

Use of Anton Paar density meter model 4500 or 5000
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SCOPE and PURPOSE:

This SOP provides a method for using, adjusting, and cleaning the Anton Paar Density meter model 4500 OR 5000 for wine and related analysis. Density and density-related analyses (specific gravity, Brix, concentration, etc) are used in wine and wine-related testing. Temperature is an important parameter of most of these tests.

NONCONFORMANCE:

Consult the Anton Paar manual for any situation not discussed in this SOP

MRO:

- Anton Paar model 4500 or 5000 density meter
- Barometer
- Air Humidity measure
- 2 mL (or larger) luer tip syringes
- Boiled and cooled distilled or deionized water for checking or adjusting
- Distilled or deionized water for cleaning
- 190 Proof food grade grain alcohol (AAPER)
- Q value adjustment report page
- Anton Paar DMA 4500/5000 Manual
- 1% Luminox (from Alconox) or 1% Aquet (from Scienceware) cleaner for bi-monthly cleaning.

SOLUTION PREPARATION

Boiled and cooled distilled or deionized water for calibration

1. Boil fresh, bi-distilled water for several minutes to remove dissolved gasses.
2. Fill a clean glass flask full with the boiled water and cover it
3. Wait until the water has cooled to the approximate measuring temperature before using it.
4. If prepared in full containers and covered to prevent gas ingress solution can be prepared in advance.

SAMPLE PREPARATION

1. Samples must be stored in a closed container prior to analysis. Immediately prior to analysis, swirl the wine gently in the container to rinse the sides of any condensate which may be present and to incorporate that condensate back into the sample.
2. Samples must be homogeneous and free of gas bubbles. Wine that is clear and does not contain carbon dioxide may be measured without further preparation.
3. If the sample to be measured tends to form bubble, the substance should be degassed before the measurement. If this is not possible then introduce the sample at a temperature higher than the measuring temperature.
4. Wines that contain carbon dioxide must be degassed. Many degassing methods may reduce the alcohol content. Ultrasonication, centrifugation, or non-vacuum filtration are some options to try.
5. Turbid samples should be centrifuged or non-vacuum filtered to remove solids.
6. Pre-thermosetting the sample reduces the measuring time

PROCEDURES

DENSITY CHECK (PERFORM EACH DAY THE INSTRUMENT IS USED)

1. Make sure there is a waste bottle at the outlet of the measuring cell.
2. Fill a luer syringe with bi-distilled, freshly degassed water.
3. Attach the syringe to the filling nozzle and inject the sample into the measuring cell by pushing the plunger of the syringe slowly and continuously until a drop emerges from the other nozzle.
4. Observe the filling through the inspection window. Take care that the entire measuring cell is filled with sample. A minimum of 1.5 mLs is required.
5. Leave the syringe in the filling position, in order to prevent sample leaking.
6. Ensure that there are no gas bubbles in the measuring cell.
7. Select "adjustment", then "density check", "check density".
8. If the measured density is within the permitted range, the display shows "density check: OK". Routine measurements can be carried out.
9. If the measurement density is out of range, the display shows "density check NOT OK". If this occurs, CLEAN AND DRY the measuring cell (below) and check the density again.
10. If the density check is still NOT OK, then perform a NEW ADJUSTMENT (below).

SAMPLE MEASUREMENT

1. Make sure there is a waste bottle at the outlet of the measuring cell.
2. Activate the desired method using the "method" soft key ("density" with a measuring temperature of 20 C are default settings. NOTE for CTS TESTING: density and specific gravity are NOT the same!) Other commonly used settings might include "Brix" (at 20C), "OIML v/v" (ethanol conc at 20C).
3. Fill a luer syringe with sample.
4. Attach the syringe to the filling nozzle and inject the sample into the measuring cell by pushing the plunger of the syringe slowly and continuously until a drop emerges from the other nozzle.
5. Observe the filling through the inspection window. Take care that the entire measuring cell is filled with sample. A minimum of 1.5 mLs is required.
6. Leave the syringe in the filling position, in order to prevent sample leaking.
7. Ensure that there are no gas bubbles in the measuring cell.
8. Print or record your results.
9. Fill the chamber with the next sample or CLEAN AND DRY (see below) the measuring cell.

