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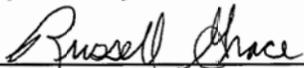
Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division

MLD SOP SAS03

**STANDARD OPERATING PROCEDURE FOR THE KARL
FISCHER (KF) DETERMINATION OF WATER WITH KF
DRYING OVEN IN CONSUMER PRODUCT**

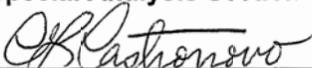
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Date

DISCLAIMER: Mention of any trade name or commercial product in Method 310 and associated Standard Operating Procedures does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedures are equipment used by the ARB laboratory. Any functionally equivalent instrumentation can be used.

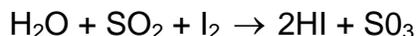
1 INTRODUCTION

This procedure is used for the measurement of water in consumer products and is based on the Karl Fischer (KF) procedures specified in ASTM D 4017-96 and ASTM D 3792-91. Water is determined by using commercially available standard Karl Fischer reagent integrated with a drying oven. The use of trade names or commercial products are examples only, and any equivalent products may be substituted.

2 SUMMARY OF METHOD

The principle of the method involves heating a sample aliquot diluted in 1-Methoxy-2-propanol (MPA) in an oven, where the moisture from the sample is carried from the oven into a titration vessel by a stream of dry, inert carrier gas (or dried ambient air). The moisture in the titration vessel is titrated continuously until the designated endpoint is reached. Although traditional direct injection sample introduction may be appropriate for some products, the use of the oven provides more consistent and more precise values when interferences are present (see references 8.1 and 8.2).

The traditional KF reagent contains iodine, sulfur dioxide in pyridine and methanol. New pyridine-free KF reagents use amines and glycol ethers to replace the pyridine and methanol. The iodine in the presence of water is reduced to colorless hydrogen iodide. The end point is the appearance of free iodine. The basic reaction of the KF reagent with water is given as:



The method requires the dilution of a pre-weighed aliquot of the consumer product with MPA. The solvent, MPA, is completely miscible with water forming an azeotrope boiling at 97.5°C. As the water in the sample is heated in the oven it is transferred quantitatively to the titration vessel as the azeotrope, where it is titrated.

If a sample does not mix with MPA analyze the sample following the procedure for an instrument check except using a smaller sample size.

3 INTERFERENCES/LIMITATIONS

Interferences in the titrimetric water determinations are associated with condensation or oxidation-reduction reactions with a number of substances and compounds (for more information refer to ASTM E 203 "Standard Test Method for Water Using Karl Fischer Reagent" 1986).

Use of certain reagents will minimize or eliminate the interferences of many classes of compounds. For example the use of non-methanol containing KF reagent and solvent will reduce the interference from aldehydes and ketones. Ammonia and amines can be eliminated by the addition of salicylic acid to the solvent.

Other possible interferences to the KF reagent are certain active metals, metal oxides, metal hydroxides, chromates, melamines, etc. (Ref. 8.1 and 8.2).

4 APPARATUS AND MATERIALS

- 4.1 Karl Fischer Titration System:
 - 4.1.1 Mitsubishi Moisturemeter, KF-200,
 - 4.1.2 Mitsubishi Sample Changer, VA-236S,
 - 4.1.2.1 Oven Temperature Setting: 130°C;
- 4.2 Volumetric Flasks, 10mL;
- 4.3 Pipettors:
 - 4.3.1 Rainin, electronic: 250 μ L and 100 μ L - 1.0 mL, with tips;
- 4.4 Syringe, 50 μ L, Hamilton;
- 4.5 Vials, 8 mL with PTFE-lined cap;
- 4.6 Analytical Balance, Sartorius MC1 or Mettler XP 205:
 - 4.6.1 capacity of 100 g x 0.00001 g (readability);
- 4.7 Headspace Vials, with 16mm screw top and PTFE/sil septa, 10 mL.

