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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 the DMA 4501 is 0 to 3 g/mL.

Supplemental Documents

1. SSD:TM:102 Ethanol Determination by Specific Gravity
2. NPL:WG:104 Maintenance and Troubleshooting for NPL DMA
3. NPL:WG:106 NPL DMA/Refractometer Potentially Useful Information for Coprincipal Analysts and Other Responsible Parties
4. SSD:Form:103 Anton Paar Density Meter Daily Log
5. NPL:Form:102 Yearly Preventative Maintenance for NPL DMA
6. SSD:WG:115 Degassing Procedure

Equipment

Density Meter (Anton—Paar, DMA-4501 or equivalent), reporting specific gravity to at least 4 decimal places, temperature controlled $20.00 \pm 0.01^\circ\text{C}$

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:

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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

Procedures

1. Verify that the DI water and 200 proof ethanol (or other appropriate cleaning and drying solvents) vessels attached to the DMA contain sufficient quantity to analyze samples (ask co-principal analyst, if needed).
2. Confirm that a passing air check and water check have been performed for the week. This is recorded on NPL:Form:102 along with other weekly pre-run checks. For more information on how to run checks please refer to NPL:WG:104.
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 brackets at least every 12 injections.
5. An 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 at 1-6°C 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 and expiration date on the LCS bottle, wrap top with parafilm and store at 1-6°C after use.
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 LCS or sample is refrigerated, allow it to warm closer to 20 °C before running on the DMA.
8. De-carbonate any carbonated samples, as per laboratory policy; see SSD:WG:115.
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 on the Anton Paar DMA model 4501 using the pre-set methods.
11. After the final water blank that brackets samples, run two vials of 200 proof ethanol and an additional two vials of water to help with cleaning the system

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before it enters standby. Acetone can be substituted if 200 proof ethanol is not available.

12. Please record all LCS values on the electronic control chart and all water densities that bracket samples on SSD:Form:103. 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 when sampling viscous samples. It has been determined that this status does not affect the quality of the data from the DMA for NPL purposes up to and including glycerol with a viscosity of 1.2 PA*s.

Quality Control

1. DI water is run after the primer at the beginning and end of every sample set, and brackets at least every 12 injections, to check the system calibration. The density value for water should fall within the allowed range; 0.99810 – 0.99830.
2. Samples bracketed by passing DI water are reportable. If the density value of a water falls outside the allowed range, run at least three DI waters and record their values in the logbook. If the three DI waters are within the allowed range, the data is reportable. If these values fall outside the allowed range, stop using the instrument, place an *Out of Service* sign on the instrument, and inform the co-principal analysts.
3. Duplicate LCS is run (non-sequentially) for accuracy control charting. The duplicate values for density need to fall within the allowed ranges; see the TM103 electronic control chart.
4. Any LCS or sample with a “Fill Warning” should be re-analyzed.
5. If the density of the LCS fails:
 - i. Run the same bottle of LCS at least three times and print the instrument report; evaluate the accuracy.
 - ii. If all of the values fall within the allowed range, rerun the sequence and initiate a CAR.
 - iii. If any of the values fall outside the allowed range, stop using the instrument, place an *Out of Service* tag on the instrument, inform the co-principal analysts and initiate a CAR.
 - iv. Record all activity in the appropriate instrument logbooks.

Sources of Uncertainty

1. Improper cleaning or drying of instrument
2. Temperature of the sample cell (DMA)

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3. Overfilling DMA tubes
4. Evaporation of ethanol due to delay in capping tubes or improperly handled samples

Calculations

Calculations are done by the instrument software.

Reporting Results

Report density to four decimal places, x.XXXX.

Report specific gravity to four decimal places, x.XXXX.

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, 2021 Issue 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 DMA operation.
2. Initial certification is achieved by analyzing 7 LCS samples and generating results of precision and reproducibility 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.

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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.

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