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REPORT ON WEMC THERMAL AUDIT PACK TH 3

FINAL REPORT

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REPORT ON WEMC THERMAL AUDIT PACK Th 3

Final report

0. Summary

Western European Metrology Club (WEMC) has appointed a working group for Calibration Service. One of the activities of this group is to organize circulation of audit devices between laboratories in the participating countries, the aim being to gain further mutual confidence in the calibration services operating in these countries. This second report describes the result of one such audit circulation in the field of thermal measurements, Th 3 carried through a second circulation between April 1980 and May 1981. The same pack was carried through a first circulation between September 1979 and April 1980. The results of the first circulation are found in a first report available from the author (SP-RAPP 1980:19).

1. Composition of the pack

The audit pack Th 3 contains a thermocouple of type S, platinum-10% rhodium/platinum with a total length of about 1620 mm. The couple is fitted with a twinbore tube of outer diameter 3 mm and length 600 mm. Most of the rest of the wires are insulated with systoflex tubing. The thermocouple is marked S 005.

2. Measurements

The thermoelectric voltage (emf) of the thermocouple was to be measured at temperatures 600°C and 1000°C with the reference junctions of 0°C. Information on the statement of the results can be found in the measuring instruction sheets which are attached to this report as Appendix 1.

3. Participating countries and laboratories

The audit pack was put together by the Swedish primary calibration centre for temperature at statens provningsanstalt (National Testing Institute). The following countries have participated in the circulations:

Country	No. of labs	Time span	Coordinator
Sweden	ref + 2	--1979-11-26	O. Mathiesen
Norway	2	-11-27--1980-01-11	K. Birkeland
Denmark	2	01-17--03-03	V. Erichsen
Sweden	ref	03-06--04-13	O. Mathiesen
Austria	1	04-16--06	G. Slanar
Italy	2	-07--10-10	P. Soardo
U.K.	7	11-05--1981-04-08	J. Wilson
Sweden	ref	1981-04-16--	O. Mathiesen

All the participating laboratories were approved by the national authorities except for the Norwegian laboratories, the Austrian laboratory and one of the Italian laboratories. However, the Austrian laboratory and one of the Norwegian laboratories are the primary laboratories for these countries. The Italian laboratory has made an application for approval.

According to the decision taken by the WEMC, the individual laboratories are not identified in this report. Instead, in the presentation and discussion of the results the reference to the different laboratories is through concecutive letters.

4. Results

The results of the second circulation at the different temperatures are given both in tabular form (tables I and II) and graphically (figures 1 and 2). In the figures are also included the results from the first circulation. The presentation of the results is in what is believed to be cronological order. Most laboratories do not give the actual day of test and the date of issue can be several months later. The error bars indicated are the total inaccuracies which are the arithmetically added contributary inaccuracies given by the laboratories. In some cases these are given i temperature but in the tables and the figures these are converted to inaccuracies in thermoelectric voltage.

4.1 Reference values

The values from the reference laboratory before and after the circulations were provided by the Swedish primary calibration centre for temperature. The measurements were done by comparison in an electric tube furnace with an at NPL fixpointcalibrated type S thermocouple standard. The furnace is equipped with a temperature equalising metal block with a protecting tube of alumina. The hot junctions of the thermocouples were tied together with pure platinum wire to achieve good thermal coupling. The immersion depth of the thermocouples was 300 mm. The reference junctions of the thermocouples were kept in separate closedend glasstubes in an icewater bath.

The arithmetic mean of the result at 600°C in the first circulation was 5238.6 µV and for the second circulation 5239.6 µV. The mean value for all laboratories was 5239.3 µV. The standard deviations were 3.6, 2.6 and 3.0 µV respectively. The arithmetic mean at 1000°C is for the first circulation (9590.6 ± 6.0) µV and for the second (9591.7 ± 2.2) µV. The value for both circulations was (9591.4 ± 4.1) µV.

4.2 Measuring methods

The measuring methods differ quite a lot for this last circulation, especially the determination of the 600°C value. The laboratories H and I made a comparison with platinum resistance thermometer in a fluidised alumina bath at about 600°C and at 1000°C the laboratories used the method of comparison with type S thermocouple. The laboratory, O, compared the audit with a platinum resistance thermometer in a nitrate salt bath at about 600°C. Also the laboratory, G, made a comparison in a nitrate salt bath with a platinum resistance thermometer at around 600°C and the 1000°C value was taken from comparison with two type S fixed point calibrated thermocouple standards.