5 REAGENTS

- 5.1 Deionized Water, reagent grade, ASTM Type 1, 18 M Ω ;
- 5.2 1-Methoxy-2-propanol (MPA), 99.5%;
- 5.3 Disodium Tartrate Dihydrate (tartrate), water content = 15.61-15.71%;
- 5.4 Pyridine-free KF titration reagent for aldehydes and ketones, 1.0mL = 5mg water:
 - 5.4.1 Hydranal Comp 5K, 1L, or
 - 5.4.2 EMD AquaStar CombiTirant 5 Keto, 1L;
- 5.5 Titration solvent for volumetric KF titration in ketones and aldehydes:
 - 5.5.1 Hydranol Ketosolver, 1 L, or
 - 5.5.2 EMD AquaStar CombiSolvent Keto, 1L;

- 5.6 Nitrogen, compressed, ultra high purity;
- 5.7 Control/Check Stock Solution. The control/check stock is a 25% acetone/water solution prepared by weighing 50g each of acetone (99.9%) and water into a 200mL volumetric flask and bringing to volume with MPA. The analytes are weighed in the preparation of the stock, so the concentration is in g/mL. Place the control/check stock in screw capped vials and store in the Standards refrigerator.
- 5.8 Trip Sample Stock. Combine 300g of water and 50g each of sodium chloride (ACS 99.0%), acetone (99.9%), methanol (99.9%), and ethanol (200 proof) into a 500mL volumetric flask. The analytes are weighed in the preparation of the stock, so the concentration is in g/mL. Place the trip sample stock in screw capped vials and store in the Standards refrigerator.

6 PROCEDURE

6.1 Instrument Preparation:

- 6.1.1 Turn on both KF-200 and VA-236S KF units,
- 6.1.2 Verify instrument is operational (see Appendix A, Section 1);

6.2 Analysis Preparation:

- 6.2.1 Conditioning Vial. Cap three empty headspace vials.
- 6.2.2 Air Blank: Only necessary when running instrument check. Prepare three air blanks by capping three empty headspace vials.
- 6.2.3 Instrument Check: An instrument check is performed once a week. Prepare two instrument checks by capping two headspace vials, each containing 0.200 g of tartrate. Record the actual weights of the tartrate.
- 6.2.4 Solvent Blank: Prepare three solvent blanks by capping three headspace vials, each containing 250µL of the same MPA used to make the dilutions in part 6.2.5 – 6.2.7.
- 6.2.5 Control/Check: Prepare the control/check by aliquotting 1.0mL of the control/check stock standard into a 10mL volumetric flask, and bring to volume with MPA. Take four headspace vials and pipette 250µL of the dilution into each. Cap the vials. Two of these vials will serve as the control. The remaining two will serve as the check.
- 6.2.6 Trip Sample: Prepare a trip solution of 60% H₂O by aliquotting 1.0 mL of the trip stock standard into a 10mL volumetric flask, and bring to volume with MPA. Take two headspace vials and pipette 250µL of the trip

solution into each. Cap the vials.

6.2.7 Sample: Weigh a 1.0mL aliquot of the consumer product sample into a 10mL volumetric flask. Record the weight of the aliquot and bring to volume with MPA. Take two headspace vials and pipette 250 μ L of the prepared dilution into each. Cap the vials. Transfer the remaining dilution to an appropriately labeled 8 ml screw capped storage vial. If a sample does not mix with MPA analyze the sample following the procedure for an instrument check (Appendix A, Section 3) except using 0.1 g of sample.

6.3 Water Analysis:

6.3.1 Perform water titer (Appendix A, Section 2) to set the calibration factor for subsequent analyses. The KF water titer (mg water per mL of titrant) is determined by directly injecting 25 μ L water into the titration vessel.

6.3.2 Perform instrument check (Appendix A, Section 3) on a once-a-week basis to verify the proper operation of the KF instrument. Air blanks will also be run with this analysis.

6.3.3 Perform sample analysis (Appendix A, Sections 4 - 8) to determine water content. The amount of water in the consumer product sample is calculated by averaging the percent water results of two replicates per sample. Solvent blanks will also be run with this analysis. The average water content of the solvent blanks is automatically subtracted from the sample results.

