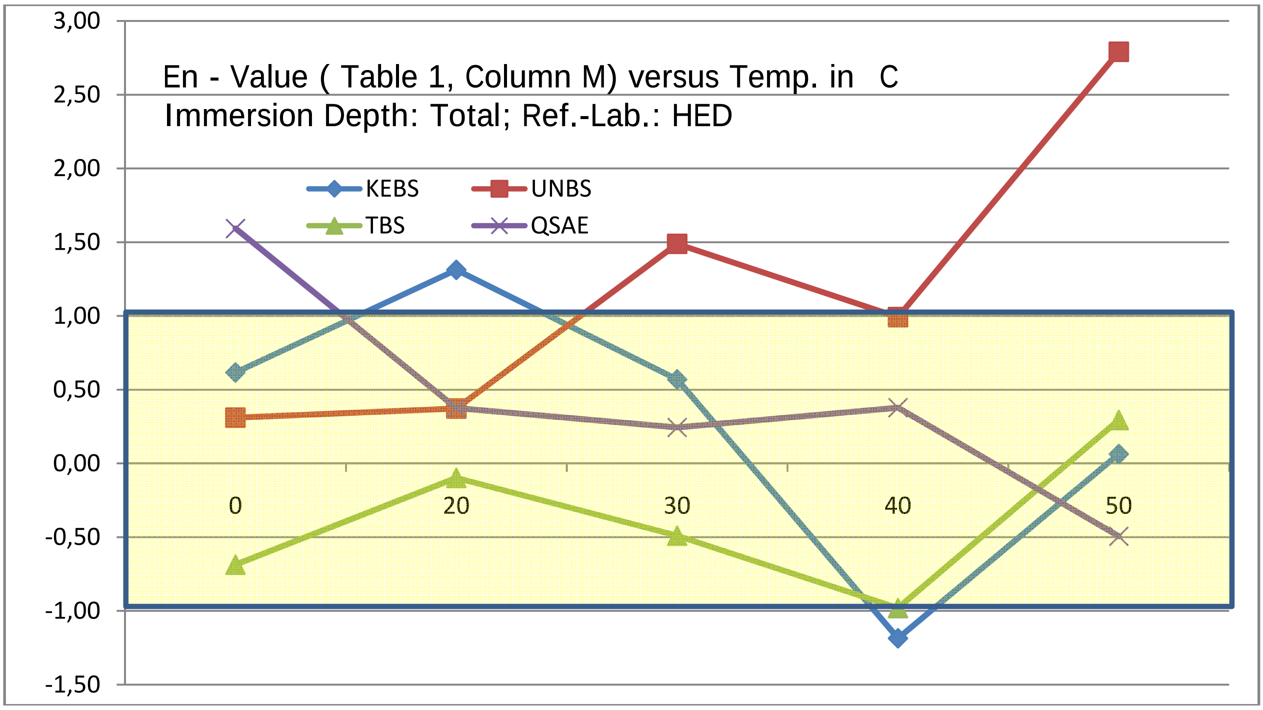
3.5 Table 4: Graphical display of E_n vs temp., ref.-lab.: HED (76 mm)



3.6 Table 5: Graphical display of E_n vs temp., ref.-lab.: HED (total)



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Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
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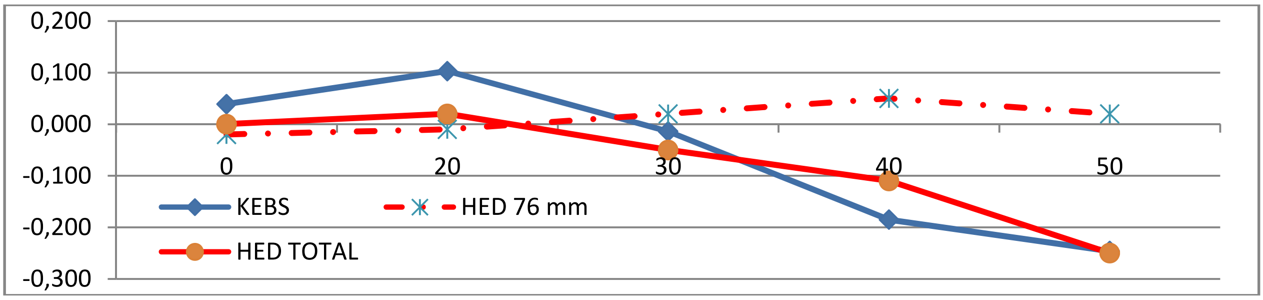
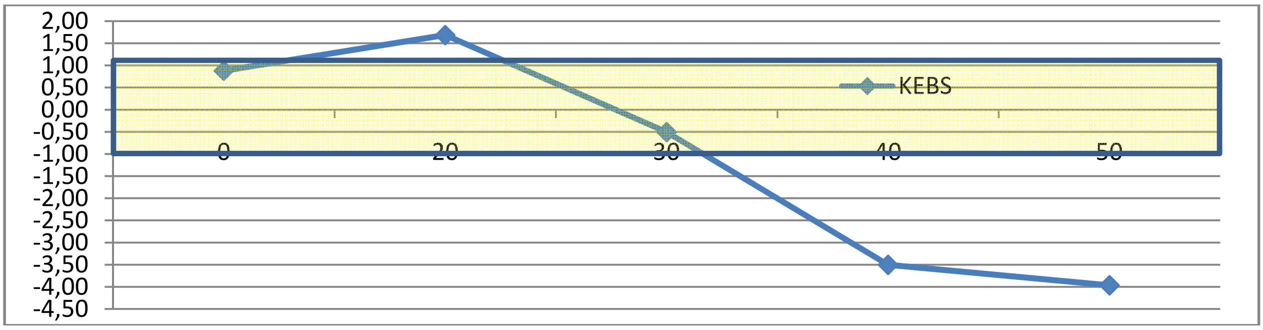
4. DETAILED RESULTS PER PARTICIPATED LABORATORY

4.1 KEBS Kenya Bureau of Standards– reference Lab

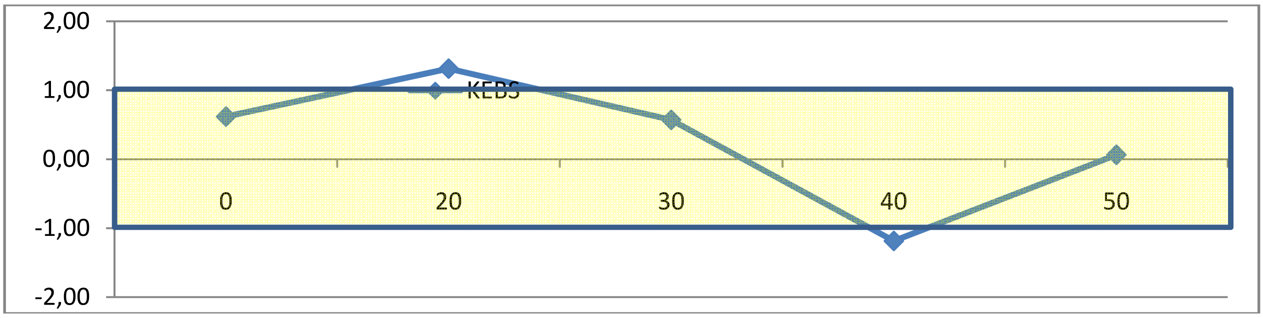
4.1.1 Table 6: Correction Value (Table 1, Column F) in °C versus Temperature in °C



4.1.2 Table 7: En - Value (Table 1, Column K) versus Temperature in °C Immersion Depth = 76 mm; Ref.-Lab.: HED



4.1.3 Table 8: En - Value (Table 1, Column M) versus Temperature in °C Immersion Depth: Total; Ref.-Lab.: HED



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Lohstraße 2, D-59368 Werne
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4.1.4 Comments

The graphic of the absolute results as per table 6 above look not so bad. The trend is exactly the same as of HED. It seems to the consultant, that there are no major deficiencies in handling, hardware and calibration process.

