Technical Protocol for the Supplementary comparison AFRIMETS.M. D-S2

Comparison on density determination of liquid samples using oscillation-type density meters

Pilot Laboratory: NIS- National Institute of Standards, Egypt

1) Outline

The aim of the supplementary comparison AFRIMETS.M. D-S2 "Comparison on density determination of liquid samples using oscillation-type density meters" is to compare the results of the density determinations of liquid samples by oscillation-type density meters of the participating laboratories.

Density measurements of liquids are mainly performed by laboratories to provide a means of calibrating or checking liquid density measuring instruments such as oscillation-type density meters.

The National Institute of Standards (NIS) is the Pilot Laboratory. For the addresses of the participants, see **Appendix A**.

For the comparison samples of Distilled Water and (Fluorinert FC-40) were prepared. The measurements should be carried out at reference temperature of 20 °C and standard atmospheric pressure by using a digital density meter.

The reference values for the comparison liquids will be calculated with Cox formalism **[1]** using density values determined by the hydrostatic method at BEV and PTB. The degree of equivalence (D_i) and the normalized error (E_n) will be used to evaluate the results of the participants.

During the 16th AFRIMETS Committee for Mass and Related Quantities (TCM) meeting held on 16th - 20th July, 2023 at the NIS, Egypt. A supplementary comparison on density determination of liquid samples using oscillation-type density meters was proposed. Draft A of the Report should be distributed until January 2025 and Draft B of the report should be available by July 2025 (for the timetable, see **Appendix B**).

There will be 5 participants including the pilot laboratory.

2) Purpose of this document

The purpose of this document is to provide the participating laboratories with instructions for the handling of the liquids and to report on the measurement results, the measuring procedure and the measuring device. It is important that all instructions given in this document be followed. This will ensure that the measurement data are obtained under comparable conditions and are presented in the same format. Any deviation from the instructions has to be reported to the Pilot Laboratory.

3) Liquid samples

For the comparison, a volume of at least 500 milliliters of Distilled Water and 200 milliliters of FC-40 as shown in **Figure 1**, will be prepared or purchased by the pilot laboratory.

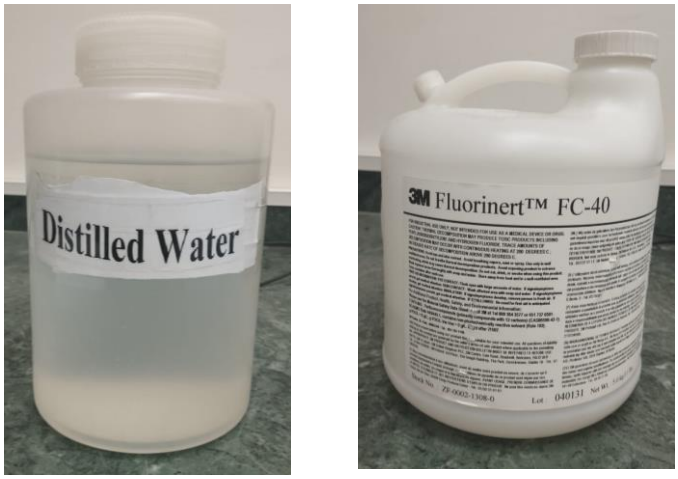

Fig. 1 Liquid samples

Before sending, each liquid will be homogenized and filled into 125 milliliters transport bottles, (5 for Distilled Waterand 5 for FC-40). Two samples of each liquid will be used for monitoring purposes by measuring the density at 20°C with an oscillation-type density meter (DSA 5000 M). This is performed by the pilot laboratory in order to assess inhomogeneity-caused deviations among the density values determined by the participants. Additionally, the density of one sample of each of the two liquids will be measured periodically by the pilot laboratory to evaluate the stability of the liquid. Thus, a total of 10 bottles will be prepared.

4) Transportation

For transportation, the liquids will be filled into glass bottles of 125 milliliter volume. The individual bottles will be provided with the name of the liquid, the volume, and a safety warning.

Each participant will receive one sample of Distilled Waterand one sample of FC-40. (A form to inform the pilot laboratory see **Appendix C**).

