



Application Note

AN 1030.00

Measuring Hot Liquid Samples

“When a material is in the form of a solid, powder or crystal, detecting small color differences can be made easier by dissolving the sample in a solvent or heating it to a liquid.”

ABSTRACT

When a sample is in a powder or crystalline form, particularly when highly white, small color differences are lost in the optical reflection and refraction from all the particulate surfaces of the sample. By dissolving or heating into a liquid form, all of this “optical noise” is eliminated and small color differences can be seen best and measured easily in transmission.

HunterLab has a solution for heating materials to a liquid state in a round vial outside the instrument; then transferring the vial to a vial holder using a pair of tongs. The transmission color measurement of the hot liquid sample is immediately taken, and the hot vial removed.

Necessary components for this hot vial technique are:

- HunterLab **ColorQuest XE, ColorQuest XT or UltraScan VIS diffuse d/8° sphere instruments** for transmission measurements.
- HunterLab **D02-1011-550 Vial Holder** to consistently hold and center the round vials in the instrument.
- **A13-1015-361 60 ml Molded Glass Vials 24 mm ID** serve as transmission cells for heating and measuring the sample color.
- **A13-1015-465 Heating Mantle**, 110V and **A13-1015-466 Temperature Controller**, 110V (220V options available) to heat a hot liquid sample to an optimal temperature range of 100°C to 250°C.
- **CMR-3079 EZMQC APHA applet for 24 mm Glass Vials** or **CMR-2958 APHA/Gardner applet for 24 mm Glass Vials** allows the display of an instrumental measurement of APHA/Gardner Color in disposable 24 mm ID glass vials that allow heating and capping for volatiles.

COLOR MEASUREMENT METHOD FOR HOT LIQUIDS

1. Install the HunterLab D02-1011-550 Vial Holder in the transmission compartment of the instrument at the TTRAN (Total Transmission) port. There are adjustment pins on the sides and bottom of the holder to ensure that the vial fits tightly in the holder.
2. Put material (powder, pellet, granule or flake) into vial. You will have to empirically determine an appropriate sample weight such that when liquid, the sample completely covers the TTRAN port.
3. Place vial in a heating device and heat sample until a target temperature is reached where the sample is in a fully liquid state. If there are any undissolved solids, you may have to filter them out.
4. Standardize the HunterLab sphere sensor in the TTRAN LAV mode using a light blocker to set the bottom-of-scale (0 % transmission) and the 60 ml vial filled with distilled water and placed in the vial holder to set the top-of-scale (100 % transmission; the white tile will be placed at the reflectance port to fill in the sphere for standardization and all measurements).
5. As a PQ (Performance Qualification) step, read back the distilled water in the vial as a sample. The expected L^* , a^* , b^* color values should be 100.0, 0.0, 0.0, APHA 24 mm = 0, Gardner 24 mm = 0 within close agreement.
6. Using a pair of tongs, lift the hot liquid vial from the heating device and transfer it to the vial holder in the instrument transmission compartment. The vial will stick out of the top.
7. Draw the transmission door close to the cell. Ambient lighting will not affect measurements made using the pulsed xenon flash lamp. The transmission door does not have to be completely closed.
8. A single sample reading is usually sufficient. Averaging 2 readings per measurement, turning the cell approximately 180 degrees between each reading, is best.
9. Report color values for the sample.

DETAILED COMPONENT DESCRIPTION

1. HunterLab D02-1011-550 Vial Holder for 27-30 mm Round Cell or Vial

The D02-1011-550 vial holder positions and centers the curved surface of the round glass vials consistently for measurement at the TTRAN port. In the TTRAN LAV mode, a 17.4 mm diameter area is imaged of these 30 mm OD vials. If the instrument has an SAV option, this will image a 12.5 mm diameter area in the center of these 30 mm ID vials.

This holder accepts all round glass vials with outside diameters from 27 mm to 30 mm. There are 4 user-adjustable screws in the holder that can be adjusted to match the outside diameter of the round vial to ensure that the vial is solidly placed in the holder.

2. A13-1015-361 60 ml Molded Glass Vials 24 mm ID

Clear, round 24 mm (0.94 in) ID, molded, borosilicate glass vial with dimensions of 27.5 mm OD x 140 mm high (1.08 in OD x 5.51 in high) that requires a minimum 20 ml of sample for measurement but can hold up to 60 ml. This size is easy to grasp with tongs and can be heated to temperatures well above 250°C. Comes with black polypropylene Screw-on caps with a PTFE disc liner to contain volatiles and prevent spills. Requires D02-1011-550 Transmission Holder for 24 mm ID Round Vial for measuring in TTRAN (preferred) or RTRAN transmission on HunterLab sphere instruments.