It seems on the other hand, that the estimation of the uncertainty is a little bit too progressive. Some more conservative estimation of accompanying parameters may lead to better results. Table 8 reflects this opinion.

Furthermore, it's hard to understand, that the E_n – values at 20 and 40 °C are both outside ± 1,0 – on the positive ***and*** negative side. One possible explanation could be, that the waiting time for thermal stabilization of the liquid bath was too short – but other influences are also possible.

In future IC's, initiated by PTB together with DKD, some more care should be spend on the matter of fact, that in no case the stated uncertainty is below the accredit uncertainty. The figures given in the accreditation reflect the best measurement capability (BMC) and should not fall short.

Table 7 is given for reference only.

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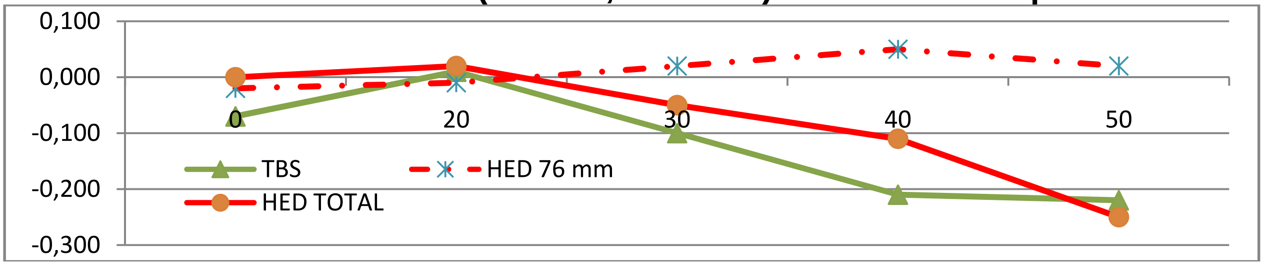
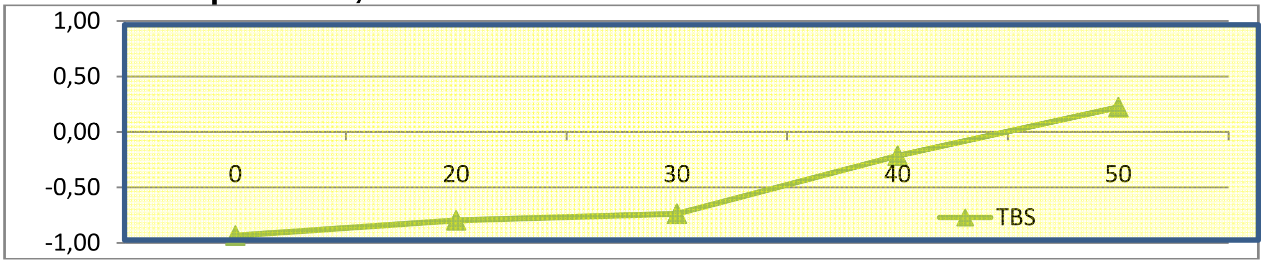
4.2 TBS Tanzania Bureau of Standards

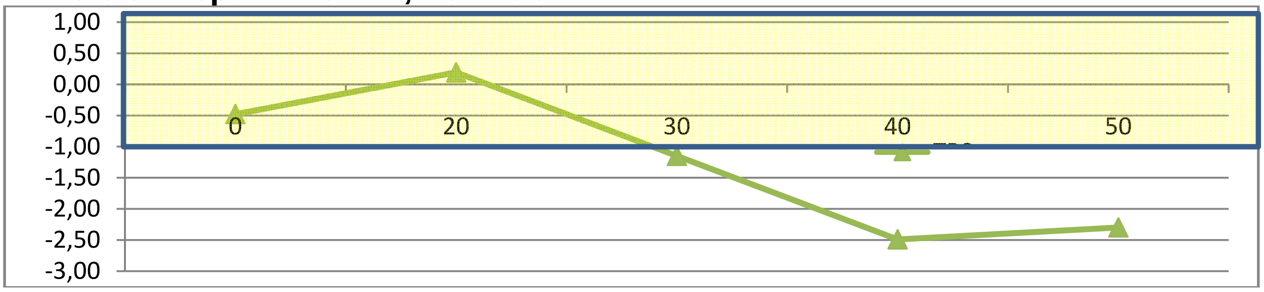
4.2.1 Table 9: Correction Value (Table 1, Column F) in °C versus Temperature in °C



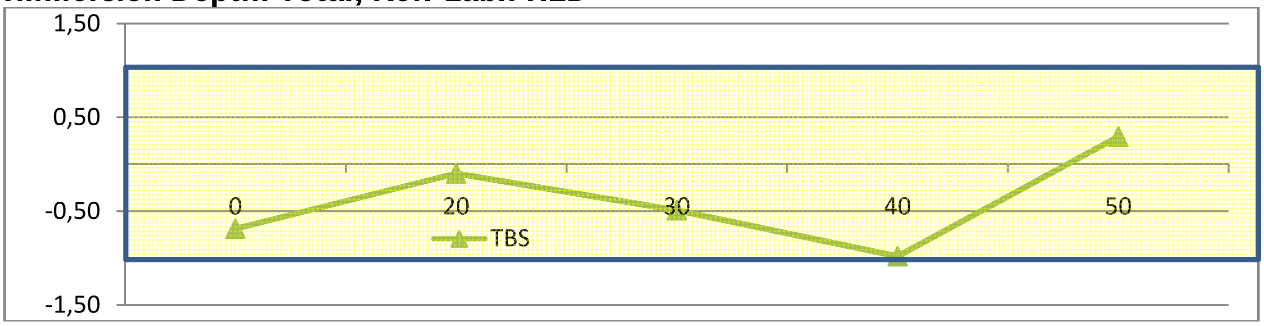
4.2.2 Table 10: E_n - Value (Table 1, Column N) versus Temperature in °C
Immersion Depth: Total; Ref.-Lab.: KEBS



4.2.3 Table 11: E_n - Value (Table 1, Column K) versus Temperature in °C
Immersion Depth = 76 mm; Ref.-Lab.: HED



4.2.4 Table 12: E_n - Value (Table 1, Column M) versus Temperature in °C
Immersion Depth: Total; Ref.-Lab.: HED



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Lohstraße 2, D-59368 Werne
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4.2.5 Comments

The graphic of the absolute results as per table 9 above looks quite good. The trend is exactly the same as of HED. It seems to the consultant, that there are no deficiencies in handling, hardware and calibration process.

Table 10: It seems on the other hand, that the estimation of the uncertainty compared with HED is a little bit too progressive. But there is no doubt, that the result is inside En = ± 1,0 and therefore excellent.

Table 12: It's hard to understand, that the E_n – values at 20 and 40 °C are both inside ± 1,0 – but are outside of the "trend". One possible explanation could be that the waiting time for thermal stabilization of the liquid bath was too short – but other influences are also possible.

Table 10: Compared with the lead lab (KEBS) the results shows best consistency.

Table 11 is given for reference only.

