

APPENDIX XXVIII, Caltest SOP W-TOC/DOC-rev10. Total and Dissolved Organic Carbon (TOC and DOC) EPA Method 415.1/SM5310B/9060 (Revision 10, 04/29/09)

CALTEST STANDARD OPERATING PROCEDURE TOTAL and DISSOLVED ORGANIC CARBON (TOC and DOC) SM5310B/9060
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This SOP outlines the exact procedure to be followed by all staff of Caltest Laboratory who are performing the indicated method. It is the responsibility of any individual performing the procedure to follow these instructions outlined in this document. Any significant modifications to this method require a revision to this SOP. Any deviations from this SOP require prior authorization from the departmental Coordinator/Manager and the QAO. In addition, all deviations from the written procedure require complete documentation in the appropriate logbook.

Amendment Pages	Summary of Changes	Date	Initials Coord., L D, QAO
#1 _____	_____	____/____/____	_____
#2 _____	_____	____/____/____	_____
#3 _____	_____	____/____/____	_____
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A maximum of 5 amendments can be added to each SOP, at which point the entire SOP warrants revision.

TOTAL and DISSOLVED ORGANIC CARBON (TOC and DOC)

1.0 Scope and Application

- 1.1 This method covers the determination of total organic carbon (TOC) and dissolved organic carbon (DOC) in drinking water and other waters which contain carbonaceous matter that is soluble, or matter which has a particle size of 0.2mm or less. The applicable range for the instrument is 0.5 mg/L to 200 mg/L. For higher concentrations, analyze a diluted sample.
- 1.2 A preserved sample contained in a 40 mL VOA vial is placed into an autosampler, Shimadzu TOC CSH. The TOC analyzer is a Shimadzu TOC CSH. The sample is sparged in acid and injected onto a furnace containing a platinum catalyst. The sample is combusted in an oxygen rich environment to form carbon dioxide which is carried to the non-dispersive infra-red, (NDIR), detector. This produces an electrical output (or peak) that is integrated and scaled by the numeric processor and displayed.
- 1.3 Dissolved Organic Carbon

An unpreserved sample is filtered through a 0.45 μm filter at the time of collection and the filtrate acidified with sulfuric or hydrochloric acid to a pH of <2 . If it is not possible to filter the sample in the field, then it must be filtered as soon as possible after arrival at the laboratory. Then follow the order in section 1.2.

2.0 Summary of Method

- 2.1 Inorganic and organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion at 680°C in an oxygen rich environment. The CO_2 formed can be measured directly by an infrared detector. The amount of CO_2 or CH_4 is directly proportional to the concentration of carbonaceous material in the sample.

3.0 Definitions

- 3.1 The carbonaceous analyzer measures all of the carbon in a sample. Because of various properties of carbon-containing compounds in liquid samples, preliminary treatment of the sample prior to analysis dictates the definition of the carbon as it is measured. Forms of carbon that are measured by the method are:
 - A) soluble, nonvolatile organic carbon; for instance, natural sugars.
 - B) soluble, volatile organic carbon; for instance, mercaptans.
 - C) insoluble, partially volatile carbon; for instance, oils.
 - D) insoluble, particulate carbonaceous materials, for instance; cellulose fibers.

E) soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter; for instance, oily matter adsorbed on silt particles.

3.2 The final usefulness of the carbon measurement is in assessing the potential oxygen demanding load of organic material on a receiving stream. This statement applies whether the carbon measurement is made on a sewage plant effluent, industrial waste, or on water taken directly from the stream. In this light, carbonate and bicarbonate carbon are not a part of the oxygen demand in the stream and therefore should be discounted in the final calculation or removed prior to analysis. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon which are measured. Instrument manufacturer's instructions should be followed.

4. Interferences

4.1 Make sure all glassware, reagents, and water are clean and of the highest purity.

4.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter type syringe or pipette. The openings of the syringe or pipette limit the maximum size of particles which may be included in the sample.

4.2.1 A low result will occur if large carbon-containing particles fail to enter the needle.

4.3 Samples with halogenated functionality, ozone, or other species may interfere with the infrared detector.

4.4 Make sure the NDIR detector is free of water by periodically replacing the permeation drying tube.

5. Safety and Precautions

5.1 Prior to performing this analysis, review the MSDS for all standards and reagents to be used. Observe the recommended safety precautions. Protective clothing and safety glasses should be worn when handling samples or reagents.

5.2 Maintain a clean and uncluttered workspace. Return all chemicals, reagents, and resultant wastes to their designated storage area at the completion of the test.