7 QUALITY CONTROL

7.1 The sensitivity, precision, and accuracy will depend on several factors, particularly the nature of the consumer product material being analyzed.

7.2 All consumer product sample analyses are done in replicate and should not have an absolute difference greater than $\pm 2\%$.

7.3 Control charts of the disodium tartrate and the 25% control/check solution are made with the upper and lower control limits set at $\pm 3s$ of the historical value. If an analysis is out of the control limits, the conditions are evaluated and the control and any affected samples will be re-analyzed.

7.4 A trip sample of 60% water is run with each sample set. The recovery for the trip sample should be within the error of the method ($\pm 3\%$).

7.5 LIMS assigns at least one duplicate sample for every sample set. Duplicate analysis should not have an absolute difference greater than $\pm 3\%$.

8 REFERENCES

- 8.1 ASTM E 203 "Standard Method for Water Using Karl Fischer Reagent" 1986.
- 8.2 ASTM D 4017 "Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method" (EPA Method 24).
- 8.3 Jenkins, V.C., Reilly, Joseph C., Sypowicz, Bob, and Wills, Max T. "VOC Testing Comparison: EPA Method 24 Versus the Cal Poly Method" Journal of Coatings Technology 67 (84), 53-59 (1995).
- 8.4 Metrohm 784 KFP Titrino Instructions for Use. Metrohm 774 Oven Sample Processor Instructions for Use.
- 8.5 Brinkmann Lab, "Karl Fischer Water Determination with the KF Drying Oven" Applications Bulletin No. 109/1e.

Appendix A

OPERATION INSTRUCTIONS FOR KF SAMPLE PROCESSOR

1. INSTRUMENT START UP

- 1.1 Turn on the VA-236S Sample Changer.
- 1.2 Turn on the KF-200 Moisturemeter.
 - 1.2.1 A message will be displayed with **OK** at the end, push Enter.
- 1.3 Push **Parameter/Character**.
- 1.4 Push appropriate arrow key to get to **01 H2O Titer**.
- 1.5 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.
- 1.6 Push **Escape**.
- 1.7 On the VA-236S push the **Heater** button, the heater light should now be on and the analysis temperature is 130°C.
- 1.8 Check levels of the liquids.
 - 1.8.1 Adjust the level of liquid in the reaction vessel to ~75mL, the blue lines indicate 50mL intervals.
 - 1.8.1.1 Draining the reaction vessel.
 - 1.8.1.1.1 Pull up on the drain line stopper so it is easy to lift out, keep drain line below liquid.
 - 1.8.1.1.2 Push down on the switch toward the back left side of the magnetic stirrer until the volume desired is reached.
 - 1.8.1.1.3 Stop pushing down on the switch and pull up the drain line so the bottom is above the liquid.
 - 1.8.1.1.4 Once you are sure the siphon is broken, put the drain line stopper back in its opening.
 - 1.8.1.2 Adding Ketosolver to the reaction vessel.
 - 1.8.1.2.1 Push up on the switch toward the back left side of the magnetic stirrer until the volume desired is reached.

- 1.8.1.2.2 Stop pushing on the switch.
- 1.8.2 Make sure there is enough Comp 5K.
- 1.9 On the PC double left click on the *DC-100 Data Collection* icon (lower left).
- 1.9.1 It will display a table. The top entry should be *KF-200*.
- 1.9.2 The Comport should be *Com1* for Mitsu #1 & 3 and *Com3* for Mitsu#2.
- 1.9.3 Left click on *OK*.
- 1.9.4 Comes up with a message about connecting, left click on Connect.
- 1.9.5 Displays the data capture screen.
- 1.10 Make sure the stirrer speed is between 3 and 4, closer to 4 is better. Check this each time you drain the reaction vessel.