In addition, the thermocouples were checked at the gold point. These informations were given after contact with the laboratory G to clarify the error components in the certificate. Four of the other laboratories, namely K, M, N and P used Pt-13%Rh/Pt thermocouple (type R) for the comparisons and of the two remaining laboratories one, L, doesn't mention any method and the other, J, made a comparison and/or a fixed point calibration.

4.3 Statement of inaccuracy

The measuring instruction (appendix 1) stated that the overall inaccuracy should be given together with a split up into different systematic errors. Several of the possible systematic errorsources are mentioned in appendix 1 and the most important ones are listed in tables I and II were the errors in temperature always are converted to the equivalent thermoelectric voltage. The Seebeck coefficients of type S thermocouple at 600°C and 1000°C are 10.2 and 11.5 μV/°C respectively.Several laboratories, namely, H, J, L and P have not given any split up of their systematic errors. Usually the laboratories have got the total error by arithmetically adding the components, but the laboratory I has added quadratically and besides added a "security" term in order to give an appropriate total inaccuracy. Under "Inaccuracy Total" in tables I and II are given the arithmetic sums of the stated systematic inaccuracy components. In case no split up is given the stated total inaccuracy in °C has been converted to μV.

The two Italian laboratories have written their reports in Italian, which caused some language problems in interpreting their reports.

5. Discussion

When all results from both circulations have been compiled, the general statement of a satisfactory result is well established. The arithmetic mean values are slightly higher for the second circulation and the standard deviations at both temperatures are smaller for this last circulation.

During the second circulation some photographs were enclosed with the audit to describe the appearance of the thermocouple when it was leaving the reference laboratory. It was evident that the audit device had become distorted at the hot junction during the circulation but the deformation does not appear to have affected the measurements to any appreciable extent.

5.1 Laboratory G

Before calibration the laboratory made an annealing at 1070°C for 1 h.

5.2 Laboratory H

This laboratory does not state the actual value at 600°C but gives several surrounding values so the given value in table I is interpolated from values between 593 and 1000°C. The lowest value 593°C is measured with a platinum resistance thermometer in a fluidised alumina bath. The measuring equipment is well described. No breakdown of the total inaccuracy has been given.

5.3 Laboratory I

Also this laboratory has made a comparison in an alumina bath at 600°C with a platinum resistance thermometer. The total inaccuracy given has come from quadratic addition added to a "security" term. The inaccuracy component "other" in the tables, originate from instabilities in the fluidised bath and furnace.

5.4 Laboratory J

The laboratory J has not given a split up of their systematic errors and the methods of measurement are not clearly stated.

5.5 Laboratory K

This laboratory measured at two immersion depths, 250 and 500 mm. The results were a 1 µV higher value at 250 mm and 600°C and the same value for both immersions at 1000°C. The values in the tables are given for the immersion depth of 500 mm. The certificate includes a list of available, certified equipment but does not state the actually used equipment. Annealing was performed but no time or temperature were given.

5.6 Laboratory L

No breakdown of the inaccuracy components was given and no calibration method is mentioned.

5.7 Laboratory M

The test conditions are well described in the certificate, but no annealing time at 1065°C is given.

5.8 Laboratory N

The voltage inaccuracy includes contributions from the potentiometer and its standard cell, current controller standard cell and standards oil bath temperature. All the contributions are added quadratically and gives ± 0,316 K.

5.9 Laboratory O

This laboratory made measurement only at 600°C but not at 1000°C. The audit were compared in a nitrate saltbath with a standard platinum resistance thermometer at several temperatures close to 600°C. The inaccuracy contribution under "other" originates from the instability of the calibration bath. The laboratory points out the deformation of the hot junction and attached a photograph in the certificate.

5.10 Laboratory P

No components of the inaccuracy has been given. The laboratory has incorporated a photograph of the junction in the certificate.

6. Conclusions

The main result of this last audit circulation is the small standard deviation of only $\pm$ 0.3°C at 600°C and $\pm$ 0.2°C at 1000°C. The mean value is within the inaccuracy bars of all the laboratories in the last circulation.

The good result of the first circulation is even more emphasized with this final circulation.