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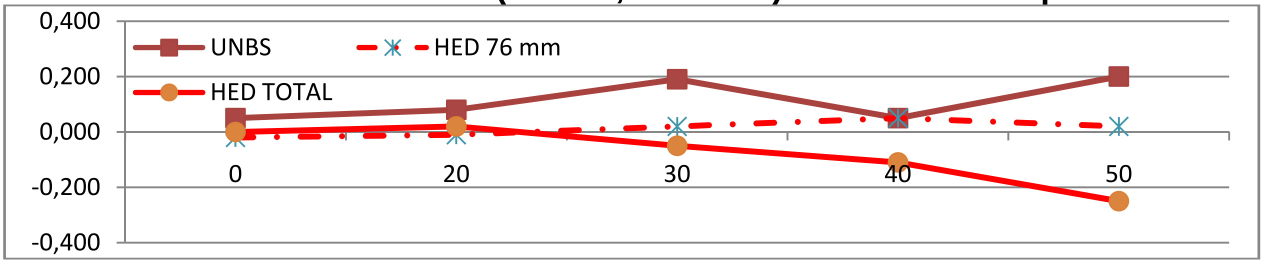
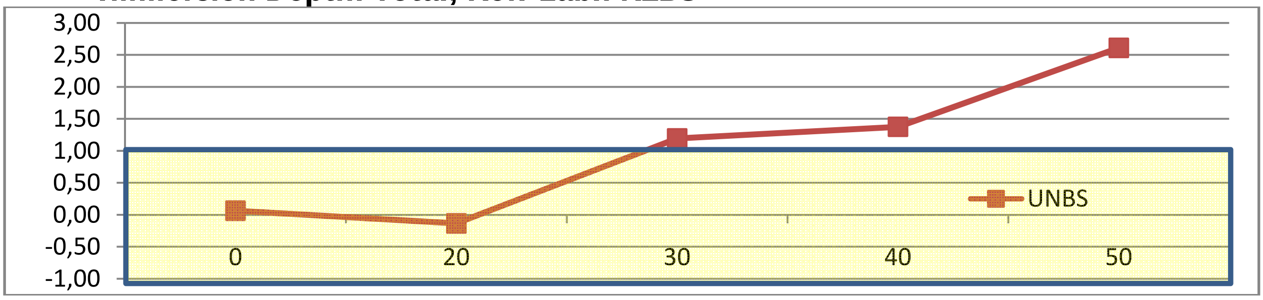
4.3 UNBS Uganda National Bureau of Standards

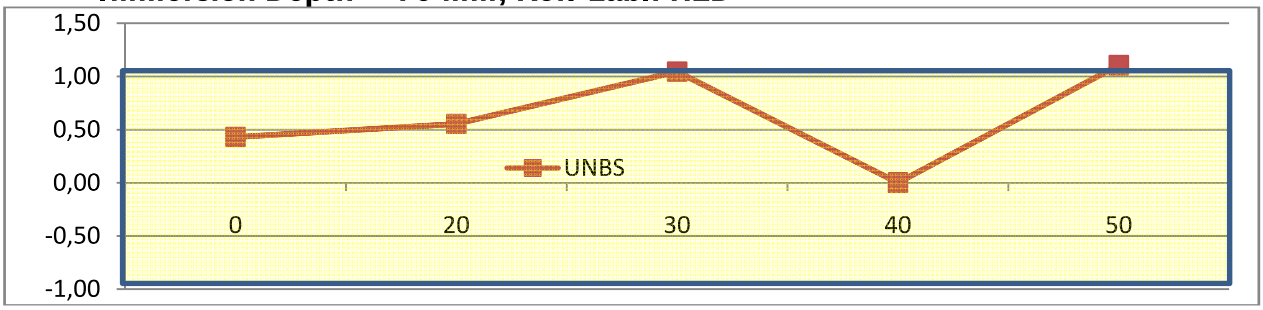
4.3.1 Table 13: Correction Value (Table 1, Column F) in °C versus Temperature in °C



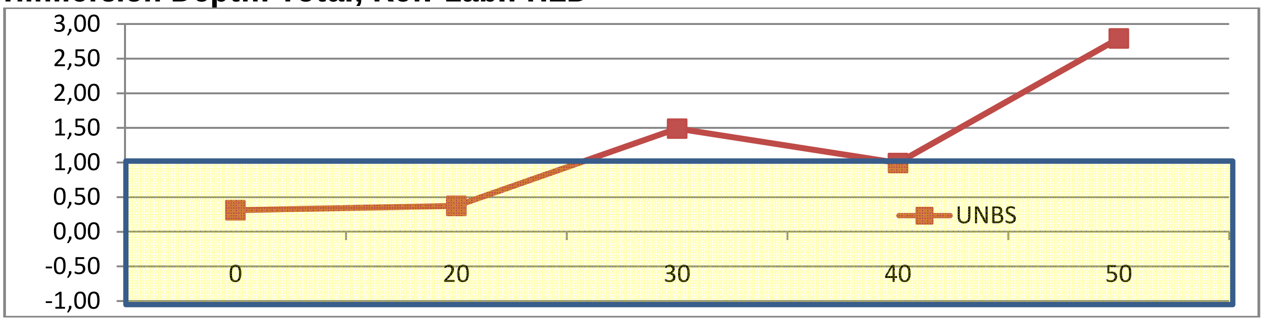
4.3.2 Table 14: E_n - Value (Table 1, Column N) versus Temperature in °C Immersion Depth: Total; Ref.-Lab.: KEBS



4.3.3 Table 15: E_n - Value (Table 1, Column K) versus Temperature in °C Immersion Depth = 76 mm; Ref.-Lab.: HED



4.3.4 Table 16: E_n - Value (Table 1, Column M) versus Temperature in °C Immersion Depth: Total; Ref.-Lab.: HED



Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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4.3.5 Comments

The graphic of the absolute results as per table 13 above looks not so bad. The trend is quite comparable as of HED with 76 mm immersion depth. See also table 15. It looks like that UNBS did the calibration at a depth of 76 mm rather than total immersed.

It seems to the consultant, that there might be some little deficiencies in handling, hardware and/or calibration process or they misinterpreted the routines given by KEBS.

Table 14: It is hardly to understand that the first two temperatures show an E_n – value close to zero while the remaining three temperatures are outside the + 1,0 borderline. Table 16 shows nearly the same result which supports the estimation given in clause 1 above.

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
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HRB 1468 Lünen
Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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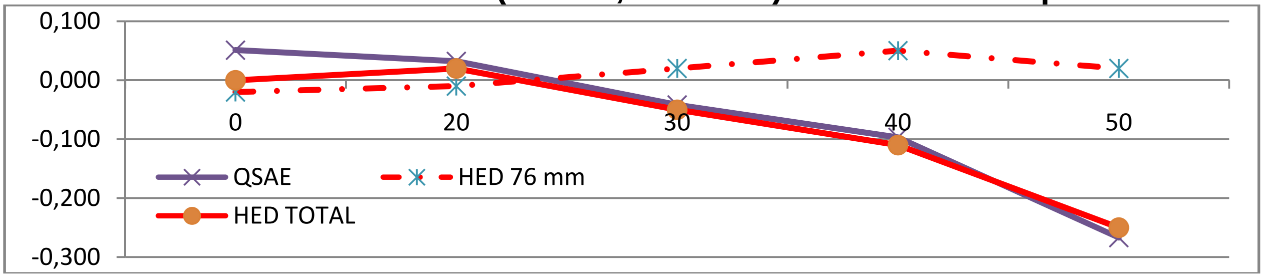
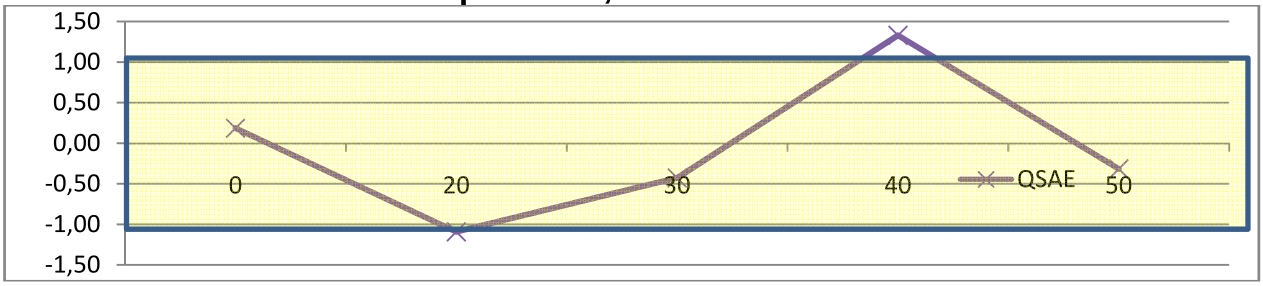
4.4 QSAE Quality and Standards Authority of Ethiopia