The liquids were transported with the persons who attend the meeting. The package will be provided with a warning: "***To be opened only by laboratory personnel.***"

The participants are responsible for completing their local customs formalities.

After delivery, the participating laboratories must inform the pilot institute without any delay giving details of the state of the packages and their contents by completing the form contained in the package and sending it back to the pilot laboratory by e-mail (pdf). The template will also be sent in parallel by e-mail see **Appendix D**.

5) Preparation of the measurements

After receiving the samples, each participant has to start the measurements as soon as possible, but any regulations of the participating laboratory (e.g. handling regulations of liquid samples according to the quality systems of the laboratory) have to be followed.

The bottles and the seals should be opened only for the measurements. Before opening, please check the individual bottles once more for obvious damage or contamination.

Any remarkable observations should be reported.

Each liquid sample should be handled according to the regulations of the laboratory. These handling regulations shall be reported.

Do not degas the samples.

Anyway, act with caution in order to prevent changing the density irreversibly.

6) Measurement procedure

The following target temperatures were chosen for the comparison:

Deionized distilled water: 20°C

FC-40: 20°C

The participating laboratories have to make measurements at 20°C.

For each liquid and temperature, a series of measurements sequences have to be performed. Afterward, the liquids can be disposed of.
Values of the cubic expansion coefficients and isothermal compressibility of the liquids are listed in **Appendix E** together with their uncertainties. These values should be used as necessary.

Mean ambient conditions (pressure and temperature) during the measurements must be reported. If required for the evaluation of your measurements. The calculation of the air density must be based on the CIPM formula (CIPM - 2007) **[2]**.

Do not send the samples or bottles back to the pilot laboratory.

Each participant must start the measurements within 14 days of delivery.

Additionally, the pilot laboratory must be informed of completion of the measurement at the participant.

7) Reports

The reports on the measurements and the results should be made using the enclosed report forms 1 and 2. The report form 1 is an MS Excel sheet and report form 2 is an MS Word document. For each liquid and temperature one

Excel sheet of report form 1 has to be completed. The report form 2 should contain a summary for all measurements, and may be reported only once if they are no differences in the procedures.

7.1) Measurement results

The following information has to be given for each liquid and target temperature using the format in **Report Form 1**.

a) Date of arrival of the liquid.
b) Condition of package, seals and bottles (Pre-Measurement Comments).
c) Date of opening the bottles and numbers of bottles used.
d) Preparation of liquid (Pre-Measurement-Comments).
e) Density meter type (being used for the comparison)
f) Date and time of measurement.
g) Mean density at actual temperature and pressure, actual temperature, actual pressure.
h) Densities and uncertainties at target temperature and pressure (1013.25 hPa) with complete uncertainty budget.
i) Ambient conditions during the measurements of the liquid.
j) Any post measurement comments if necessary

7.2) Detail information about the national density standard

Use the format in **Report Form 2.**

a) What is your national reference in density?
b) Principle of the reference apparatus.
c) To which primary standard is the density traceable? In mass:
d) To which institute is it traceable?
e) To which primary standard is the density traceable? In volume:
f) To which institute is it traceable?

g) Give a short description of the hierarchic scheme in density at your institute.

7.3) Detailed information about the density meter used

Use the format in **Report Form 2.**

a) Density meter type (being used for the comparison),
b) Readability,
c) Accuracy,
d) Repeatability,
e) Measuring range,
f) Calibration interval,
g) Internal adjustments of the density meter,
h) Used reference materials for the adjustment a. How often?
    1) With water?
    2) With air?
    3) With other reference materials (which one)?
    4) At which temperatures?
i) How do you validate the device, that means how do you check / monitor the measuring capability?
j) How do you detect e.g. contamination of the tube?
k) According to your quality system: How do you document these adjustments or checks?
l) Give a short description of these tests (especially maximum permissible errors of these tests)
m) Describe the procedures in the case of deviations from normal conditions (e.g. one of the tests is outside a limit)
n) Do you perform calibration of your density meter? a. in density?

1) in temperature?
2) in viscosity?
3) How often?
4) How?

o) According to your quality system: give a short description of these calibrations. How do you document these measurements?
p) How do you consider the results of these calibrations?
q) Do you consider any other calibrations factors to your final density results? Which one?