2. WATER TITER

- 2.1 On KF-200 push **Sample**.
- 2.1.1 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.
- 2.1.2 Push **Escape**.
- 2.2 Push **Titration** – wait for **Stable** (upper left of display), it will beep three times.
- 2.3 Push **Start/Stop**.
- 2.3.1 When it displays **Add Water** in blue area at bottom of display inject 25uL of water through the white plug in the front on top of the reaction vessel.
- 2.3.2 It will beep and transfer the results to the PC when done. Print those results.
- 2.4 On KF-200 push **Sample**.
- 2.4.1 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.
- 2.4.2 Push **Escape**.

- 2.5 Push **Start/Stop**.
- 2.5.1 When it displays **Add Water** in blue area at bottom of display inject 25uL of water through the white plug in the front on top of the reaction vessel.
- 2.5.2 It will beep and transfer the results to the PC when done. Print those results.
- 2.6 Repeat 2.4 and 2.5 one more time each for a total of three injections.
- 2.7 The average should be around 5.

3. INSTRUMENT CHECK (“Tartrate”)

The instrument check is a weekly verification of the KF instrument performance. The instrument check uses both the prepared tartrate and air blank headspace vials. This procedure is also used to analyze samples that do not mix with MPA.

- 3.1 Sample transfer line.
 - 3.1.1 On top of the reaction vessel remove the glass stopper (wipe off with Kimwipe) and put it into the 50mL beaker near the front of the VA-236S Sample Changer.
 - 3.1.2 Remove the sampler transfer line from the tube behind the reaction vessel and insert it from where you just removed the glass stopper.
- 3.2 Push **Parameter/Character**.
 - 3.2.1 Use appropriate arrow key to get to **04 Blanks**.
 - 3.2.2 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.
 - 3.2.2.1 Check the water titer value, if incorrect enter the correct value.
 - 3.2.3 Push **Escape**.
- 3.3 Prepare samples.
 - 3.3.1 Recap air vials, make sure ambient air gets into them.
 - 3.3.2 Recap any pierced “condition” vials in tray positions P1-3.
 - 3.3.3 Weigh out 0.200g of tartrate into 2 vials.

- 3.3.4 Put air vials in positions 1-3 and the tartrate vials in positions 4 and 5.
- 3.4 Push **Sample**.
- 3.4.1 Use arrow keys if necessary to select **01 Groupings**.
- 3.4.1.1 Push **Enter**.
- 3.4.1.2 Make sure **01 GO1 Start:** is **1**, then push **Enter**.
- 3.4.1.3 Make sure **02 GO1 End:** is **3**, then push **Enter**.
- 3.4.1.4 Make sure **03 GO1 File:** is **4**, then push **Enter**.
- 3.4.1.5 Make sure **04 GO2 Start:** is **4**, then push **Enter**.
- 3.4.1.6 **05 GO2 End:** for normal tartrate would be **5**. If doing more, increase the number by 2 for each additional sample (for 3 additional samples it would be 11, etc.), then push **Enter**.
- 3.4.1.7 For **06 GO2 File:** push **2**, then push **Enter** for Tartrate.
- 3.4.1.8 If **07 GO3 Start:** is blank go to 3.4.1.9.
- 3.4.1.8.1 Push **Clear**, then **Enter**, repeat until entries are blank.
- 3.4.1.9 Push **Escape**.
- 3.4.1.10 *Optional: Printing out the Groupings list.*
- 3.4.1.10.1 *Use arrow keys to select **01 Groupings**.*
- 3.4.1.10.2 *Push **Enter**.*
- 3.4.1.10.3 *Push **Print**.*
- 3.4.2 Use arrow keys to select **02 SampleName, No, W, w**.
- 3.4.2.1 Push **Enter** – this starts sample information entry.
- 3.4.2.2 Can push the **Clear** key to “clear out” an entry and can use the arrow key to move to, then change characters at and after the spot moved to.
- 3.4.2.3 **SampleName** will be **B** for Blanks (Air & MPA), **T** for Tartrate, **C** for Controls, **TS** for Trip Samples, **CH** for Checks, and the sample number for samples.