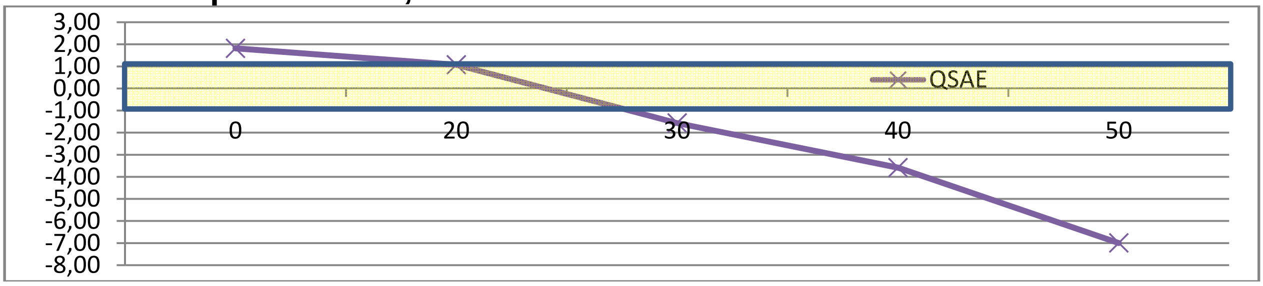
4.4.1 Table 17: Correction Value (Table 1, Column F) in °C versus Temperature in °C



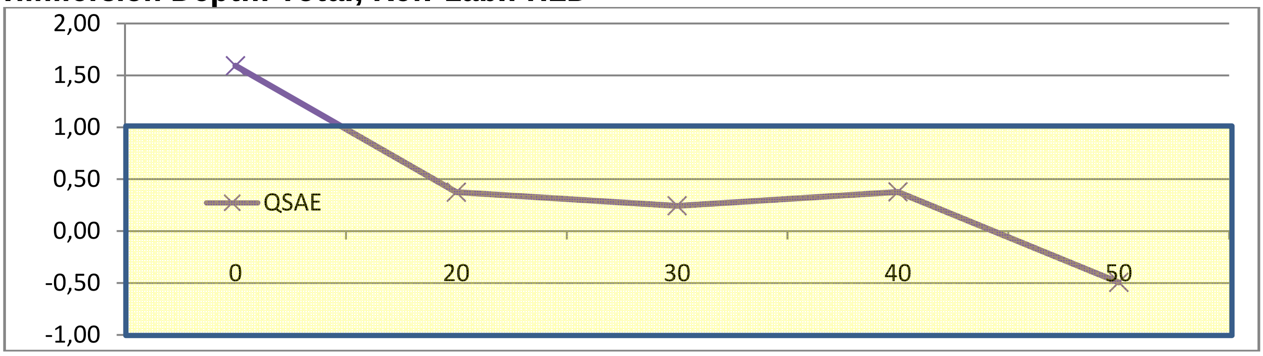
4.4.2 Table 18: E_n - Value (Table 1, Column N) versus Temperature in °C
Immersion Depth: Total; Ref.-Lab.: KEBS



4.4.3 Table 19: E_n - Value (Table 1, Column L) versus Temperature in °C
Immersion Depth = 76 mm; Ref.-Lab.: HED



4.4.4 Table 20: E_n - Value (Table 1, Column N) versus Temperature in °C
Immersion Depth: Total; Ref.-Lab.: HED



Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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4.4.5 Comments

The graphic of the absolute results as per table 17 above look very good. The trend and the values extremely close to them of HED. It seems to the consultant, that there are no major deficiencies in handling, hardware and calibration process.

It seems on the other hand, that the estimation of the uncertainty is a little bit too progressive. Some more conservative estimation of accompanying parameters may lead to better results. Table 18 reflects this opinion.

Furthermore, it's hard to understand, that the E_n – values at 20 and 40 °C are outside or directly on the borderline ± 1,0 – on the positive ***and*** negative side. One possible explanation could be, that the waiting time for thermal stabilization of the liquid bath was too short – but other influences are also possible.

In future IC's, initiated by PTB together with DKD, some more care should be spend on the matter of fact, that in no case the stated uncertainty is below the accredit uncertainty. The figures given in the accreditation reflect the best measurement capability (BMC) and should not fall short.

Table 19 is given for reference only.

The results given in table 20 show a high degree of professionalism in performing calibrations compared with laboratories long time established in the German DKD. On the other hand, the given value at 0 °C in table 20 is not understandable. Some investigations at QSAE may clarify this circumstances.

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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HRB 1468 Lünen
Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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5. FINAL CONCLUSION

It has to be mentioned that the consultant sees some points where difficulties appeared:

The direct and fast communication within the participating labs should be upgraded.

Transportation of the artifacts from one lab to the next might cause problems.

The tracebility status of the instruments used in the labs should be clear.

The uncertainty budgets were not originally included in the reports of calibration

The calibration procedures used were not clearly described

The procedure of estimating the measurement uncertainty needs to be harmonized

In general, the results under the above and the premise of the first EAC-IC are truly satisfactionary. Partly they are acceptable; partly some “fine tuning” is needed. But all labs did a professional work and demonstrated a very high degree of engagement.

The initial sense while choosing LiG – thermometers for the first EAC-IC was to get an appraisal of capabilities from the participating labs. Beside the less robustness (its without any doubt!) the calibration is quite easy and might be a part of the dayly work.

The next EAC-IC will be done with two industrial platinum resistance thermometers (IPRT’s) with a base resistance at 0 °C of 100 Ohm. The thermometrers are in the accuracy class B according to IEC 751. Handling and transportation will be much easier.
The consultant provides the two calibrated artifacts as well as detailed calibration procedures. See appendix 3 of this report. **The pilot lab will be QSAE, Addis Ababa, Ethiopia.**

Most likely, based on the experiences of the actual finished EAC-IC the next IC will provide better results and a further step onward in terms of personnel skills, lab procedures, uncertainty budget and communication.

Werne (Germany) April 2nd, 2008

R. Klemm, Consultant

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
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Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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APPENDIX 1: Uncertainty – Budged

Analysis of measurement uncertainty in calibration of liquid-in-glass thermometers

The following uncertainty analysis for liquid-in-glass thermometers is given as an example for the determination of measurement uncertainty. It must be clearly stated, that this document is a guide-line rather than a "cooking book". Special circumstances like measurement ensemble, available hardware equipment, ambient conditions, personnel, …….. have to be taken in consideration while estimating the individual uncertainty budget.
The thermometer in this particular ring comparison (EAC - Interlab Comparison → EAC - IC) was originally calibrated with an immersion depth of 76 mm in a stirred liquid bath. After its return to Germany calibration is performed in both – full immersion and 76 mm immersion depth.
In any case, it is useful to spend some words into the evaluation of a measurement uncertainty budget. Basically, only four steps are to be done in a systematical order as following:

1st step: Estimation of the uncertainty associated with the temperature of the liquid bath
2nd step: Estimation of the uncertainty associated with the unit under test
3rd step: Combination of the different uncertainties and parameters
4th step: Result of the calibration together with uncertainty, probability level & expansion factor.
It is strongly recommended to do the a.m. steps in a tabulary form – either by hand on paper or (better) in a PC - based worksheet - i.e. Microsoft® EXCEL® or equivalent.