7.4) Detail information about the measurements with the density meter

Use the format in **Report Form 2.**

a) Describe the storage conditions of liquid samples according to your quality system.
b) How do you prepare the liquid samples for the measurement?
c) How do you insert the liquid samples into the unit? What amount do you use?
d) Describe your measurement procedure.
e) Describe your cleaning procedure of the unit between the measurement series.
f) Describe your cleaning procedure of the unit after the measurement series.

Give the mathematical equations (model) for calculating the density of the liquid samples at the target temperature and pressure.

Describe how the standard uncertainties of the individual influence quantities of Report Form 1 in the uncertainty of the liquid density were estimated by participant.

Please give references to publications about your apparatus if existing.

7.5) Uncertainty of measurement

Appendix F gives a list of main components of the uncertainty budget. Please add any additional uncertainty component (due to e.g., the measurement principle, calibration correction, viscosity correction, resolution (display accuracy) etc.) occurring in your measurements. Do not include a term for a potential long-term drift of the density of the liquid sample.

The uncertainty of the density is to be given as expanded uncertainty *U*95 for a confidence level of 95%. This is obtained by combining the individual standard uncertainties obtained from Type A and Type B evaluations. The uncertainty evaluation should include the list of all influence quantities, the values and the standard uncertainties, together with their degrees of freedom and the combined standard uncertainty, as well as the effective degrees of freedom. The uncertainties are to be calculated and reported according to ISO "Guide to the Expression of Uncertainty in Measurement" **[3]**.

7.6) Deadline

The reports must be sent to the pilot laboratory by e-mail as soon as possible and **six weeks** after the measurements are completed at the latest. The exact deadline (for all participants the same) will be fixed by the pilot institute after sending the liquids and receiving the reports of delivery. A result is not considered to be complete if no associated uncertainty supported by a complete uncertainty budget is given. The results are confidential until Report

Draft A will be distributed. Draft A is confidential to the participants of the comparison.

8) Special problems

8.1) Failure of travelling standard

If a liquid sample is spoiled or lost during transportation, a new sample can be sent as long as enough liquid is available at the pilot laboratory.

8.2) Late entry of a participant

Due to the tight timetable, it is not possible for an additional participant to join after the samples were distributed.

9) References

[1] Cox, Metrologia 2002, **39**, 589-595.

[2] A. Picard, R. S. Davis, M. Gläser, K. Fujii: “Revised formula for the density of moist air (CIPM-2007)”, Metrologia, 2008, **45**, 149-155.

[3] "Guide to the Expression of Uncertainty in Measurement," International Organization for Standardization (ISO), 1995.

[4] T. Brunet et al, “Resonant ultrasonic attenuation in emulsions”, Journal of Physics: Conference Series 457 (2013) 012006

Appendices

A. Participants

Laboratory (country)	Mailing address for the packages	Person responsible for the comparison
Egypt: NIS	**National Institute of Standards (NIS)** Tersa St., El-Haram, El-Giza, Egypt P. O.Box 136 Giza-Code 12211	**Dr. Mohamed Hamdy** Mohamed.hamdy@nis.sci.eg Tel.: +2 01001929796 Fax: +202 33867452
Kenya: KEBS	**Kenya Bureau of Standards (KEBS)** Popo Road, off Mombasa Road P.O Box 54974 Code 00200 Nairobi – Kenya	**Mrs. Beatrice Lugadiru** lugadirub@kebs.org Tel.: +254722377640 Fax: +254 020 6948000
Tanzania: TBS	**Tanzania Bureau of Standards (TBS)** P O Box 9524, Sam Nujoma Road / Morogoro Road, Ubungo, Dar es Salaam.	**Mrs. JENITHA ISHABAGANZI** Jenitha.ishabaganzi@tbs.go.tz Tel.: +255 756 453 679 Fax: +255 689 929 936
Uganda: UNBS	**Uganda National Bureau of Standards (UNBS)** Plot 2-12 By-Pass Link, Bweyogerere Industrial And Business Park, P.O Box 6329 Kampala	**Mr. Mukwaya Simon Peter** simon.mukwaya@unbs.go.ug Tel.: +256 704418960 **Mr. Jonathan Omara** jonathan.omara@unbs.go.ug Tel.: +256 0703 243087
Kingdom of Saudi Arabia: SASO-NMCC	**National Measurement and Calibration Center (NMCC)- SASO** Riyadh - Al Muhammadiyah - in front of King Saud University PO. B 3437 Riyadh 11471	**Mr. Rayan AlYousefi** r.yousefi@saso.gov.sa Tel :+966 50 547 4746