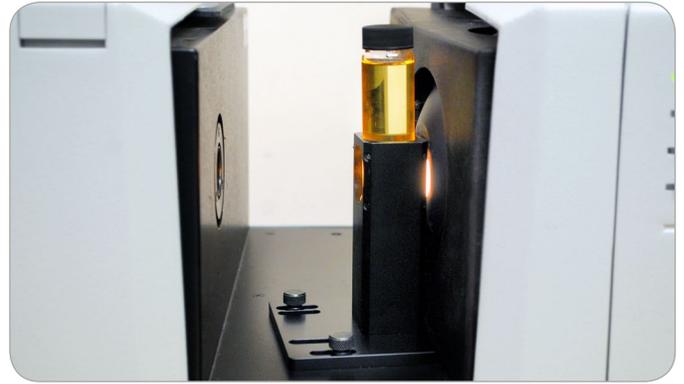


Figure 1. 60 ml Heated Glass Vial (A13-1015-361) in HunterLab Vial Holder (D02-1011-550) for TTRAN Transmission Measurement

- The vials come in different height/volumes with the same OD to fit the holder. The 60 ml size works best for measuring the color of hot liquids, as the extra height allows the tube to be more easily grasped by a set of tongs.
- These round tubes work well when the materials must be centrifuged for mixing or separating, or if the materials need to be heated. The polypropylene caps and molded borosilicate glass are autoclavable. These glass vials are cost effective for disposal or recycling. They can also be cleaned as appropriate and re-used. The black polypropylene caps can be heated to 110°C (230°F) and they will not be in direct contact with the heating mantle. The glass vials can be heated to around 400°C, which is higher than any application should require.

Additional vial options include:

- Polypropylene caps in solid polypropylene and injectable silicone septa are available assembled with the vials, or separate.
- A variety of internal cap liners can also be chosen as appropriate for the material being heated. These include metal, foamed polyethylene, polyethylene, vinyl and Teflon (minimal chemical reaction) disc liners.
- Individual barcode labels can be affixed for sample identification.
- There are a variety of other cleaning procedures (vacuum ionization, particulate washing and gamma sterilization) that can be done to ensure that there is no residual particulates (dirt, dust, carton lint, fine glass particulates, bacteria) or film (bottle release agents, aerosols, oil) on the glass surface. The most cost effective to order is vials that are cleaned but do not have a certificate enclosed with each box. This provides an EPA-cleaning without the additional costs of certification.

3. Heating Device

It does not matter how you heat the sample in the vial. In general, three levels of heating:

- Temperatures from ambient to 100°C (boiling water), use a water bath available from any laboratory supply store.
- Temperatures from 100°C to in excess of 250°C, HunterLab offers a heating mantle (3a) and temperature controller (3b).
- Temperatures above 300°C to 400°C, typically a hot air furnace is used. The borosilicate glass of the cell will melt at 450°C.

3a) A13-1015-465 Heating Mantle, 110V

Cylindrical Heating Mantle 102B 000200121 with aluminium insert (7.5 in dia x 7.5 in high) having 4 round openings (28 mm or 1 5/16 in OD) with a 100 mm depth (4 in). It is designed to heat materials in the 60 ml Molded Glass Vials (A13-1015-361) used in hot liquid color applications. Requires 110 volt line source. Used with A13-1015-466 Mantle Temperature Controller, 110V, to control and maintain the target temperature of the sample. Also available - A13-1015-463 Heating Mantle, 220V.

3b) A13-1015-466 Temperature Controller, 110V

can be set to automatically raise the temperature of materials to a pre-defined level up to 250°C, and hold it to within +/- 1 degree C. Requires 110 volt line source. Used with A13-1015-465 Heating Mantle, 110V. Also available - A13-1015-464 Mantle Temperature Controller, 220V.

FAQ: "I notice you have three different Molded Glass Vials available - A13-1015-359 (20 ml), A13-1015-360 (40 ml) and A13-1015-361 (60 ml). They appear to come in three height/volumes. Do they have the same OD or the same ID?"

There are 3 sizes of Molded, Round Glass Vials that we are using as transmission cells – 20 ml (small), 40 ml (medium) and 60 ml (large) volumes. The ID – inside diameter is the same – 24 mm. The OD – outside diameter varies a little with the vial selected. They are made in volume and that's just the way they are. The key dimension for our use of this round vial as a transmission cell, is the inside diameter (ID) which is consistent at 24 mm. All 3 vial types fit in the same D02-1011-550 Transmission Holder.

The 40 ml (1 1/3 oz) borosilicate glass vial with a 27.5 mm OD and 95 mm height is recommended for general use as a round disposable glass transmission cell on HunterLab sphere instruments. There is a 20 ml (2/3 oz) glass vial with a 27.25 mm OD and 57.5 mm height which is suitable for small sample volumes. These vials will fit in the transmission compartment of HunterLab sphere instruments with the door closed.