- 3.4.2.4 **No** will be the replicate number, 1-3 for Blanks and 1-2 for all others.
- 3.4.2.5 **W** is the dilution weight for samples and 1 for all others.
- 3.4.2.6 **w** is to be left blank.
- 3.4.2.7 Make sure you push **Enter** after every entry.
- 3.4.2.8 After completing the number of entries from 3.4.1.6 you will get the red line and a longer beep.
- 3.4.2.9 Push **Escape**.
- 3.4.3 *Optional: Printing out the Sample list.*
 - 3.4.3.1 Use arrow keys to select **02 SampleName, No, W, w**.
 - 3.4.3.2 Push **Enter**.
 - 3.4.3.3 Push **Print**.
- 3.4.4 Push **Escape**.
- 3.5 Start the autorun.
 - 3.5.1 Check the temperature, if not 130 “reload” the method (3.2).
 - 3.5.1.1 If “reloading” the method does not work you can manually set the temperature through the VA-236S Sample Changer.
 - 3.5.1.2 Push **Function**.
 - 3.5.1.3 It displays **1. TEMP SETTING** so push **Enter**.
 - 3.5.1.4 Push the Right arrow once so the second digit (should be **2**) is highlighted.
 - 3.5.1.5 Push the Up arrow to change the digit to **3**.
 - 3.5.1.6 Push **Enter**.
 - 3.5.1.7 Push **Escape**.
 - 3.5.1.8 Push **Escape**.

- 3.5.2 If the KF-200 display **does not** show **Standby** in the upper left go to 3.5.3.
- 3.5.2.1 Push **Titration**.
- 3.5.2.2 Wait for the display to show **Stable** and beeps three times.
- 3.5.3 Push **Start/Stop**.
- 3.5.3.1 If the display shows **BG**. **Wait** after a short time, things are good, go to 3.6.
- 3.5.3.2 Push **Titration**, wait for display to show **Standby** and start again at 3.5.2.
- 3.6 When run finishes check to see if Tartrate values are within QC limits and remove vials.

4. **SAMPLE ANALYSIS**

- 4.1 Sample transfer line if not already done.
- 4.1.1 On top of the reaction vessel remove the glass stopper (wipe off with Kimwipe) and put it into the 50mL beaker near front of the VA-236S Sample Changer.
- 4.1.2 Remove the sampler transfer line from the tube behind the reaction vessel and insert it from where you just removed the glass stopper.
- 4.2 Push **Parameter/Character**.
- 4.2.1 Use appropriate arrow key to get to **04 Blanks**.
- 4.2.2 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.
- 4.2.2.1 Check the water titer value. If incorrect enter the correct value.
- 4.2.3 Push **Escape**.
- 4.3 Push **Sample**.
- 4.3.1 Use arrow keys if necessary to select **01 Groupings**.
- 4.3.1.1 Push **Enter**.
- 4.3.1.2 Make sure **01 GO1 Start:** is **1**, then push **Enter**.

- 4.3.1.3 Make sure **02 GO1 End:** is **3**, then push **Enter**.
- 4.3.1.4 Make sure **03 GO1 File:** is **4**, then push **Enter**.
- 4.3.1.5 Make sure **04 GO2 Start:** is **4**, then push **Enter**.
- 4.3.1.6 For **05 GO2 End:** Use the number pad to enter the number of analyses that you will be doing, (for 8 samples the number would be 27). If doing more, increase the number as appropriate.
- 4.3.1.7 Push **Enter**.
- 4.3.1.8 For **06 GO2 File:** push **3**, then push **Enter** for Samples.
- 4.3.1.9 If **07 GO3 Start:** is blank go to 4.3.1.10.
- 4.3.1.9.1 Push **Clear**, then **Enter**, repeat until entries are blank.
- 4.3.1.10 Push **Escape**.
- 4.3.1.11 *Optional: Printing out the Groupings list.*
- 4.3.1.11.1 *Use arrow keys to select **01 Groupings**.*
- 4.3.1.11.2 *Push **Enter**.*
- 4.3.1.11.3 *Push **Print**.*
- 4.3.2 Use arrow keys to select **02 SampleName, No, W, w**.
- 4.3.2.1 Push **Enter** – this starts sample information entry.
- 4.3.2.2 Can push the **Clear** key to “clear out” an entry and can use the arrow key to move to, then change characters at and after the spot moved to.
- 4.3.2.3 **SampleName** will be **B** for Blanks (Air & MPA), **T** for Tartrate, **C** for Controls, **TS** for Trip Samples, **CH** for Checks, and the sample number for samples. While on **SampleName** you can push the Parameter/Character button to access Alpha-numeric and other characters.
- 4.3.2.4 **No** will be the replicate number, 1-3 for Blanks and 1-2 for all others.
- 4.3.2.5 **W** is the dilution weight for samples and 1 for all others.

