Specific Gravity and Density Determination in Nonbeverage Products

Scope and Application

This method follows from SSD:TM:102. The purpose of this method is to apply the specific gravity determination methodologies from SSD:TM:102 to nonbeverage products, as well as to determine density of nonbeverage products.

This method applies to the determination of the specific gravity and density of liquid products (flavors, medicines, dietary supplements, perfumes, and products found unfit for beverage purposes). The specific gravity is reported as a dimensionless quantity. Density is reported as g/mL or lb/gal.

Levels and Limitations

The range of density meters is typically 0 to 3 g/mL.

Supplemental Documents

1. SSD:TM:102 Ethanol Determination by Specific Gravity
2. WG:Density Meter:001 Anton Paar Density Meter Operating Instructions
3. WG:Density Meter:003 Anton Paar Density Meter Maintenance Schedule and Instructions (Formerly SSD:WG:531)
4. Form:Density Meter:001 Anton Paar Density Meter Daily Log (Formerly SSD:Form:103)
5. Form:Density Meter:003 Densitometer Air Water Check Maintenance Schedule (Formerly BAL:Form:102-4)
6. WG:SSD:1040:004 Beer and Wine Degassing Procedure (Formerly SSD:WG:115)

Equipment

Density Meter (Anton-Paar, DMA-4501 or equivalent), reporting specific gravity to at least 4 decimal places, temperature controlled 20.00±0.01°C

Automatic Sample Changer (Anton-Paar XSample 530 or equivalent)

Fisherbrand VersaClosure Tube Closures, Globe Scientific part number 113148, or equivalent

Kimble Disposable Culture Tubes (16 x 100 mm), Fisher Scientific part number 03-341-4, or equivalent

Reagent and Sample Preparation and Handling

Reagents:

Deionized (DI) water, 18 MΩ/cm or better
Ethyl Alcohol, 200 Proof, (CAS#64-17-5)
Any other solvents deemed appropriate for cleaning and drying the instrument, as needed
Distilled spirit LCS (LCS)

Procedures

1. Verify that the DI water and 200 proof ethanol (or other appropriate cleaning and drying solvents) vessels attached to the density meter contain sufficient volume to analyze samples (ask instrument team, if needed).
2. Confirm that a passing air check and water check have been performed for the month This is recorded on Form-Density Meter-003 along with other pre-run checks. For more information on how to run checks please refer to WG-Density Meter-003.
3. At the beginning of the sample set run a Primer (DI water). The primer is used to prime the system and does not count as a bracketing DI water (e.g. QC sample) nor are the values of the Primer recorded for QC purposes.
4. DI water is run after the Primer, at the end of every sample set, and after no more than every 12 injections.
5. The LCS is run in duplicate (non-sequentially) with each sample set for accuracy and precision and counts as a sample for determining when to run bracketing DI water.
6. Opened LCS bottles are stored in the refrigerator and have a shelf life of 3 weeks:
    a. Before using an opened LCS bottle, verify that the opened date is within 3 weeks (21 days or less) of the current date. If the opened date is more than 3 weeks (more than 21 days) prior to the current date, properly dispose of that LCS and obtain an unopened bottle of LCS.
    b. When a new, unopened bottle of LCS is used, write the opened date and initials on the LCS bottle, wrap the top with parafilm and store in the refrigerator after use. It is also recommended to write the expiration date.
7. Care should be taken to minimize evaporation of the sample prior to instrument sampling. This is accomplished by capping each tube before placing it in the autosampler.

    NOTE: If the LCS or sample is refrigerated, allow it to warm closer to 20℃ before running on the density meter.

8. De-carbonate any carbonated samples, as per laboratory policy; see WG:SSD:1040:004.

9. A rinse/wash DI water can be run after viscous samples. Rinse/wash waters count as injections when determining where to run bracketing DI waters. Rinse/wash water values are not recorded for QC purposes.
10. Determine density and specific gravity on the density meter using the pre-set method.
11. After the final DI water that brackets samples, run two tubes of 200 proof ethanol and an additional two tubes of rinse/wash DI water to help with cleaning the system before it enters standby. Acetone can be substituted if 200 proof ethanol is not available.
12. Record all LCS values on the electronic control chart and all water densities that bracket samples on Form:Density Meter:001. Print the final sample report and place it in the appropriate logbook.

