Standard Operating Procedure for Water Determination in Consumer Products Using Karl Fischer (KF) Drying Oven

SAS03
Revision 4.0

Northern Laboratory Branch
Monitoring and Laboratory Division

Approval Signatures	Approval Date
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Standard Operating Procedure for Water Determination in Consumer Products Using Karl Fischer (KF) Drying Oven

1 Introduction

This standard operating procedure (SOP) describes a procedure for the measurement of water in a non-aerosol sample or the non-propellant portion of an aerosol sample, following Method 310 as required by The California Consumer Products Regulations. Development of this SOP was aided by procedures specified in ASTM D4017-96. Water content is determined by using commercially available standard Karl Fischer (KF) reagents integrated with a drying oven.

2 Summary of Method

This method involves heating a sample aliquot or sample dilution in an oven. A stream of dry, inert carrier gas (or dried ambient air) transfers the water from the oven into a titration vessel where it is titrated continuously until reaching the endpoint. KF instrumentation generates results as percent water. These results are subsequently converted and reported as weight fraction of water in the product.

3 Acronyms and Definitions

Acronym or Term	Definition
ACS Grade	Chemicals meeting standards set by the American Chemical Society.
Air Blank	The air blank is a measurement of the ambient air used to establish a zero baseline or background value.
aliquot	A representative portion of a non-aerosol sample or the non-propellant portion of an aerosol sample.
analytical batch	A set of samples analyzed together as a group for a particular analysis.
ASTM	ASTM International, formally known as American Society for Testing and Materials.
Batch Sample (BS)	A laboratory prepared sample of known concentration for QC evaluation under Method 310.
°C	Degrees Celsius
CARB	California Air Resources Board
Control/Check standard	A quality control standard prepared from a source different from the calibration standards. This QC standard is also separately identified as a control standard and a check standard.
duplicate	A second analysis of a sample submitted for analysis under Method 310.

Acronym or Term	Definition
duplicate aliquot	An additional sample aliquot from the same sample carried through all steps of the sampling and analytical procedures of Method 310 in an identical manner.
g	Gram(s)
H&SC	Health and Safety Coordinator
ID	Identification
KF	Karl Fischer
LIMS	Laboratory Information Management System
LIMS Manual	Consumer Products Database Special Analysis Section (Oracle Database and Applications Manual for LIMS)
mL	Milliliter
mm	Millimeter
MPA	1-methoxy-2-propanol
NLB	Northern Laboratory Branch
QC	Quality Control
QCM	Quality Control Manual
replicate	An additional analysis of the same sample aliquot or sample dilution.
RPD	Relative percent difference
sample	The sample submitted for analysis under Method 310.
sample aliquot	The sample aliquot is any aliquot used for analysis, and includes the duplicate aliquot, the Batch Sample, or any archive aliquot undergoing a re-test.
sample batch	A set of samples analyzed together under Method 310.
sample dilution	Dilution made from the sample aliquot (prepared per SAS14).
sd	Standard deviation
solvent blank	A measurement of the reagent used in the sample dilutions, in addition to ambient air used to establish a zero baseline or background value.
SOP	Standard Operating Procedure
tartrate	Disodium Tartrate Dihydrate
VOC	Volatile Organic Compound(s)

4 Interferences

4.1 Interferences in the titrimetric water determinations are associated with condensation or oxidation-reduction reactions with a number of substances and compounds (reference 13.2).

4.2 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 (reference 13.2).

4.3 Other possible interferences to the KF reagent are certain active metals, metal oxides, metal hydroxides, chromates, melamines, etc. (references 13.2 and 13.3).

4.4 Products containing cyanoacrylate should not be analyzed by this method as it will damage equipment.

4.5 Samples that do not dissolve well in solvent (i.e., MPA) may be analyzed from the sample aliquot using the tartrate method. This shall be documented in LIMS and noted on the report to the client.

5 Personnel Qualifications and Training

5.1 Prior to performing this method, new personnel must be trained by staff with detailed knowledge of this method. Personnel must be trained to understand the program's requirements per any applicable state and federal regulations and/or guidance, and this SOP. Personnel will also be trained on how to safely and properly operate the equipment needed to perform the method, the quality assurance components, and LIMS functionality pertaining to the program.

5.2 Personnel should provide an initial demonstration of capability prior to performing this method on real world samples (i.e., data for record).