1st step: Estimation of the uncertainty associated with the temperature of the liquid bath → Table 1

Temperature range 0 ... 100 °C

Model equitation: $t_x = t_N + \sigma t_D + \sigma t_{Sh} + \sigma t_{HD} + \sigma t_R + \sigma t_{Br} + \sigma t_{Hom} + \sigma t_{Stab} + \sigma t_{Res}$

Dim.	Item	Estimate	UOM	Uncert	UOM	Distri-bution	Divisor	Sensit.	UOM	u_i (t)	UOM	(u_i (t))^2
t_x	Average of measured temp.	50,000	°C		mK	Normal	2	1	---		mK	
σt_D	Drift of standard(s)				mK	Rect.	1,73205	1	---		mK	
σt_{Sh}	Self-heating of standard(s)				mK	Rect.	1,73205	1	---		mK	
σt_{HD}	Heat dissipation of standard(s)				mK	Rect.	1,73205	1	---		mK	
σt_R	Uncertainty of std. Resistor				mΩ	Normal	2	XX,X	K/Ω		mK	
σt_{Br}	Bridge or meter uncertainty				mΩ	Normal	2	XX,X	K/Ω		mK	
σt_{Hom}	Homogeneity of temperature device (Liquid Bath)				mK	Rect.	1,73205	1	---		mK	
σt_{Stab}	Stability of temp. device				mK	Rect.	1,73205	1	---		mK	
σt_{Res}	Resolution of Bridge or indicating meter				mΩ mK	Rect.	1,73205	XX.X			mK	
t_N	**Temperature of tempering device**	**50,000**	**°C**						**±**		**mK**	

Messgröße DKD-K-09701 Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 5552-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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Geschäftsführer:
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Agenda to table 1:

Numeric values given either by certificate or best available estimation are to be inserted into the green column named "Uncert." The sensitivity has to be calculated in accordance to the UOM (Units Of Measure → mK, °C, mΩ or ......) given by uncertainty's UOM.

t_x Average of measured temperature of the liquid bath in case two standards are used. In case of one standard only, its particular output is used

σt_D Drift of standard(s) since last calibration. This is an estimation only. A guideline could be the history of the standard(s) in the recent repeated calibrations.

σt_{Sh} In case SPRT's are used, the self heating due to the excitation current must be calculated.

σt_{HD} The heat transfer of the standard to / from the ambient must be estimated.

stR The uncertainty of the external standard resistor of the bridge, derivated from its calibration certificate, must be calculated. In case a direct indicating instrument rather than a bridge is used, this parameter is set to zero.

σt_{Br} The uncertainty of the bridge or the indicating instrument from its calibration certificate is used here. In case of bridge in mΩ, in other cases in mK. The sensitivity has to be set in accordance to the UOM of the uncertainty

σt_{Hom} Homogeneity of the tempering device (f.e. liquid bath) → difference in temperature between the position of the standard and the position of the unit under calibration (UUC). This parameter can't be zero!!

σt_{Stab} Stability of the tempering device (f.e. liquid bath) → Stability must be measured and calculated over a period of 20 times measuring time or 10 minutes, whatever is longer.

σt_{Res} Resolution of the used bridge, digital multi-meter or digital indicator. Mostly expressed as LSD → Least Significant Digit. If an analog instrument is used, f.e. a liquid-in-glass thermometer, the resolution is a function of scale division and spacing as well as human parameters like using of a magnifying glass or not and training.

Blue column: $u_i(t)$ = Uncertainty * Divisor * Sensitivity → Calculated from the column-values

Pink column: Simply the square of adjacent column

Purple line: Result = Square root of the sum of the pink column. This is the standard uncertainty with expansion factor **k = 1 → probability level ~ 68 %**

Messgröße DKD-K-09701 Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 5552-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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HRB 1468 Lünen
Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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2nd step: Estimation of the uncertainty associated with the unit under calibration

The UUC in is a liquid-in-glass thermometer. To observe the specific characteristics it is therefore necessarily to divide this step into two sub steps:

Step 2.1: Correction of the ice-point indication

The correction C_0 of the ice-point is given by following equitation:

$$\mathbf{C_0 = t_{Ice} - t_{Ind.Ice} - \sigma t_{Th}}$$

with

t_{Ice} - "True" temperature of the ice-point

$t_{Ind.Ice}$ - Indication of the UUC at ice-point

σt_{Th} - Correction of indication due to different response times between standard and UUC at ice-point (short-time stability)

Step 2.2: Reduced correction for indication at calibration temperature

The reduced correction C_R of the indication at the calibration temperature is given as following:

$$\mathbf{C_R = t_N - t_{Ind} - C_0 - C_F - C_{Hys}}$$

with

t_N - Temperature of tempering device (f.e. stirred liquid bath from step 1)

t_{Ind} - Indication of UUC at calibration temperature

C_0 - Correction of ice-point indication from step 2.1 above

C_F - Correction of filament to compensate different linear expansion coefficients between liquid, capillary and scale or glass body.

C_{Hys} - Correction of hysteresis between indication from upscale and downscale to the same temperature of the tempering device. An estimated value by experience or an average value of repeated tests. With analog instruments, the hysteresis is never zero!

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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HRB 1468 Lünen
Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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3rd step: Combination of the different uncertainties and parameters

The calculations according to step 1, 2.1 and 2.2 together form the overall model for the uncertainty analysis. The complete situation is given in the table 2 below

Table 2

Item	Source of uncertainty	Value	Standard Uncer-tainty	Distri-bution	Sensitivity Coefficient	Uncertainty
t_N	Temperature of tempe-ring device	XX,XXX °C	X,X mK	Normal	1	X,X mK
t_{Ind}	Temperature indica-tion at calibr. point	XX,XXX °C	XX mK	Normal	-1	-XX mK
t_{Ice}	Ice point realisation	X K	X,X mK	Rect.	-1	-X,X mK
$t_{Ind.Ice}$	Temperature indication at ice-point	X,XX K	X mK	Normal	1	X mK
σt_{Th}	Short term stability	X K	X,X mK	Rect.	-1	-X,X mK
C_F	Filament correction	X,XX K	XX,X mK	Rect.	-1	-XX,X mK
C_{Hys}	Hysteresis correction	X K	X,X mK		-1	-X,X mK
C_{Res}	Resolution correction	X K	X,X mK		-1	-X,X mK
C_r	Reduced correction	X,XX °C	XX mK			

Agenda:

t_N Temperature of tempering device → see table 1

t_{Ind} Temperature indication at calibr. Point → average of several readings

t_{Ice} “True” value & uncertainty of ice-point realization

$t_{Ind.Ice}$ Temperature indication at ice-point → average of several readings

σt_{Th} Short term stability → estimated value (see above)

C_F Filament correction → Zero at total immersion of UUC

C_{Hys} Hysteresis correction → estimation by test or experience

C_{Res} Resolution correction → estimation depending human influences and hardware used

C_r Reduced correction → Square root of the sum of squared values in the column „Uncertainty“

Messgröße
DKD-K-
09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 5552-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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Dipl.-Ing. Reinhard Klemm

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4th step: Result of the calibration together with uncertainty, probability level & expansion factor

In the 4th and final step the complete measuring result is stated for the calibration certificate. It should be given in the following form:

At the calibration temperature of XX,XX °C, the reduced correction C_R for the thermometer [Serial Number] is

$$C_R = (\text{X,XX} \pm \text{uncertainty})\ °\text{C}$$

Denoted is the expanded uncertainty, which is calculated from the standard uncertainty by multiplication with the expansion factor k = 2.