NOTE: The system will display a Filling Warning when gas bubbles are detected in the density measuring cell, usually while sampling viscous samples. It has been determined that this status does not affect the data quality of customer samples.

Quality Control

1. DI water is run after the primer at the beginning of every sample set, at the end of every sample set prior to the four cleaning injections, and after no more than every 12 injections, to check the system calibration. The density value for DI water should fall within the allowed range of 0.99810 – 0.99830.

2. Samples bracketed by passing DI water injections are reportable. If the density value of a DI water injection falls outside of the allowed range, run a verification sequence (see number 5, below).

    a. If the verification sequence results are all within the allowed ranges, rerun all non-reportable samples as part of a new sample set on the same density meter using freshly prepared tubes.
    b. If any of the verification sequence results fall outside of the allowed ranges, place an *Out of Service* tag on the instrument and inform the instrument team.

3. The LCS is prepared in duplicate and run (non-sequentially) with each sample set for accuracy and precision verification. Any LCS failure requires the initiation of a CAR. If the density of an LCS injection is outside of the control limits, run a verification sequence (see number 5, below) using the same bottle of LCS.
    a. If the verification sequence results are all within the allowed ranges, re-run the sample set on the same density meter using freshly prepared tubes.
    b. If any of the verification sequence results fall outside of the allowed ranges, place an *Out of Service* tag on the instrument and inform the instrument team.

4. Any LCS or DI water injection with a “Filling Warning” is considered to have a result outside of the allowed range. Refer to numbers 2 and 3, above, as appropriate with the exception that a CAR is not required. Per the above note, Filling Warnings for customer samples do not affect reportability of the data.
5. A verification sequence is composed of Primer (DI water), DI water, duplicate LCS, and DI water tubes, in that order. Record both DI water densities on Form:Density Meter:001 and place a printed copy of the sample report in the appropriate logbook.

Sources of Uncertainty

1. Improper cleaning or drying of instrument
2. Temperature of the sample cell (DMA)
3. Overfilling DMA tubes
4. Evaporation of ethanol due to delay in capping tubes or improperly handled samples

Calculations

Calculations are performed by the instrument software.

Reporting Results

Report density (g/mL) to five decimal places, x.XXXXX; ex. 0.99820.

Report density (lb/gal) to three decimal places, xx.XXX; ex. 10.106. Note that reporting in these units is currently only applicable to FONL samples.

Report specific gravity to five decimal places, x.XXXXX; ex. 1.00001.

Safety Notes

Normal laboratory safety protocol should be followed. High proof alcoholic products are flammable. Ethanol burns with an almost invisible blue flame.

References

1. Anton Paar Instruction Manual and Safety Information DMA 4101/4501/5001. Document # DB98IB001EN-F. Rev: April 26, 2021Issue Date: 12/22/2021.

2. Anton Paar Instruction Manual and Safety Information for Xsampler 530. Document # C91IB001EN-N Rev: April, 2021. Issue Date: 12/22/2021.

Required Training, Certification and Re-certification

1. In-house training by a certified chemist in density meter operation.

2. Initial certification is achieved by analyzing 7 LCS samples and generating results of precision and accuracy in agreement with the consensus results.

3. Proficiency testing or re-demonstration of competency every 5 years.

Revision History

Rev. 1 – Initial revision

Rev. 2 – Added new procedures for LCS storage and LCS shelf life. Made minor spelling corrections.

Rev. 3 – Updated LCS shelf life requirements.

Rev. 4 - Updated control charting from specific gravity to density. Also clarified the QC procedures for QC/LCS issues.

Rev. 5 – Removed distillation training requirement, removed determination of ethanol and clarified analysis procedures.

Rev. 6 – Removed refractive index and updated instrument information to new model. Incident Report removed from Quality Section.

Rev. 7 – Removed reference to retired document NPL:Form:103-2. Added DI water density range. Referenced electronic control charts for LCS. Brought Quality Control section more in line with TM102 for LCS failures. Added reference for degassing working guideline for samples. Added additional solvent/rinse instructions to the procedure section. Removed reference to NPL:WG:107.

Rev. 8 – Changed document IDs to the new ID structure. Changed “Co-Principal Analysts” to “instrument team”. Updated DI water naming throughout for consistency. Removed all references to the Rudolph DMA platform as it is not used for TM103. Grammatical updates for readability. Updated Procedures and QC section for clarity.