5.3 Training will be documented and maintained by the laboratory supervisor.

6 Safety Requirements

6.1 All personnel must follow the general health and safety requirements found in NLB's Chemical Hygiene Plans.

6.2 Ensure engineering controls are in place and operating (i.e., adequate ventilation).

6.3 The KF instrumentation heats the vials during sampling; ensure they have cooled before handling.

7 Hazardous Waste

KF chemicals and waste should be kept separate from other consumer product waste streams. KF waste should be placed in the KF satellite waste container. When the satellite container reaches approximately 75%, then it should be moved to the main storage area. The satellite container should not be allowed to exceed 75% before moving to the main storage area. Notify the NLB H&SC when a satellite container is moved to the main storage area so arrangements can be made with the hazardous waste disposal contractor for pick up and disposal.

8 Equipment, Supplies, and Chemicals

8.1 Karl Fischer Titration System configured with volumetric titrator (e.g., Nittoseiko Analytech CA-310), autosampler (e.g., VA-236S autosampler), and laboratory work station

8.2 Analytical Balance, capacity of at least 200 g x 0.00001 g readability (e.g., Mettler XP205) configured with laboratory work station

8.3 1 g Mass, ASTM class 1 or better

8.4 Top-loader Balance, capacity of at least 1000 g x 0.01 g readability

8.5 Software for data transfer and collection (e.g., BalanceTalk, Excel, LabX)

8.6 Laboratory Information Management System (LIMS)

8.7 Laboratory vented enclosure

8.8 Standards Refrigerator(s)

8.9 Desiccator with hygrometer and desiccant

8.10 Volumetric Flasks, Class A, various sizes

8.11 Pipettor, 250 µL, with tips (e.g., Rainin, electronic)

8.12 Syringe, 50 µL (e.g., Hamilton)

8.13 Transfer Tubes, disposable, 3-5 mL capacity

8.14 Vials, approximately 4 mL with cap

8.15 Headspace Vials, 10 mL with 16 mm screw top and PTFE/sil septa (e.g., MicroLiter)

8.16 Vial racks, various sizes

8.17 Task wipes (e.g., Kimwipes)

8.18 Gloves, non-powdered nitrile or suitable alternative

8.19 Solvent squeeze bottles

8.20 Desiccant

8.21 Reagents and Samples

8.21.1 Deionized Water, ASTM Type I

8.21.2 1-methoxy-2-propanol (MPA), 99+%

8.21.3 Acetone, ACS grade or better

8.21.4 Disodium Tartrate Dihydrate (tartrate), water content = 15.61-15.71%

8.21.5 Pyridine-free KF titration reagent for aldehydes and ketones, 1.0 mL = 5 mg water (e.g., EMD AquaStar CombiTirant 5 Keto)

8.21.6 Titration solvent for volumetric KF titration in ketones and aldehydes (e.g., EMD AquaStar CombiSolvent Keto)

8.21.7 Nitrogen, compressed, ultra-high purity

8.21.8 Control/Check Standard dilution prepared using SAS14 from a stock solution of 25% each acetone and water. Stock solutions may be purchased as certified solutions or prepared as follows:

The control/check standard stock solution (0.25 g/mL) is prepared by weighing 25.00 g each of acetone and water into a 100 mL volumetric flask and brought to volume with MPA. Mix by inversion. An alternative volume may be prepared with the same final concentration.

Fill 4 mL vials with no less than 1.5 mL, and no more than 3.0 mL each of the control/check standard stock and cap.

Label each vial with “Acetone/Water Control/Check” and the concentration level, preparation date, expiration date, and the preparer's initials.

The expiration date shall be three years from the date of preparation or the expiration date of the reagents or stock solution from which they are prepared, whichever is sooner.

Store the Control/Check standard aliquots under refrigeration (stored aliquots may be used, it is not necessary to prepare a new stock each analysis).

8.21.9 Sample dilutions prepared using SAS14.

8.21.10 Solvent blank prepared using SAS14.

9 Procedure

9.1 Instrument Preparation

9.1.1 Turn on the KF volumetric titrator and autosampler units.

9.1.2 Verify instrument is operational (refer to APPENDIX A, Section 1).

9.2 Sample Preparation

9.2.1 Use the Consumer Products Analytical Batch Oracle Database application to enter the analytical batch in LIMS. LIMS will randomly assign a replicate for the analytical batch.