This is equivalent to a normal distribution with a probability level of approx. 95 %.

The calibration was done in the a.m. sequence and temperature range. To achieve the stability of the instrument, the indicated temperature range should in no case over- or under stepped. The uncertainty given is calculated at the time and under the procedures of the calibration stated in this document. It contains no fractions for the long term stability.

Addendum:

Here following are some recommendations and / or guidelines for a realistic estimation of the particular uncertainties to be inserted into the a.m. tables or equitations. Please keep in mind, that each laboratory has its own "specialties", personnel, equipment and experiences. Therefore a global valid algorithm for the calculation of uncertainty is basically impossible. But some points of interest may be valid to put the focus onto:

Calibrated SPRT's mostly have an uncertainty of 3 .... 5 mK including k = 2
Normal distribution may be estimated → $u(t_N)$ is in the range of 1,5 to 2,5 mK

Uncertainties of the electric measuring equipment (bridges, DMM's, standard resistors, drift, ...) may be estimated to be in sum 5 mk and rectangular distributed → 5 mK / SQRT 3 = 2,9 mK

Uncertainty due to the inhomogenity of the liquid bath without any equalizing block or other facilities may be estimated to be 10 mK and rectangular distributed → 10 mK / SQRT 3 = 5,9 mK *

Uncertainty due to the stability of the liquid bath without any equalizing block or other facilities may be estimated to be 10 mK and rectangular distributed → 15 mK / SQRT 3 = 8,7 mK *

*Both parameters may be even better depending on the hardware used.

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 2389 - 409 - 02
Fax: +49 (0) 2389 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 41051605)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 5552-467 (BLZ 44010046)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

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Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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Uncertainty associated with the readings at calibration temperature, depending on scale spacing, magnifying glass with/without nonius, Hardware etc may be estimated to be 38 mK and normal distributed → 38 mK / 2 = 19 mK

The ice-point has to be realized by using demineralized and distilled water in a (clinical) clean ambient. It is known from earlier experiments that the temperature while using this procedure differs from 0 °C by approx. 3 mK. If it is not possible to follow a.m. procedure (trap water, no stainless container, no latex gloves, dusty environment, ...) the deviation may be higher by factor 2 to 4. The uncertainty is estimated to be in the range of 1,7 to 6,9 mK (rectangular distribution).

Uncertainty associated with the readings at ice-point, depending on scale spacing, magnifying glass with/without nonius, Hardware etc may be estimated to be 18 mK and normal distributed
→ 18 mK / 2 = 9 mK

The short term stability is estimated on the base of long experience and multi repeated tests with an uncertainty of 0,05 scale divisions = 5 mK rect. distribution → 5 mK / SQRT 3 = 2,9 mK

A correction of the filament by using a special filament thermometer is not necessarily in that particular case because the UUC was totally immersed into the liquid bath.

Every laboratory is an individuum with its individual equipment, routines, personnel, environment etc. and, of course, individual requirements of its customers.

The purpose of this document is to give a common valid recommendation for the estimation of uncertainty in measurement with liquid-in-glass thermometers. It is not in the status of a directive. It shall guide every laboratory to investigate its own capabilities - for the customer's benefits.

The calibration certificates which came back to Germany are o.k. The informations therein are not really satisfactionary from our side of view. None of the labs in the EAC – IC provided with the certificate a detailed description of the calibration routine as well as a list of equipment and its traceability they used in this case. Furthermore we miss a detailed uncertainty budget. We therefore ask each of the involved labs to give us the missing budget following the a.m. recommendations. It would be very helpful, if we could get the required documentation by latest end of week 8/2008 (February 22nd, 2008) per e-mail to:

Stefan.wallerath@ptb.de **and** Reinhard.klemm@centrocal.de

As soon as we get the results from the recalibration of the thermometers in Germany, we will create the final report and send it to the involved labs.

We thank in advance all involved labs for their cooperation.

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
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APPENDIX 2: Report EAC-IC by KEBS

Technical Protocol for E.A Supplementary Comparison in Temperature Metrology

Introduction

This report describes the results of an interlaboratory comparison of a liquid in glass thermometer calibration capabilities from 0 to 50 degrees Celsius. The laboratories that participated were Kenya, Uganda, Tanzania and Ethiopia

The intecomparison is designated as the EAST FRICAN COMMUNITY (EAC) intercomparison. Based on its success, it is hoped that another comparison using IPRTs will be organised in future with the aim of enhancing the region’s measurement capability.

Measurement procedures

One liquid in glass thermometer was circulated to the participating laboratories

Description: solid stem liquid in glass thermometer.
Manufacturer: Thermoschneider
Serial number: 7610199
Full graduation:0 ºC to 100 ºC

The comparison protocol was issued to all the participating labs. The inspection and measurement procedure may be summarised as follows:

1. a visual inspection using magnifying glass
2. rejoining if necessary of the mercury column
3. a rest period of 3 days after initial inspection and possible rejoining of a broken mercury column
4. measurements at 0 ºC,20 ºC,30 ºC,40 ºC and 50 ºC

It was not necessary for any lab to rejoin the mercury column of the thermometer.

Table 1: Circulation schedule

Laboratory	Measurement period
Kenya Bureau of Standards **(KEBS)**	2007-01-10 to 2007-02-15
Tanzania Bureau of Standards **(TBS)**	2007-02-16 to 2007-03-30
National Metrology Laboratory(NML), Uganda National National Bureau of Standards (UNBS)	2007-04-01 to 2007-05-01
Quality and standards authority of Ethiopia **(QSAE)**	2007-08-05 to 2007-09-05

Table 2: Contact details of the participants:

NMI	PHYSICAL ADDRESS	CONTACT PERSONS	EMAIL ADDRESS
Kenya Bureau of Standards **(KEBS)**	P. O. Box 54974-00200 Nairobi, Kenya Kapiti Road, Off Mombasa Road Tel: +254 20 605490, 602350/1 Fax: +254 20 609660	- Mr. Joel Kioko - Mr. Wilson Egadwa	- jkioko@kebs.org - egadwaw@kebs.org
Tanzania Bureau of Standards **(TBS)**	P.O. Box 9524 Dar-es-Salaam, Tanzania Junction of Morogoro/Sam Nujoma Roads, Ubungo Fax: +255-022-2450959 Tel: +255-022-2450298 / 2450206 / 2450949	Mrs. Edna Ndumbaro - Mr. Juliua Kisamo	- ednasmn @yahoo.co.uk - ijkisamo @yahoo.com

National Metrology Laboratory(NML), Uganda National National Bureau of Standards (UNBS)	P. O. Box 6329 Kampala, Uganda Plot M217 Nakawa Industrial Area Tel: +256 41 505995 Fax: +256 41 286123	- Mr. Yasin Lemeriga - Mr. simon rwashana	- yasin.lemeriga @unbs.go.ug - simonswashana @yahoo.com
Quality and standards authority of Ethiopia **(QSAE)**	P.O. Box 2310 Addis Ababa, Ethiopia Near AMCE Car Assembly; Tel: + 251 11 6460542 Fax: + 251 11 6460880/81	- Mr. Wondwosen Fisseha Mrs.Fitsum Tesfaye	-qsaemetrology @ethionet.et

Realisation

A time schedule for the intercomparison was fixed. The measurement started in early 2007 to be finished in late 2007.organization of the intercomparison measurement the transportation of the artefact and collection of the results were carried through by the Kenya Bureau of Standards.