Table I. Measurement results at 600°C.

Laboratory	Emf (μV)	Inaccuracy (± μV) Total	Standard	Thermal coupling, gradients	Ref. junction	Voltage	Parasitic emf in connection	Other	Total inaccuracy (±°C)
Ref.	5237	5	3	1		1			0,5
G	5235	6	1[a]	3[b]		2			0,6
H	5241.4[e]	10							1
I	5241	3.9	1.8	0.7	0.2	0.2		1	0.3[c]
J	5240	10							1
K	5244	10	3	2.5	0.5	4			1
L	5238	10							1
M	5240	10	3	2	1	4			1
N	5242	4.4	3	1	0.1	0.3			1[d]
O	5240	3	0.2	1.3		1		0.5	1[d]
P	5241	20							2
Ref.	5236	5	3	1		1			0.5

a) for realization of IPTS-68
b) includes interpolation
c) quadratic addition added to a "security" term (0.12°C)
d) approved inaccuracy
e) interpolated from several values between 593 and 1000°C

Table II. Measurement results at 1000°C.

Laboratory	Emf (μV)	Inaccuracy (± μV) Total	Standard	Thermal coupling, gradients	Ref. junction	Voltage	Parasitic emf in connection	Other	Total inaccuracy (±°C)
Ref.	9590	6	3	—— 2 ——		—— 1 ——			0.5
G	9594	15	3[a]	10[b]		2			1.3
H	9591.1	11.5							1
I	9591	14.1	5.7	5.6	0.1	0.4		2.3	1[c]
J	9592	11.5							1
K	9596	11.5	3.5	2.9	0.5	4.6			1
L	9591	11.5							1
M	9589	11.5	3.5	2.3	1.1	4.6			1
N	9592	5.1	3.5	1.2	0.1	0.3			1[d]
O	-								
P	9594	34.5							3
Ref.	9589	6	3	—— 2 ——		—— 1 ——			0.5

a) for realization of IPTS-68
b) includes interpolation
c) quadratic addition added to a "security" term (0.33°C)
d) approved inaccuracy