9.2.2 Appropriately label headspace vials for the analytical batch.

9.2.3 Conditioning Vials: Prepare three conditioning vials by capping three headspace vials.

9.2.4 Air Blank Vials: Required when running instrument check once per week. Prepare three air blank vials by capping three headspace vials.

9.2.5 Tartrate Instrument Check Vials: Required when performing an instrument check once a week.

9.2.5.1 Perform a balance control on the analytical balance prior to use.

Using forceps, place the 1 g mass on the balance. When the reading becomes stable as indicated by the analytical balance, use the Balance QC Oracle Database application to record the weight in LIMS. The 1 g mass must be within ±2 standard deviations of the target value. If the weight is outside the control limits, there may be a problem with the balance or the mass. Contact appropriate personnel for service.

9.2.5.2 Prepare tartrate by capping a headspace vial, containing 0.200 g of tartrate recorded to the 0.00001 g on the vial.

9.2.5.3 Sample aliquots: Prepare by capping headspace vials containing approximately 0.100 g of the sample aliquot recorded to the 0.00001 g on the vial for samples to analyze by the tartrate method.

Products containing cyanoacrylate should not be analyzed by this method.

Samples that do not dissolve well in solvent may be analyzed from the sample aliquot using the tartrate method.

9.2.5.4 Perform a balance check on the analytical balance at the end of the weighing session.

Using forceps, place the 1 g mass on the balance. When the reading becomes stable as indicated by the analytical balance, use the Balance QC Oracle Database application to record the weight in LIMS. The 1 g mass must be within ±2 standard deviations of the target value. If the weight is outside the control limits, there may be a problem with the balance or the mass. Contact appropriate personnel for service.

Tartrate and samples weighed without successful bracketing by a balance control and check shall not be used for analysis and must be reprepared with successful balance QC.

9.2.6 Solvent Blank Vials: Prepare by pipetting 250 µL of the same solvent used to make the dilutions into three headspace vials, and cap.

9.2.7 Control/Check Standard Vials: Prepare by pipetting 250 µL of the control/check standard dilution into two headspace vials one for the control and one for the check standard, and cap.

9.2.8 Sample dilution vials: Prepare by pipetting 250 µL of the sample dilution into headspace vial and cap. Repeat for each sample dilution and the assigned replicate.

9.3 Water Analysis:

9.3.1 Perform water titer (APPENDIX A, Section 2) to set the calibration factor (B1 (Buret 1) factor), for subsequent analyses. The KF water titer (mg water per mL of titrant) is determined by directly injecting 25 µL water into the titration vessel.

9.3.2 Perform tartrate instrument check (APPENDIX A, Section 3) on a once-a-week basis to verify the proper operation of the KF instrument.

9.3.3 Perform sample analysis to determine water content.

9.3.3.1 The following sequence should be followed with a maximum of ten samples between control and check standards, and ending with a check standard:

Solvent Blanks (three)
Control standard
Sample dilutions
Replicate
Check standard
Repeat sample dilutions, replicates, and check standards as necessary.

9.3.3.2 Follow APPENDIX A, Sections 4 – 6 for operating the KF instrument.

9.3.3.3 The KF instrumentation calculates the amount of water in the consumer product sample by automatically subtracting the average water content of the three solvent blanks.

9.3.4 Use the KF Water Analysis Oracle database application to upload data to LIMS. Refer to APPENDIX A Section 6.

10 Quality Control

10.1 Table of Quality Controls

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Balance Control	Prior to weighing session	±2 sd of the target value	The Balance Control must be within criteria for a valid weigh session. If outside control criteria, re-weigh the 1 g mass standard and record in LIMS. If the weight is still outside the control limits, there may be a problem with the balance or the mass standard. Notify management and contact appropriate personnel for service.
Balance Check	After weighing session	±2 sd of the target value	If outside criteria, the weighing session is invalid. See corrective actions for Balance Control.
Tartrate Instrument Check	Each calendar week prior to sample analysis	Upper and lower control limits set at ±10 percent of the target value.	If outside the control criteria, sample results are invalidated and the tartrate analysis repeated.
Control/ Check Standard	Bracketing sample dilutions, with a maximum of 10 between control/check standard analyses	Warning and control limits are set at ±8 and ±10 percent difference respectively from the target value.	If an analysis is out of the control limits, the affected samples are invalid. Take action to bring the system back into control and reanalyze the control/check standard and any samples not bracketed by successful control/check standards. Three consecutive control

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
			standards falling between the warning and control limits require investigation and corrective action as described in the QCM.
Replicate	One of ten or fewer samples in the analytical batch	For replicate results ≥ 1%: RPD ≤ 25	Re-analyze the analytical batch. If criteria not met after subsequent re-analysis, or if re-analysis not possible, analytical result will be invalidated.
Duplicate	One of ten or fewer samples in the sample batch	No QC criteria for this SOP. Evaluate duplicate results after calculating total VOC per SAS13.	Not applicable. Refer to SAS13 for overall % VOC criteria.