- 4.3.2.6 **w** is to be left blank.
- 4.3.2.7 Make sure you push **Enter** after every entry.
- 4.3.2.8 After completing the number of entries from 4.3.1.6 you will get the red line and a longer beep.
- 4.3.2.9 Push **Escape**.
- 4.3.2.10 *Optional: Printing out the Sample list.*
 - 4.3.2.10.1 *Use arrow keys to select **02 SampleName, No, W, w.***
 - 4.3.2.10.2 *Push **Enter**.*
 - 4.3.2.10.3 *Push **Print**.*
- 4.3.3 Push **Escape**.
- 4.4 Prepare and load vials of QC and samples (can start the autorun after loading a few vials).
- 4.5 Check the temperature, if not 130 “reload” the method (4.2).
 - 4.5.1 If “reloading” the method does not work you can manually set the temperature through the VA-236S Sample Changer.
 - 4.5.2 Push **Function**.
 - 4.5.3 It displays **1. TEMP SETTING** so push **Enter**.
 - 4.5.4 Push the Right arrow once so the second digit (should be **2**) is highlighted.
 - 4.5.5 Push the Up arrow to change the digit to **3**.
 - 4.5.6 Push **Enter**.
 - 4.5.7 Push **Escape**.
 - 4.5.8 Push **Escape**.
- 4.6 Start the autorun:
 - 4.6.1 If the KF-200 display **does not** show **Standby** in the upper left go to 4.6.2.

- 4.6.1.1 Push **Titration**.
- 4.6.1.2 Wait for the display to show **Stable** and beeps three times.
- 4.6.2 Push **Start/Stop**.
- 4.6.2.1 If the display shows **BG. Wait** after a short time, things are good, go to 4.7.
- 4.6.2.2 Push **Titration**, wait for display to show **Standby** and start again at 4.6.1.
- 4.7 Check the difference between replicates.
- 4.7.1 If less than 2, go to 4.8.
- 4.7.2 Adding reruns:
 - 4.7.2.1 Push **Sample**.
 - 4.7.2.2 Select **01 Groupings**, push **Enter**.
 - 4.7.2.2.1 Change **05 GO2 End:** to the new value (add 2 for each sample being reran).
 - 4.7.2.2.2 Push **Enter**.
 - 4.7.2.2.3 Push **Escape**.
 - 4.7.2.3 Select **02 SampleName, No, W, w**, push **Enter**.
 - 4.7.2.3.1 Hold down arrow key until you get to where you added the rerun(s).
 - 4.7.2.3.2 Then enter the information as you did in 4.3.2.
 - 4.7.2.3.3 Push **Escape** when done.
 - 4.7.2.4 Push **Escape**.
- 4.8 When run finishes check to see if Check values are within QC limits.

5. **IF INSTRUMENT STOPS DURING AUTORUN**

- 5.1 Do NOT move any vials that have not been analyzed!! By doing the following you do not need to change the sample information.