There is also a 60 ml glass vial with a 27.5 mm OD and 140 mm height that fits above the door for exterior venting or external loading if required. The 60 ml size works best for measuring the color of hot solutions, as the extra height allows the tube to be more easily grasped by a set of tongs or hot mitts.

FAQ: "The 60 ml glass vial with a 27.5 mm OD and 140 mm height fits above the transmission door for exterior venting or external loading if required. Does it mean the 60 ml glass vial is too tall, so when we measure the sample, can we leave the transmission compartment door open?"

You can close the transmission door with the 20 ml and 40 ml vials. Given the height of the 60 ml vial, it will stick out above the door. Just pull the door close to the 60 ml vial even if the door isn't completely closed. The pulsed xenon flash of the instrument is so powerful that the ambient room light will not affect the reading.

FAQ: "Can the sample be heated inside the HunterLab sensor or should the customer heat the liquid sample outside the sensor and then transfer the sample into the transmission compartment?"

In general, heat is never good for sensors. The sample should always be heated in the vial outside the instrument, then be transferred to the D02-1011-550 Transmission Holder for the brief few seconds it takes to make a measurement.

FAQ: How can we clean the glass vials? Are the vials robust enough for scrubbing?"

The glass vials are molded and will stand up to cleaning and re-use many times. They are also inexpensive and can be used once if cleaning is difficult.

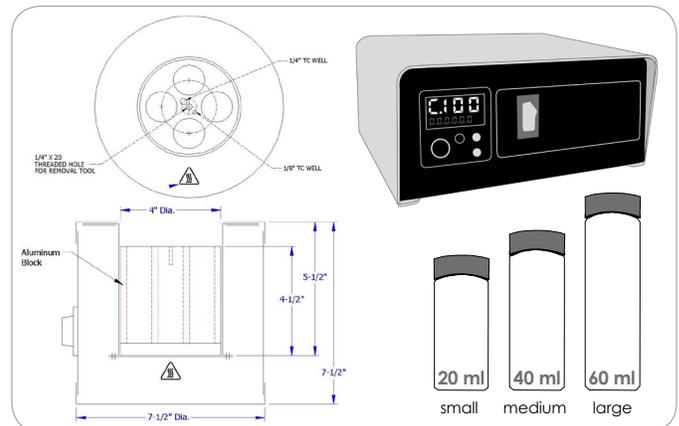


Figure 2. Schematic of the A13-1015-465 Heating Mantle, Temperature Controller and different sizes of glass vials

FAQ: If the sample is volatile when heated, are the Molded Glass Vials safe enough?"

While not designed to contain extreme pressure, these vials are very robust and should not be affected by some pressure generated in heating.

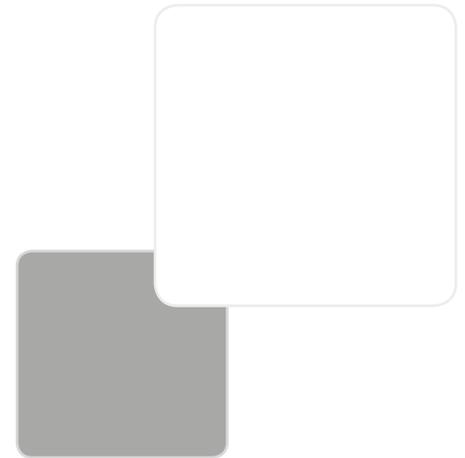
FAQ: "When the customer needs to heat a liquid prior to measurement, should A13-1015-463 Heating Mantle be used with A13-1015-464 Mantle Temperature Controller? Can the customer use the Heating Mantle alone but no Mantle Temperature Controller?"

The Heating Mantle can be used alone but the customer would have to watch to see if the material had melted, and it is possible to overheat the sample. The controller allows the customer to dial in a target temperature, put the probe in a sample vial and the controller will heat to that temperature and keep it there within +/- 1 degree C. We only sell the two pieces together – mantle and controller as this is the best way to heat and liquefy these crystal or powder samples consistently and efficiently.

Clients can heat materials in the vial using any heating method that works for their application.

FAQ: "The A13-1015-463 Heating Mantle has 4 round openings, so we can heat 4 samples at the same time?"

Yes, from 1 – 4 samples can be heated at a time. If you are running higher production than this, purchase a second mantle or use alternative methods of sample heating.



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Color Measurement on our
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ABOUT HUNTERLAB

HunterLab, the first name in color measurement, provides ruggedly dependable, consistently accurate, and cost effective color measurement solutions. With over 6 decades of experience in more than 65 countries, HunterLab applies leading edge technology to measure and communicate color simply and effectively. The company offers both diffuse/8° and a complete line of true 45°/0° optical geometry instruments in portable, bench-top and production in-line configurations. *HunterLab, the world's true measure of color.*

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