10.2 Equipment Requirements

10.2.1 The balance requires calibration by an outside source annually.

10.2.2 The 1 g mass is calibrated by an outside source annually.

10.2.3 Pipettors require certification by an outside source annually.

11 Sample and Data Management

11.1 Data management consists of samples logged into the LIMS, documentation of unusual occurrences and their resolutions, creation of data packages (monthly, amendments, and special projects) for peer review and management approval, submittal of data to clients, and archival procedures for sample media and respective chains of custody. Program and maintenance notebooks and/or logbooks are to be kept with the instrumentation at all times.

11.2 Sample and data management follow procedures outlined in the QCM. The LIMS Manual describes data management procedures as they pertain to LIMS for this SOP. Additional SOPs that cover sample and data management as they pertain to sample preparation and data reporting under Method 310 include SAS13 and SAS14.

11.3 Information that has been designated as confidential, proprietary, or trade secrets must be maintained in a locked file cabinet in a secure area. Access to this file cabinet is subject to management approval.

12 Calculations

12.1 Percent water as determined by KF instrumentation:

$$\text{Percent water} = \frac{(\text{KF titration reagent (mL)} \times \text{Water Titer (mg/mL)}) - (\text{Blank (mg)})}{(0.25\ \text{mL} \times \text{sample dilution weight (g)})/\ 10\ \text{mL}} \times \frac{1\ \text{g}}{1000\ \text{mg}} \times 100$$

Where:

Blank = average of the three solvent blanks

12.2 Relative percent difference (RPD):

$$\text{RPD} = \frac{(Y-X)}{(Y+X)/2} \times 100$$

Where:

X = sample result

Y = replicate result

13 References

13.1 Method 310 Determination of Volatile Organic Compounds (VOC) in Consumer Products and Reactive Organic Compounds (ROC) in Aerosol Coating Products, August 1, 2022

13.2 The California Consumer Products Regulations, Title 17, California Code of Regulation, Division 3, Chapter 1, Subchapter 8.5, Article 1 – Article 5

13.3 ASTM E203-01, Standard Test Method for Water Using Volumetric Karl Fischer Titration, (October 1, 2001)

13.4 ASTM D4017-96a, Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Method (July 10, 1996)

13.5 US EPA Method 24, Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings, Title 40 Code of Federal Regulations (CFR) Part 60, Appendix A (July 1, 1996)

13.6 Jenkins, V.C., Reilly, Joseph C., Sypowiez, 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)

13.7 Instruction Manual For Karl Fischer Moisture Meter Model CA-310 Volumetric Titration Method (KF Mode), Nittoseiko Analytech

13.8 Instruction Manual for Water Vaporizer Model VA-236S, Mitsubishi Chemical Analytech

13.9 NLB Laboratory Quality Control Manual, Revision 5.0, December 7, 2021

13.10 MLD076 Standard Operating Procedure Preparation of Northern Laboratory Branch's Standard Operating Procedures, December 30, 2021

13.11 Chemical Hygiene Plan for Northern Laboratory Branch, 1927 13th Street, 1900 14th Street, July 19, 2023 or current

13.12 Consumer Products Database Special Analysis Section (Oracle Database and Applications Manual for LIMS)

13.13 SAS14 Revision 0.0, Standard Operating Procedure for Consumer Product Sample Preparation, August 5, 2019

13.14 SAS13 Revision 0.0, Standard Operating Procedure for Consumer Product Sample Batch Management and Reporting, August 5, 2019

14 Revision History

SOP/Addendum Identification	Approval Date	Description of Change
MLD SOP 302 Revision 1	October 11, 1996	Clarification of QC and addition of trip sample.
MLD SOP ES03 Revision 2	March 10, 1998	Adjusted document font to Times New Roman 12. Inserted appendix A formerly a stand-alone document.
Unknown	December 16, 2002	Modified SOP to reflect new Karl Fischer instrumentation and renumbered to new section number.
MLD SOP SAS03 Revision 2.1	July 8, 2003	Changed document font to Arial 12. Corrected version enumeration.