Lab C3

Temp. section

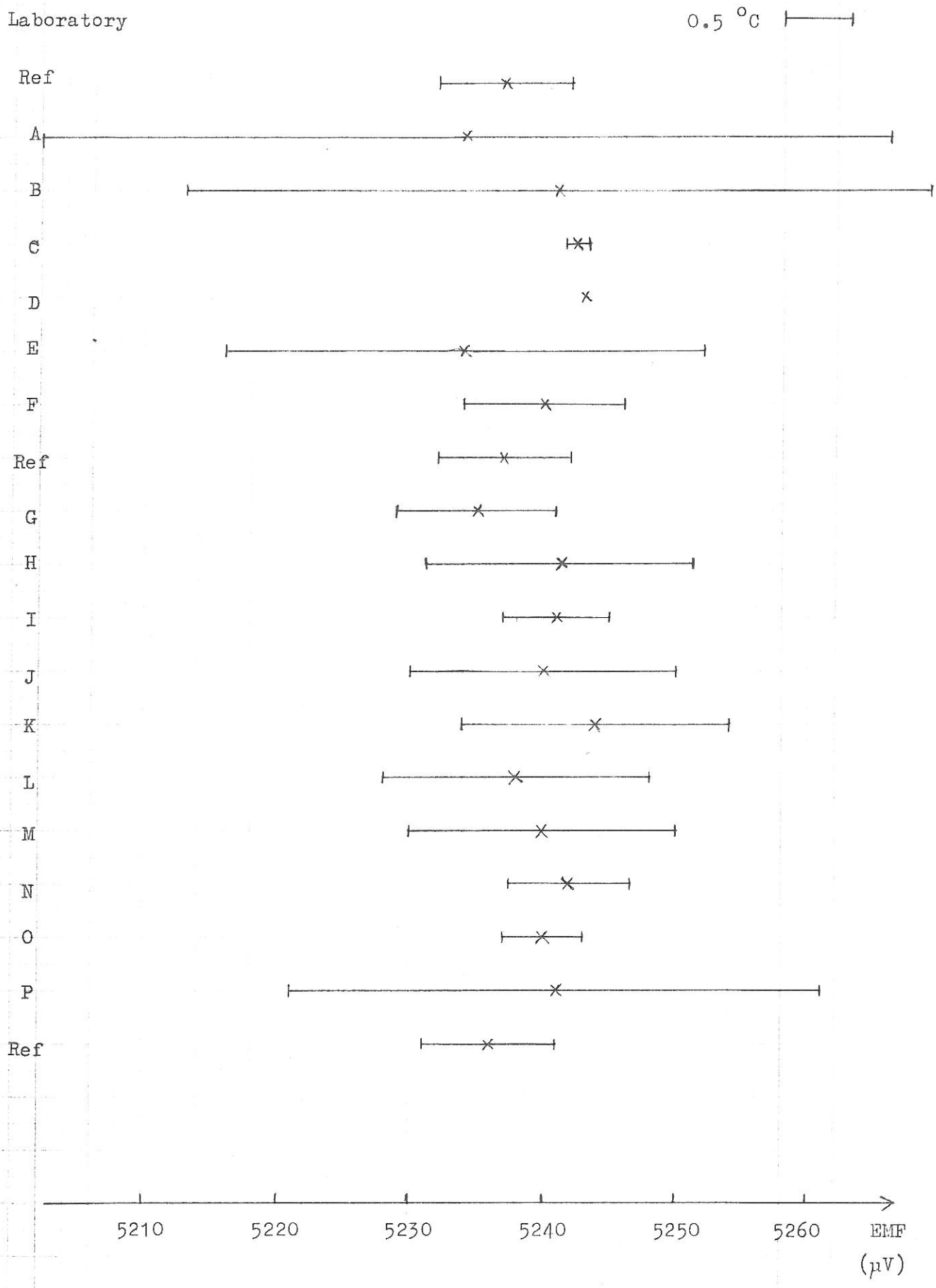
Figure 1

WEMC

Audit Th 3

Measurements at 600 °C

(Results from both circulations of the audit)