- 5.2 Push **Sample**.
- 5.2.1 Use arrow keys if necessary to select **01 Groupings**.
- 5.2.1.1 Push **Enter**.
- 5.2.1.2 Change **01 GO1 Start**: to the position number of the sample you want start with (example: if you want to start with Trip then you would enter 6).
- 5.2.1.3 Push **Enter**.
- 5.2.1.4 Change **02 GO1 End**: to the value currently in **05 GO2 End**: (the position number for the final sample).
- 5.2.1.5 Push **Enter**.
- 5.2.1.6 Change **03 GO1 File**: to **3**.
- 5.2.1.7 Push **Enter**.
- 5.2.1.8 If **04 GO2 Start**: is blank go to 5.2.1.9.
- 5.2.1.8.1 Push **Clear**, then **Enter**, repeat until entries are blank.
- 5.2.1.9 Push **Escape**.
- 5.2.1.10 *Optional: Printing out the Groupings list.*
- 5.2.1.10.1 *Use arrow keys to select **01 Groupings**.*
- 5.2.1.10.2 *Push **Enter**.*
- 5.2.1.10.3 *Push **Print**.*
- 5.3 Check the temperature. If not at 130° C “reload” the method.
- 5.3.1 Reload Method:
- 5.3.1.1 Push **Parameter/Character**.
- 5.3.1.2 Use appropriate arrow key to get to **03 Samples**.
- 5.3.1.3 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.

5.3.1.3.1 Check the water titer value. If incorrect enter the correct value.

5.3.1.4 Push **Escape**.

5.3.2 If “reloading” the method does not work, you can manually set the temperature through the VA-236S Sample Changer.

5.3.2.1 Push **Function**.

5.3.2.2 It displays **1. TEMP SETTING** so push **Enter**.

5.3.2.3 Push the Right arrow once so the second digit (should be **2**) is highlighted.

5.3.2.4 Push the Up arrow to change the digit to **3**.

5.3.2.5 Push **Enter**.

5.3.2.6 Push **Escape**.

5.3.2.7 Push **Escape**.

5.4 Start the autorun:

5.4.1 If the KF-200 display **does not** show **Standby** in the upper left go to 5.4.2.

5.4.1.1 Push **Titration**.

5.4.1.2 Wait for the display to show **Stable** and beeps three times.

5.4.2 Push **Start/Stop**.

5.4.2.1 If the display shows **BG**. **Wait** after a short time, things are good.

5.4.2.2 Push **Titration**, wait for display to show **Standby** and start again at 5.4.1.

6. **NEED MORE THAN 33 POSITIONS (“No Blank”)**

6.1 Let the autorun finish the analysis of the samples through position 33.

6.2 Push **Sample**.

6.2.1 Use arrow keys if necessary to select **01 Groupings**.

6.2.1.1 Push **Enter**.

- 6.2.1.2 Change **01 GO1 Start:** to **8** (This way you do not mess up the entries for the Blanks, Control, and Trip).
- 6.2.1.3 Push **Enter**.
- 6.2.1.4 Change **02 GO1 End:** to the position number for the final sample.
- 6.2.1.5 Push **Enter**.
- 6.2.1.6 Change **03 GO1 File:** to **3**.
- 6.2.1.7 Push **Enter**.
- 6.2.1.8 If **04 GO2 Start:** is blank go to 6.2.1.9.
- 6.2.1.8.1 Push **Clear**, then **Enter**, repeat until entries are blank.
- 6.2.1.9 Push **Escape**.
- 6.2.1.10 *Optional: Printing out the Groupings list.*
- 6.2.1.10.1 *Use arrow keys to select **01 Groupings**.*
- 6.2.1.10.2 *Push **Enter**.*
- 6.2.1.10.3 *Push **Print**.*
- 6.2.2 Use arrow keys to select **02 SampleName, No, W, w**.
- 6.2.2.1 Push **Enter** – this starts sample information entry.
- 6.2.2.2 Can push the **Clear** key to “clear out” an entry and can use the arrow key to move to, then change characters at and after the spot moved to.
- 6.2.2.3 **SampleName** will be **B** for Blanks (Air & MPA), **T** for Tartrate, **C** for Controls, **TS** for Trip Samples, **CH** for Checks, and the sample number for samples. While on **SampleName** you can push the Parameter/Character button to access Alpha-numeric and other characters.
- 6.2.2.4 **No** will be the replicate number, 1-3 for Blanks and 1-2 for all others.
- 6.2.2.5 **W** is the dilution weight for samples and 1 for all others.