SOP/Addendum Identification	Approval Date	Description of Change
MLD SOP SAS03 Revision 2.2	April 21, 2004	Updated Appendix A to include recent modifications to the Karl Fisher operating method.
Unknown	June 26, 2007	Updated Appendix A to include recent modifications to the Karl Fischer operating method. Appendix B was added to detail maintenance schedule.
MLD SOP SAS03 Revision 2.4	August 19, 2010	Updated Appendix A to include recent modifications to the Karl Fischer operating method. Appendix B was modified to include changes.
MLD SOP SAS03 Revision 3.0	September 19, 2012	Revised SOP to reflect new Karl Fischer analyzers.
SAS03 Revision 4.0	June 14, 2024	Revised SOP to reflect new instrumentation. Reviewed for grammar and content, and compliance with the most recent versions of the Laboratory QCM Revision Number 5.0 and MLD076 Revision 1.0. Replaced the “Trip Sample” with “Batch Sample”. Changes made to QC processes and requirements. Changed SOP title from Standard Operating Procedure for the Karl Fischer (KF) Determination of Water with KF Drying Oven in Consumer Product.

APPENDIX A

Operation Instructions for KF Sample Processor

1 INSTRUMENT START UP

1.1 Log into the laboratory work station.

1.2 Turn on the autosampler

1.3 Take the multicontroller out of sleep mode by pressing and quickly releasing the power button (less than two seconds). The Reagent Supply and Drainage Unit will turn on and stirrer will begin stirring. Make sure the stir bar control is set between 3 and 4. The touch panel control unit of the multicontroller can be used by touchscreen or by mouse.

1.4 On the VA-236S autosampler push the Heater button, the heater light should now be on and the analysis temperature is 130°C.

1.5 Check levels of the liquids.

1.5.1 Adjust the level of liquid in the reaction vessel to approximately 75mL by using the buttons on the automatic solvent dispenser unit to empty and fill the vessel.

1.5.2 Make sure there is enough KF titration reagent.

2 WATER TITER

2.1 On the multicontroller, select the tab for Ch.1 KF to view the 1ch measurement display of the touch panel control unit.

2.2 Select the [≡] key to access the menu display.

2.3 Select Parameter from the menu to navigate to the parameter selection display.

2.4 Select the [Parameters for factors] key at the bottom of the parameter selection display.

2.5 Check the box for “01 Titer”.

2.6 Select the [OK] key.

2.7 The Navigation line will give the instruction “Touch [Titration] key to start dehydration…”. Select the [Titration] key. Wait for the status to be stable, it will beep three times.

2.8 Perform a water titer injection.

2.8.1 Prepare the syringe for the titer by drawing up 25µL of water. This should be done before proceeding to section 2.8.2 as the instrumentation will automatically advance through the titration process once selecting the [Start] key.

2.8.2 The Navigation line will give the instruction “Touch [Start] Key to start measurement.” Select the [Start] key.

2.8.3 When the Navigation line gives the instruction “Inject sample” inject the 25µL of water through the white plug in the front on top of the reaction vessel. Inject the water quickly as the instrument will automatically begin the titration after a brief pause.

2.8.4 The Navigation line will read “Completing start delay. Please wait. Touch [Skip] key to skip it.” Wait for the delay.

2.8.5 The Navigation line will give the instruction “Touch [Sample] key to input sample information. Select the [Sample] key and verify or enter the information as follows:

Sample name	Titer	
Sample + tar weight (W)	0.025	g
Tare weight (w)		g
Sample weight	0.025	g
Acceptance Judge	OFF	

2.8.6 Select the [OK] key.

2.8.7 When the measurement is complete, the instrument will beep three times and indicate “Measurement Ended” in the Navigation line.

2.9 Repeat from Section 2.8 for a total of three injections to determine the KF reagent factor (B1 (Buret 1) factor).

2.10 Confirm the Reagent Information B1 Factor value (in red) is the mean of the three (3) Titer results.

2.10.1 Select the [≡] key, select Measurement results key to display the results in the Detail window. Select the [Deselect All] key, and then select only the three (3) titer results. The Detail window will show the quantity of measurements selected, the average of those measurements, and the RSD.