Results

Initial measurements at the Kebs temperature laboratory are used as the reference laboratory.

The difference Lab value –Reference Value (LV-RV) are determined as follows:

LV-RV = correction determined at the participating lab-Correction determined at the reference lab

The En values are used to quantify the agreement between the participating lab and the refernce values

$$En = \left| \frac{X_{lab} - X_{KEBS}}{\sqrt{U_{LV}^{2} + U_{RV}^{2}}} \right|$$

Where U_{LV} and U_{RV} are the expanded uncertainties of measurements

Table 3: Calibration results

Date	Lab Name	Immer-sion	Actual temp. ˚C	Indic. Temp ˚C	Elc Corr. ˚C	Corr ˚C	Uncert. ˚C (k=2)	LV-RV ˚C	En
18/05/07	KEBS	TOTAL	0.0390	0.00	0	0	0.06	0.000	0.000
		TOTAL	20.143	20.04	0	0.0638	0.1	0.000	0.000
		TOTAL	29.986	30.00	0	-0.0534	0.08	0.000	0.000
		TOTAL	40.015	40.20	0	-0.2242	0.08	0.000	0.000
		TOTAL	49.854	50.10	0	-0.285	0.08	0.000	0.000
19/02/07	UNBS	TOTAL	0.0000	-0.05	0	0.05	0.22	0.05	0.2192
			20.000	19.92	0	0.08	0.22	0.02	0.0670
			30.100	29.91	0	0.19	0.3	0.24	0.7729
			39.950	39.90	0	0.05	0.3	0.27	0.88313
			50.100	49.90	0	0.20	0.3	0.49	1.5620
27/04/07	TBS	TOTAL	0.0000	0.07	0	-0.07	0.1	-0.07	-0.6002
			20.000	19.99	0	0.01	0.1	-0.0538	-0.3804
			30.000	30.10	0	-0.1	0.1	-0.0466	-0.3638
			40.000	40.21	0	-0.21	0.1	0.0142	0.1108
			50.000	50.22	0	-0.22	0.1	0.065	0.5075
29/08/07	QSAE	TOTAL	0.0009	-0.050	0	0.051	0.025	0.051	0.7846
			20.103	20.071	0	0.032	0.025	-0.0318	-0.3081
			30.045	30.087	0	-0.042	0.026	0.0114	0.1355
			40.058	40.156	0	-0.097	0.028	0.1272	1.5007
			50.072	50.340	0	-0.267	0.028	0.018	0.2123

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 55 52-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
USt-IdNr.: DE 170848458

Eingetragen unter
HRB 1468 Lünen
Geschäftsführer:
Dipl.-Ing. Reinhard Klemm

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Challenges

a. Communication with the different labs was difficult
b. There was no way to verify the metrological status of the instruments used.
c. The protocol's uncertainty budget was changed eventually

Observations

The results of the intercomparison are in general satisfactory, with minor exceptions.

The En values for UNBS 50 ˚C are > 1 these could be due to several factors among them being that they calculated their combined uncertainty linearly instead of the "square root sum of squares". As to the other reasons for the differences between Kebs and UNBS, that is not clear. Its not also clear as to why the En values for Ethiopia at 40˚C is greater than 1. Perhaps this needs to be investigated more. Perhaps it could as well be the problem with the pilot laboratory.

6. Conclusion

The success of this intercomparison provides a motivation to organise a regional comparison to compare the BMCs of the labs over a wider range. Industrial PRTs with readout may be suitable travelling thermometer (they are more robust than liquid in glass thermometers and can generally be used over a wider range). It is important to note that PTB has organised to train all of us before we embark on the second intercomparison. This will enable the participating labs own the document as well as harmonise our measurement procedures.

Messgröße
DKD-K-09701
Temperatur

CENTROCAL GmbH
Lohstraße 2, D-59368 Werne
Postfach 1461, D-59357 Werne
Tel.: +49 (0) 23 89 - 409 - 02
Fax: +49 (0) 23 89 - 409 - 80
E-mail: info@roesseltemp.de
www.roesseltemp.de

Stadtsparkasse Werne
Kto. 1420 (BLZ 410 516 05)
IBAN DE65 4105 1605 0000 0014 20
Postbank Dortmund
Kto. 5552-467 (BLZ 440 100 46)
Steuer-Nr. 333 598 801 93
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APPENDIX 3: 2nd EAC-IC with IPRT's

A. Artifacts:

Two off MI-Pt 100/0 with 3 mm sheath diameter by 150 mm immersion length.

4-wire-technology from measuring resistor

4 meter silicon-cable with 4 copper conductors. The length is not important.

Table and polynomial calculation according to IEC 751 A1 + A (EN 60751)

The artifacts are of a rugged construction. Therefore transport from lab to lab by a parcel service (UPS, FedEx or whatever is available) or post-parcel is not a problem if proper packed. For reliability, the shipping in two different parcels is strongly recommended.

CAUTION: Although the artifacts are MI-RTS's, bending is not desired. Especially 40 mm starting at the tip the first 20 mm behind the transition sleeve may be in no case bended.

B. Minimum immersion depth

For MI-IPRT's the min. required immersion depth is 20 times outer diameter plus length of the measuring resistor →20 * 3mm +25 mm = 85 mm
An immersion depth of 100 mm is recommended, the max. depth may not exceed 140 mm

C. Incoming procedure

Upon receipt of the artifacts an annealing procedure as following has to be done by **every** of the participating laboratories:

1. Measuring ice-point value
2. Ramp of 100 to 150 K/h up to 220 °C
3. Soak for one hour, max. 90 min at 220 °C
4. Ramp of 100 to 150 K/h down to room temperature
5. Measuring ice-point value
6. Ramp of 100 to 150 K/h up to 220 °C
7. Soak for one hour – max. 90 min. at 220 °C
8. Ramp of 100 to 150 K/h down to room temperature
9. Measuring ice-point value

The comparison of values as per item 1 and 5 above shows any damage occurred during transport, comparison of values as per item 5 and 9 above shows the 1st figure for stability. Repeat steps 6 to 9 until stability is inside the estimated uncertainty of each laboratory – but not more than 3 times. Take the final value for the further calibration steps.

D. Measuring Sequence

The measuring sequence is as following:

1. Ice point – may be taken from item C. above
2. -20 °C only for the labs who are accredit for this temperature – others ignore this step.
3. Next step 50 °C followed by 100 °C
4. Slowly cool down room to temperature
5. Ice-point value for checking stability
6. Next step 150 °C (ramp up as per item C above) followed by 200 °C
7. Slowly cool down room to temperature
8. Ice-point value for checking stability

E. Report and calibration certificate

Please provide any written report or comment in form of a Word® document, any table with values in form of an Excel® worksheet and the certificate in form of a PDF-document or JPG-picture.

The report of results from any lab is requested with following documentation:

1. Initial and final ice-point value as per item C above.
2. Values as per Item D above in the order mentioned.
3. Complete traceable calibration certificates for any of the instruments used.
4. Complete and detailed description of the calibration procedure used, hardware included. Parts and/or chapters from the working instruction(s) included in the QMS may be added for information – if available in English language.
5. Complete and detailed uncertainty budget similar to the guide as per appendix 1. Estimated values on the base of best available knowledge, experience and/or former measurements
6. Calibration certificate

Last lab in the queue please returns the artifacts to the address given in the bottom line.