Lab C3

Temp. section

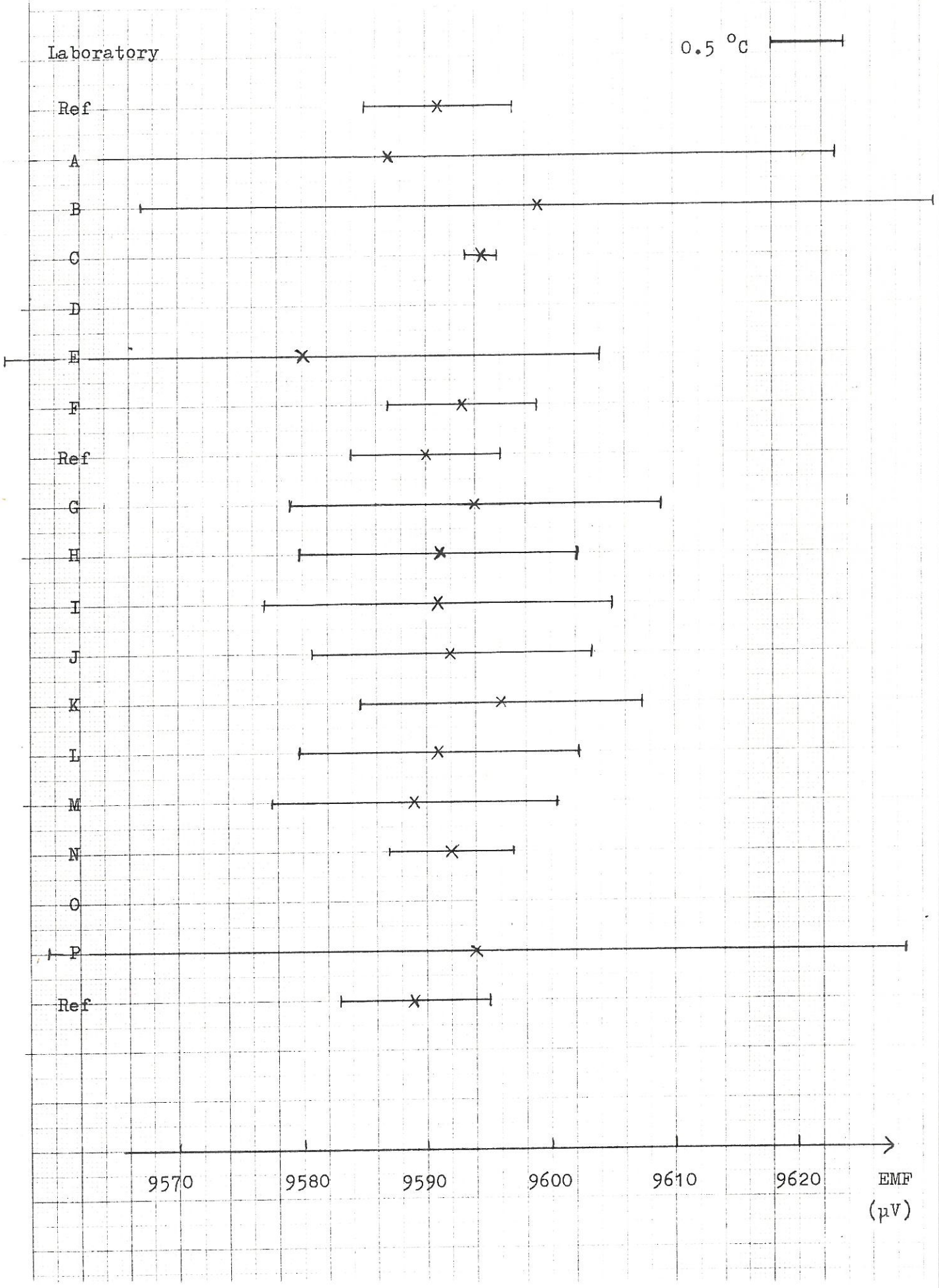
Measurements at 1000 °C

(Results from both circulations of the audit)

Figure 2

WEMC

Audit Th 3



Lab C 12
Temperature section

1980-04-16

Appendix 1

Measuring instructions for WEMC temperature audit Th 3

On receipt, unpack the thermometer carefully, checking for damage.

1 Description of the temperature audit

The thermocouple is of type S, platinum-10% rhodium/ platinum with a total length of ca 1620 mm. The couple is fitted with a twin bore tube of outer diameter 3 mm and length 600 mm and most of the rest of the wires are isolated with systoflex tubing. The thermocouple is marked S 005.

2 Measurements

The thermoelectric voltage of the thermocouple shall be determined at temperatures 600°C and 1000°C with the reference junctions at 0°C.

It is recommended that the thermocouple is annealed before calibration, but in any case the thermometer must not be dismantled and not exposed to temperatures higher than 1100°C.

3 Handling precautions

The thermometer was realeased for the test in full annealing conditions. Therefore a reasonable care in handling is required, in order not to introduce cold working. It is suggested that the thermometer, when not in operation, is left in its case and kept far away from the risk of accidental shocks or contaminatic The thermometer can be sent from one laboratory to another unaccompanied provided that great care is exercised during transportation.

The enclosed photos describe the appearance of the thermocouple when it was leaving the reference laboratory. Every laboratory must carefully check the state of the audit device, especially the hot junction and the transition from the twin bore tube to the systoflex tubing. In case of deformation, this should be noted or still better the deformed zone of the thermocouple photographed and the photo enclosed with the documentation of the audit.

4 Statement of the results

Please state the corrected result of the measurement, i.e. the result corrected for all known systematic effects, like calibration factors, deviations of the standards from their nominal values etc.

The uncertainty of the corrected result is for this type of measurement governed by remaining systematic errors. The error sources which contribute to the overall systematic error include, among others, the uncertainty of the calibration factors, the uncertainty of the voltage measurement, the instability and the gradients of the furnace, the inadequacy of the thermal coupling between the audit device and the reference standard, the uncertainty of the temperature of the reference junctions, etc.

Please state the uncertainty of the results in the same way as you do in your ordinary work. In addition to this, in order to facilitate the comparison of the results from the different participants, it is appreciated if you can include a list of the different systematic error components taken into consideration and their magnitude, and a statement of how the components have been combined to obtain the overall error.

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Please contact the audit scheme coordinator in your country if you need additional information about the carrying out of the measurements or the reporting of the results.

Please repack the thermometer carefully as received and deliver it promptly to the next laboratory according to the instructions given by the audit scheme coordinator.

Your results are needed for the preparation of the report on this audit circulation. The sooner you deliver your results, the sooner will you get the report containing a comparison of the results of all the participants. Therefore, please deliver your results promtly to the audit scheme coordinator in your country.