2.10.2 B1 Factor should be equal to the average (AVG). The average should be approximately 5.

2.10.3 If the AVG value does not match the B1 Factor value listed in the Reagent information, assign the AVG to the B1 Factor.

2.10.4 Select the [Factor assignment] key.

2.10.5 Select “ch.1 KF Factor can be assigned.”

2.10.6 Select the [OK] key

2.10.7 Select the [<] key to exit the measurement results display.

3 INSTRUMENT CHECK (“Tartrate”)

3.1 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. If tartrate does not need to be performed, proceed to Appendix A Section 4.0.

3.2 Prepare samples and QC as described in SAS03 Section 9.2.

3.2.1 Place “conditioning” vials in tray positions P1-3.

3.2.2 Place air blank vials in positions 1-3 and the tartrate vials in positions 4 and 5.

3.3 Select the [≡] key to access the menu on the multicontroller.

3.3.1 Select the Schedule key from the menu to navigate to the schedule list window.

3.3.2 Check the checkbox for “01 Blanks & Tartrate” to use and enter the schedule mode for Tartrate.

3.3.3 Select the Edit key

3.3.4 The schedule table for Blanks & Tartrate will open.

3.3.5 Edit the schedule table for the tartrate method.

3.3.5.1 “No.” corresponds to the vial position.

3.3.5.2 “Status” should be “Ready” for those lines within the schedule that are to be run. Change the status to “Ready” using the drop-down arrow.

3.3.5.3 “Parameter” should be “Blanks” for the Air Blanks and “Tartrate” for the Tartrate and any sample aliquots.

3.3.5.4 “Sample name” should be “Air Blank” for the Air Blanks and “Tartrate” for the Tartrate.

3.3.5.5 “Sample weight” should be empty for the Air Blanks and should be the measured weight for the tartrate and samples. To input weight, select “W”

and type in the weight on the numeric key pad window.

3.3.5.6 “Unit” should be “g” for all.

3.3.5.7 “Note” should have “BL1, BL2, BL3” for each of the three respective blanks, and be empty for all others.

3.3.5.8 Rows can be deleted by [Delete] key.

3.3.5.9 Rows can be inserted above a selected row by touching [Row insert] key.

3.3.6 Select [OK] twice.

3.3.7 The schedule is ready and you can view it in the display.

3.4 Start the autorun:

3.4.1 On the autosampler, check the temperature, if not 130 ºC you can manually set the temperature through the autosampler:

3.4.1.1 Push Function.

3.4.1.2 It displays 1. TEMP SETTING so push Enter.

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

3.4.1.4 Push the Up arrow to change the digit to 3.

3.4.1.5 Push Enter.

3.4.1.6 Push Escape.

3.4.1.7 Push Escape.

3.4.2 On the multicontroller, select the [Start] key to start running the schedule.

3.4.2.1 If the display shows BG Wait after a short time, go to 3.4.3.

3.4.2.2 Push Titration, a pop-up window will state, “Do you want to finish the titration?” Select [Yes] and wait for the autosampler to return to the home position and the display to show Standby.

3.4.2.3 When the display shows Standby, select the [Titration] key again and wait for the display to show Stable.

3.4.2.4 When the display shows Stable, select the [Start] key.

3.4.3 When run finishes a pop-up window will indicate “[Ch.1] Schedule end”.

Select the [OK] key.

3.5 Verify QC criteria in SAS03 section 10.1 are met.

3.6 Remove vials.

3.6.1 Pushing the Home button on the autosampler will move it to position 31.

3.6.2 Pushing the arrow keys will move it one position in that direction, be patient if needing to move more than one position over.

4 SAMPLE ANALYSIS

4.1 Prepare samples and QC as described in SAS03 section 9.2.

4.2 Select the [≡] key to access the menu on the multicontroller.

4.2.1 Select the Schedule key from the menu to navigate to the schedule list window.

4.2.2 Check the checkbox for “02 Blanks & Samples” to use and enter the schedule mode for Samples.

4.2.3 Select the Edit Key.

4.2.4 The schedule table for Blanks & Samples will open.

4.2.5 Edit the schedule table for the analytical batch.

4.2.5.1 “No.” corresponds to the vial positions on the autosampler.