B. Timetable for the comparison

July, 2023	Decision of comparison on the 16th AFRIMETS Committee for Mass and Related Quantities (TCM) meeting
31st July, 2023	Questionnaire
31st Aug., 2023	measurements and stability test of liquids at NIS
31st Aug., 2023	Deadline for answers to questionnaire
15th Sep., 2023	Decision on participants
30th Sep., 2023	First draft of the Technical Protocol
16th Nov., 2023	Agreement on Technical Protocol
17th Dec., 2023	Reports from all participants
31st Jan., 2024	Draft A of comparison report
31st Mar., 2024	Draft B of comparison report

C. E-mail: Receipt of a comparison package

To monitor the progress of the comparison, we ask on receipt of the packages to kindly send a report by e-mail to

Mohamed Hamdy Mohamed

National Institute of Standards (NIS)

Tersa St., El-Haram, El-Giza, Egypt P. O.Box 136 Giza-Code 12211

Mohamed.hamdy@nis.sci.eg

This report should contain the following information:

- Participating laboratory
- Contact person
- Telephone
- Mail address and a text like this:

The packages of the AFRIMETS.M.D-S2 were received on (date).

The seals of the bottles were (not) broken.

The packages seem, after short inspection, (not) to be damaged.

If damaged: (not) seriously.
The contents is probably (not) suitable for use.
Remarks: (e. g. No. of bottles with broken seals)

Date of delivery

D. E-mail: Progress report

To monitor the progress of the comparison, we ask to kindly send a report by e mail to

Mohamed Hamdy Mohamed

National Institute of Standards (NIS)

Tersa St., El-Haram, El-Giza, Egypt P. O.Box 136 Giza-Code 12211

Mohamed.hamdy@nis.sci.eg

When the measurements are completed, this report should contain the following information:

Participating laboratory
Contact person
Telephone
Fax
and a text like this:
Supplementary comparison AFRIMETS.M.D-S2 "Comparison on density determination of liquid samples using density meters"
The measurements were completed on (date).
Remarks:

E. Physical constants of the liquids

Uncertainties are standard uncertainties (k = 1).

Cubic thermal expansion

Liquid	Nominal density in kg/m^3	Cubic thermal expansion in kg/(m^3 K)	Uncertainty (k = 1) in kg/(m^3 K)
Deionized distilled water	998	0.21	0.02
FC-40 (20 °C)	1866**	2.23**	0.06***

* Measured

** From Datasheet 3M

*** Estimated from Datasheet 3M

Isothermal Compressibility

Liquid	Isothermal compressibility in 10^{-11}/Pa	Uncertainty (k = 1) in 10^{-11}/Pa
Deionized distilled water	46	2.1
FC-40 (20 °C)	131 [4]	5*

* Estimated

F. Main components of uncertainty

The uncertainties of the following components can be taken into consideration for the calculation of the uncertainty of the liquid density. If you have additional uncertainty components, please add them in Report Form 1.

Mean density and experimental standard deviation

Type A uncertainty, please give the experimental standard deviation of the mean, see "Guide to the Expression of Uncertainty in Measurement," chapter 4.2.3.

Temperature of liquid

Uncertainty contribution has to be taken into consideration.

This must contain the uncertainty of the temperature.

Cubic thermal expansion of liquid

The density of the liquid has to be given for the desired temperatures.

The values of Appendix E can be used.

Pressure in liquid

Uncertainty contribution has to be taken into consideration.

This must contain the uncertainty of the pressure measurement.

Isothermal compressibility of liquid

The density of the liquid has to be given for 1013.25 hPa.

The values of Appendix E can be used.