- 6.2.2.6 **w** is to be left blank.
- 6.2.2.7 Make sure you push **Enter** after every entry.
- 6.2.2.8 After completing the number of entries from 6.2.1.4 you will get the red line and a longer beep.
- 6.2.2.9 Push **Escape**.
- 6.2.2.10 *Optional: Printing out the Sample list.*
 - 6.2.2.10.1 Use arrow keys to select **02 SampleName, No, W, w**.
 - 6.2.2.10.2 Push **Enter**.
 - 6.2.2.10.3 Push **Print**.
- 6.2.3 Push **Escape**.
- 6.3 Check the temperature. If not at 130°C “reload” the method.
 - 6.3.1 Reload Method.
 - 6.3.1.1 Push **Parameter/Character**.
 - 6.3.1.2 Use appropriate arrow key to get to **03 Samples**.
 - 6.3.1.3 Push **Enter** repeatedly until you get to a red line and it gives a longer beep.
 - 6.3.1.3.1 Check the water titer value, if incorrect enter the correct value.
 - 6.3.1.4 Push **Escape**.
 - 6.3.2 If “reloading” the method does not work you can manually set the temperature through the VA-236S Sample Changer.
 - 6.3.2.1 Push **Function**.
 - 6.3.2.2 It displays **1. TEMP SETTING** so push **Enter**.
 - 6.3.2.3 Push the Right arrow once so the second digit (should be **2**) is highlighted.
 - 6.3.2.4 Push the Up arrow to change the digit to **3**.
 - 6.3.2.5 Push **Enter**.

- 6.3.2.6 Push **Escape**.
- 6.3.2.7 Push **Escape**.
- 6.4 Start the autorun.
 - 6.4.1 If the KF-200 display **does not** show **Standby** in the upper left go to 6.4.2.
 - 6.4.1.1 Push **Titration**.
 - 6.4.1.2 Wait for the display to show **Stable** and beeps three times.
 - 6.4.2 Push **Start/Stop**.
 - 6.4.2.1 If the display shows **BG**. **Wait** after a short time, things are good.
 - 6.4.2.2 Push **Titration**, wait for display to show **Standby** and start again at 6.4.1.

7. **SHUT DOWN**

- 7.1 Remove vials.
 - 7.1.1 Pushing the **Home** button on the Autosampler will move it to position 31.
 - 7.1.2 Pushing the arrow keys will move it one position in that direction, be patient if needing to move more than one position over.
- 7.2 Remove transfer line.
 - 7.2.1 Carefully pull out transfer line, wiping it off with Kimwipes, and put it into the tube behind the reaction vessel.
 - 7.2.2 Put the glass stopper from the 50mL beaker into where you just remove the transfer line.
- 7.3 Turn off both instruments.

8. **DATA CAPTURE**

- 8.1 Following *LIMS Karl Fischer Water Analysis* instructions (pages 38-42) - Left click on the **Excel** button near the top of the data capture window on the PC.

- 8.1.1 In the Excel spreadsheet that opens, highlight the data, including the column headers.
- 8.1.2 Copy the highlighted data (Ctrl-C or right click and select Copy).

SOP REVISION HISTORY

DATE	VERSION	NOTES
October 11, 1996	1	Clarification of QC and addition of the Trip sample.
March 10, 1998	1.1	Adjusted document font to Times New Roman 12. Inserted Appendix A formerly a stand-alone document.
December 16, 2002	2.0	Modified SOP to reflect new Karl Fischer instrumentation and renumbered to new section number.
July 8, 2003	2.1	Changed document font to Arial 12. Corrected version enumeration.
April 21, 2004	2.2	Updated Appendix A to include recent modifications to the Karl Fischer operating method.
June 26, 2007	2.3	Updated Appendix A to include recent modifications to the Karl Fischer operating method. Appendix B was added to detail maintenance schedule.
August 19, 2010	2.4	Updated Appendix A to include recent modifications to the Karl Fischer operating method. Appendix B was modified to include changes.
September 19, 2012	3.0	Revised SOP to reflect new Karl Fischer analyzers.