TO CLEAN AND DRY THE CELL

1. Check the air humidity. If the relative humidity (RH) is higher than 70% (at 20C), or 50% (at 25%), or 38% (at 30C) adjustment and measurement errors may occur.
2. Fill the measuring cell with 1 syringe full of DI water. Move the plunger of the syringe in and out several times to create gas bubbles which improve the cleaning action.
3. Remove the water from the measuring cell
4. Fill the measuring cell with 1 syringe full of 190 P ethanol. Move the plunger of the syringe in and out several times to create gas bubbles which improve the cleaning action.
5. Remove the ethanol from the measuring cell.
6. Attach the air hose to the filling nozzle
7. Turn on the air pump using the PUMP key
8. Let dry air (see step 1 above) blow through for approximately 10 minutes
9. Turn off the air pump.
10. Remove the air hose from the filling nozzle.

TO SET OR ADJUST THE DENSITY CHECK PARAMETERS

1. Select "adjustment", then "density check", then "density check settings".
2. Make the setting correspond to the density calibration fluid:
3. FOR WATER
 - a. *Density:*
 - i. For DMA 5000 water density is 0.998203
 - ii. (the DMA 4500 the density of water is 0.99820).
 - b. *Max density deviation*
 - i. For DMA 5000 = 0.00005 g/cm³
 - ii. (the DMA 4500 = 0.00010 g/cm³)
 - c. *Temperature*
 - i. "20.00 C"
 - d. *check interval:*
 - i. "1 day" (this is the recommended setting, but the interval can be set from 1 to 999 days).
 - e. *check density:*
 - i. "on" (this triggers the machine to tell you to check density at the interval set above).
4. Press the "Exc' key to return to the "density check "menu.

MAKING NEW ADJUSTMENTS USING AIR AND WATER AT 20 C

This procedure takes between 5 & 10 minutes if the instrument is already at 20 C

1. CLEAN AND DRY THE MEASURING CELL (SEE PROCEDURE ABOVE)
2. Press the "menu" key and select the menus by using the "up" and "down", and back arrow keys:
 - a. "adjustment", ",", then
 - b. "adjust", then
 - c. "density (air, water)'
3. Start the adjustment by pressing the "OK" key.
4. Press the back arrow key and enter the **current air pressure** (FROM THE BAROMETER) using the "up", "down", "left", "right", and back arrow keys.
5. Wait until the air adjustment is finished. Write down the current Q value in the adjustment report
6. Fill the measurement cell with bi-distilled, freshly degassed water, checking for the presence of bubbles through the inspection window.
7. Start the water adjustment by pressing the "OK" key.
8. Wait until the water adjustment is finished. Write down the current Q value of water in the adjustment report. The deviation of the new adjustment from the last adjustment performed is displayed at a density of 1 g/cm³.
9. If "recommendation: SAVE" is displayed, select "SAVE" to save this adjustment.
10. If "recommendation: REPEAT" is displayed, repeat from step 1 above. If the deviation remains unchanged, the adjustment can be stored by selecting "SAVE".

BIMONTHLY OR MAJOR CLEANING

1. Fill the measuring cell with 1 syringe full of DI water. Move the plunger of the syringe in and out several times to create gas bubbles which improve the cleaning action.
2. Remove the water from the measuring cell.
3. Fill the measuring cell with 1 syringe full of 1% cleaning solution (luminol or aquet). Leave the cleaning solution in the cell for several minutes to take effect. Move the plunger of the syringe in and out several times to create gas bubbles which improve the cleaning action.
4. Remove the cleaning solution from the measuring cell.
5. Repeat steps 1 and 2 at least five times (a thorough DI water rinse).
6. Fill the measuring cell with 1 syringe full of 190 P ethanol. Move the plunger of the syringe in and out several times to create gas bubbles which improve the cleaning action.
7. Remove the ethanol from the measuring cell, Attach the air hose to the filling nozzle, and Turn on the air pump using the PUMP key.
8. Let dry air (see step 1 above) blow through for approximately 10 minutes.
9. Turn off the air pump and Remove the air hose from the filling nozzle.