Each lab please sends all the a.m. paperwork as a sampler per mail to following addresses:

QSAE,Mr. Wondwosen Fesseha, , as the pilot lab: qsaemetrology@ethionet.et

QSAE will prepare the final report and send it to:

Stefan.wallerath@ptb.de ***and*** Reinhard.klemm@centrocal.de

Thank you for your cooperation.

APPENDIX 4: Determination of uncertainty in measurement

Temperature range -20 ... 250 °C

$$t_x = t_N + \sigma t_D + \sigma t_{Eig} + \sigma t_{wa} + \sigma t_R + \sigma t_{Br} + \sigma t_{Hom} + \sigma t_{Stab}$$

Dimension	Item	Estimate	UOM	Uncert.	UOM	Distribution	Divisor	Sensit.	UOM	u_i (t)	UOM	(u_i (t))^2
t_n	Average of measured temperature	-20,000	°C	2,0	mK	Normal	2	1	---	1,0	mK	1,0000
dt_D	Correction: Drift of standard(s)			1,0	mK	Rectangular	1,73205	1	---	0,6	mK	0,3333
dt_{Eig}	Correction: Self-heating of standard(s)			3,0	mK	Rectangular	1,73205	1	---	1,7	mK	3,0000
dt_{Wa}	Correction: Heat dissipation of standard(s)			2,0	mK	Rectangular	1,73205	1	---	1,2	mK	1,3333
dt_R	Correction: Uncertainty of std. resistor			0,03	mΩ	Normal	2	9,97	K/Ω	0,1	mK	0,0224
dt_{Br}	Correction: Bridge uncertainty			0,3	mΩ	Normal	2	9,97	K/Ω	1,5	mK	2,2387
dt_{Hom}	Homogeneity of tempering device			2,0	mK	Rectangular	1,73205	1	---	1,2	mK	1,3333
dt_{Stab}	Stability of tempering device			2,0	mK	Rectangular	1,73205	1	---	1,2	mK	1,3333
t_X	**Temperature of tempering device**	**-20,000**	**°C**							**3,3**	**mK**	

Determination of uncertainty for a RTD with ~ 100 Ohm @ Watertripelpoint (WTR)

$$R_W(t_X) = R_W + \sigma R_{Ohm} + \sigma R_{Drift} + \sigma R_{Auf} + \sigma t_{P,Eig} + \sigma t_{P,Wa} + \sigma R_{Hys}$$

Dimension	Item	Estimate	UOM	Uncert.	UOM	Distribution	Divisor	Sensit.	UOM	u_i (t)	UOM	(u_i (t))^2
R_W	Bridge display	92,160	Ω	3,3	mK	Normal	1	0,39	Ω/K	1,3	mΩ	1,6375
dR_{Ohm}	Correction: Bridge uncertainty			0,3	mΩ	Normal	2	1,00	---	0,2	mΩ	0,0225
dR_{Drift}				0,2	mΩ	Rectangular	1,73205	1,00	---	0,1	mΩ	0,0133
dR_{Auf}	Correction: Bridge resolution			0,01	mΩ	Rectangular	1,73205	1,00	---	0,0	mΩ	0,0000
$dt_{P,Eig}$	Correction: Self-heating of artifact			3,0	mK	Rectangular	1,73205	0,39	Ω/K	0,7	mΩ	0,4637
$dt_{P,Wa}$	Correction: Heat-dissipation of artifact			2,0	mK	Rectangular	1,73205	0,39	Ω/K	0,5	mΩ	0,2061
dR_{Hys}	Correction: Hysteresis of artifact			2,0	mK	Rectangular	1,73205	0,39	Ω/K	0,5	mΩ	0,2061
R_X (t_X)	**Single uncertainty**									**1,6**	**mΩ**	

u_i (t)	UOM	Exp. Fact.		Expand. uncert.	UOM	Pt 100	UOM	BMC
1,6	mΩ	2		0,003193245	Ω	8	mK	10 mK

APPENDIX 4: Determination of uncertainty in measurement

Temperature range -20 ... 250 °C

$t_x = t_N + \sigma t_D + \sigma t_{Eig} + \sigma t_{wa} + \sigma t_R + \sigma t_{Br} + \sigma t_{Hom} + \sigma t_{Stab}$

Dimension	Item	Estimate	UOM	Uncert.	UOM	Distribution	Divisor	Sensit.	UOM	u_i (t)	UOM	$(u_i(t))^2$
t_n	Average of measured temperature	200,000	°C	2,0	mK	Normal	2	1	---	1,0	mK	1,0000
dt_D	Correction: Drift of standard(s)			1,0	mK	Rectangular	1,73205	1	---	0,6	mK	0,3333
dt_{Eig}	Correction: Self-heating of standard(s)			3,0	mK	Rectangular	1,73205	1	---	1,7	mK	3,0000
dt_{Wa}	Correction: Heat dissipation of standard(s)			2,0	mK	Rectangular	1,73205	1	---	1,2	mK	1,3333
dt_R	Correction: Uncertainty of std. resistor			0,03	mΩ	Normal	2	10,67	K/Ω	0,2	mK	0,0256
dt_{Br}	Correction: Bridge uncertainty			0,3	mΩ	Normal	2	10,67	K/Ω	1,6	mK	2,5596
dt_{Hom}	Homogeneity of tempering device			2,0	mK	Rectangular	1,73205	1	---	1,2	mK	1,3333
dt_{Stab}	Stability of tempering device			2,0	mK	Rectangular	1,73205	1	---	1,2	mK	1,3333
t_X	**Temperature of tempering device**	**200,000**	**°C**							**3,3**	**mK**	

4.1.2 Determination of uncertainty for a RTD with ~ 100 Ohm @ Watertripelpoint (WTR)

$R_W(t_X) = R_W + \sigma R_{Ohm} + \sigma R_{Drift} + \sigma R_{Auf} + \sigma t_{P,Eig} + \sigma t_{P,Wa} + \sigma R_{Hys}$

Dimension	Item	Estimate	UOM	Uncert.	UOM	Distribution	Divisor	Sensit.	UOM	u_i (t)	UOM	$(u_i(t))^2$
R_W	Bridge display	175,856	Ω	3,3	mK	Normal	1	0,37	Ω/K	1,2	mΩ	1,4760
dR_{Ohm}	Correction: Bridge uncertainty			0,3	mΩ	Normal	2	1,00	---	0,2	mΩ	0,0225
dR_{Drift}	Correction: Drift of bridge			0,2	mΩ	Rectangular	1,73205	1,00	---	0,1	mΩ	0,0133
dR_{Auf}	Correction: Bridge resolution			0,01	mΩ	Rectangular	1,73205	1,00	---	0,0	mΩ	0,0000
$dt_{P,Eig}$	Correction: Self-heating of artifact			3,0	mK	Rectangular	1,73205	0,37	Ω/K	0,6	mΩ	0,4055
$dt_{P,Wa}$	Correction: Heat-dissipation of artifact			2,0	mK	Rectangular	1,73205	0,37	Ω/K	0,4	mΩ	0,1802
dR_{Hys}	Correction: Hysteresis of artifact			10,0	mK	Rectangular	1,73205	0,37	Ω/K	2,1	mΩ	4,5061
$R_X(t_X)$	**Single uncertainty**									**2,6**	**mΩ**	

u_i (t)	UOM	Exp. Fact.		Expand. uncert.	UOM	Pt 100	UOM	BMC
2,6	mΩ	2		0,005139559	Ω	14	mK	10 mK