4.2.5.2 “Status” should be “Ready” for those lines within the schedule that are to be run. Lines that are not changed to “Ready” will NOT run on the schedule, and do not need to be deleted. Change the status to “Ready” using the drop-down arrow.

4.2.5.3 “Parameter” should be “Blanks” for the solvent blanks and “Samples” for all others.

4.2.5.4 “Sample name” should be “solvent blank” for the solvent blanks, “Control” for the controls, and the sample ID for all others. Touch the sample name input column and “Input” to input a sample name.

4.2.5.5 “Sample weight” should be empty for blanks, “1.0” for controls/checks, and the dilution weights for all others. To input weight, select “W” and type in the weight on the numeric keypad window.

4.2.5.6 “Unit” should be “g” for all.

4.2.5.7 “Note” should have “BL1, BL2, BL3” for each of the three respective blanks, and be empty for all others.

4.2.5.8 Rows can be deleted by [Delete] key.

4.2.5.9 Rows can be inserted above a selected row by touching [Row insert] key.

4.2.6 Select the [OK] key twice.

4.2.7 The schedule is ready and you can view it in the display.

4.3 Load vials of QC and sample dilutions.

4.4 Start the autorun:

4.4.1 Check the temperature, if not 130 ºC you can manually set the temperature through the autosampler:

4.4.1.1 Push Function.

4.4.1.2 It displays 1. TEMP SETTING so push Enter.

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

4.4.1.4 Push the Up arrow to change the digit to 3.

4.4.1.5 Push Enter.

4.4.1.6 Push Escape.

4.4.1.7 Push Escape.

4.4.2 Select the [Start] key to start running the schedule.

4.4.2.1 If the display shows BG Wait after a short time, go to 4.4.3.

4.4.2.2 Push Titration, a pop-up window will state, “Do you want to finish the titration?” Select [Yes] and wait for the autosampler to return to the home position and the display to show Standby.

4.4.2.3 When the display shows Standby, select [Titration] again and wait for the display to show Stable.

4.4.2.4 When the display shows Stable, select the [Start] key.

4.4.3 When the run finishes a pop-up window will indicate “[Ch.1] Schedule end”. Select [OK].

4.5 Verify QC criteria in SAS03 section 10.1 are met.

4.6 If reanalysis is needed:

4.6.1 Select the [≡] key to access the menu on the multicontroller.

4.6.2 Select the Schedule key from the menu to navigate to the schedule list window.

4.6.2.1 Check the checkbox for “02 Blanks & Samples” to use and enter the schedule mode for samples.

4.6.2.2 Select the [Edit] key.

4.6.2.3 The schedule table for Blanks & Samples will open.

4.6.3 Edit the schedule:

4.6.3.1 Change the “Status” to “Ready” for the samples and QC to be reanalyzed.

4.6.3.2 Prepare and load the vials into the positions that correspond to vial positions in the sequence indicated by “No.”

4.6.3.3 Select the [OK] key twice.

4.6.4 The schedule is ready and can be viewed in the display.

4.6.5 Start the autorun repeating sections 4.3 - 4.4.

4.7 When run finishes a pop-up window will open and state “[Ch. 1] Schedule end”. Select [OK].

4.8 Verify QC criteria in SAS03 section 10.1 are met.

5 SHUT DOWN

5.1 Remove vials.

5.1.1 Pushing the Home button on the autosampler will move it to position 31.

5.1.2 Pushing the arrow keys will move it one position in that direction, be patient if needing to move more than one position over.

5.2 Return the multicontroller to sleep mode by pressing and quickly releasing the power button (less than two seconds).

5.2.1 A pop-up window will state, “Do you wish to set CA-310 to sleep mode?” Select [Yes].

5.2.2 The Reagent Supply and Drainage Unit will also turn off.

5.3 Turn off the autosampler.

5.4 Log out of the laboratory work station.

6 DATA CAPTURE

6.1 Print the first page of each text file for all analyses (i.e., Water titers, Air Blanks, Tartrate, Controls/Checks, and samples).

6.2 Transfer the data from each file to the Excel workbook kf_data22.xlsx (located at https://carb.sharepoint.com/sites/MLD/SAS/Oracle Database/Oracle Upload Worksheets). Save the spreadsheet under a naming system that includes the sample ID numbers.

6.3 Use the KF Water Analysis Oracle database application to upload the data to LIMS.