6. Equipment and Supplies

6.1 Shimadzu TOC-Vcsh Analyzer

6.2 Shimadzu 68 place autosampler model ASI-V

6.3 40 mL VOA vials

- 6.4 45 µm filter syringe with filter attachment.
- 6.5 Various volumetric flasks, class A, for dilutions
- 7. **Reagents and Standards**
 - 7.1 Carbon-free DI water
 - 7.2 TOC standards diluted from 1000 mg/L standard of potassium hydrogen phthalate (this solution is usually purchased).
 - 7.3 25% Phosphoric Acid.
 - 7.4 Hydrocarbon free air
 - 7.5 2N Hydrochloric Acid
- 8. **Sample Collection, Preservation, Shipment and Storage**
 - 8.1 Samples should be sampled in duplicate and stored in Teflon sealed 40 mL VOA vials with no headspace. Alternatively, larger sealed glass containers may be used.
 - 8.2 If samples cannot be analyzed within 2 hours from the time of sampling, the sample should be acidified ($\text{pH} \leq 2$) with H_2SO_4 or HCl and stored at 4°C . The preserved sample must be analyzed within 28 days. HCl is the preferred acid for this instrument.
- 9. **Quality Control**
 - 9.1 A blank, LCS, will be run in each batch of 20 samples or less. If analyzing DOC's, a filter blank must also be analyzed.
 - 9.2 The blank consists of carbon free DI water.
 - 9.3 The LCS is prepared from the same solution in which the calibration curve is made.
 - 9.4 QC Samples.
 - 9.4.1 Run one blank per batch.
 - 9.4.2 Run CCV every 10 samples.
 - 9.4.3 Run one spike and spike duplicate sample for every 10 samples.
 - 9.5 A 10 mg/L spike solution and sample value is prepared in the following manner: 1 mL of a 1000 mg/L TOC standard is diluted to 100 mL with sample or diluted sample in a volumetric flask. A CCV is run every ten samples and an ICV every 20 samples.

10. System Startup

- 10.1 Turn on hydrocarbon free air tank, set to 60 psi.
- 10.2 Turn on the analyzer and autosampler. Allow the system to startup for 2 minutes.
- 10.3 Turn on the computer. Click on TOC software, connect to instrument.
- 10.4 Wait for the system to reach 680°C. Open the "background monitor" to check the system status.
- 10.5 Wait for the baseline to stabilize, at least 1 hour. The "background monitor" should show all green checks. If any of the indicators are red, address the problem, and wait for the system to stabilize.

11. Procedure

- 11.1 Remove samples from the refrigerator. Choose a sample with extra volume to perform QC. Run an MS/MSD for every 10 samples.
- 11.2 Creating a curve:
 - 11.2.1 Open the "Sample Table", select 'Calibration Curve'.
 - 11.2.2 Make the following selections:
 - Page 1: <enter>
 - Page 2: 'Edit calibration curves manually
 - Page 3: Select NPOC, clear default lines, select linear regression, assign a file name. Leave checked boxes unchecked.
 - Note: you need to create two curves monthly, (a low and a high range), or when conditions change. Use the following format DDMMYYcurve10 and DDMMYYcurve200 as names.
 - Page 4: Units – mg/L, injections 2of3, washes 2, SD and CV max 5%
 - Page 5: Create curve, curve 10 – 0.0, 0.5, 1.0, 2.0, 5.0, and 10.0. Curve 200 – 10.01, 20.0, 50.0, 100.0, and 200.0. **SELECT 100uL** as an injection volume.
 - Page 6: Select 'Correlation Coefficient Check' – 1st time – Repeat, 2nd time – Stop, Lower limit – 0.995.
 - Page 7: <enter>
 - Page 8: Do not enable history log, just <enter>.
- 11.3 Creating a Method:
 - 11.3.1 Page 1: <enter>
 - 11.3.2 Page 2: Select NPOC, clear default lines, assign a file name. Use format DDMMYYmethod

- 11.3.3 Page 3: Select Curve 10 in box 1 created above, Curve 200 in box 2 created above.
- 11.3.4 Page 4: <enter>
- 11.3.5 Page 5: <enter>
- 11.3.6 Page 6: Leave box unchecked, <enter>.
- 11.3.7 Page 7: <enter>
- 11.4 Creating a Schedule:
 - 11.4.1 Click 'Sample Run'
 - 11.4.2 Save schedule: use format DDMMYY, a blank table will appear.
 - 11.4.3 Select Autogenerate under the Insert toolbar.
 - 11.4.4 Select method you just created
 - 11.4.5 Select 2 vials starting on vial 1, <enter> -these are the initial blanks
 - 11.4.6 For vial 3, select calibration curve, (curve10), vials 1 starting with vial 3.
 - 11.4.7 For vial 4, select calibration curve, (curve 200), vials 1 starting with vial 4.
 - 11.4.8 For vial 5 on select method and vials needed for the batch starting with vial 5.
Make sure to account for all QC when planning the number of vials.
 - 11.4.9 For the rest of the pages, just <enter>. At the end another window will come up showing the autosampler tray. All vials should be blue. If not, fix the position number at this time in the vial field.
 - 11.4.10 Omit the calibration curve for subsequent runs until another curve is required.
 - 11.4.11 Once the table is created, save it, and now you can edit the sample information in the table. Save and print the table after editing.
 - 11.4.12 Remove the autosampler tray, verify that the samples are preserved, and load the tray according to the schedule.
 - 11.4.13 Replace the tray and check the 'Background monitor' to verify that the system is ready to run. Check that all liquid levels are sufficient for the run, the gas is at least 500 psi, and check the waste drum.
- 11.5 For Method 9060
 - 11.5.1 Homogenize the sample in a blender.
 - 11.5.2 Lower the pH of the sample to 2.
 - 11.5.3 Run quadruplicate analysis.
 - 11.5.4 Report both average and the range.
- 11.6 Starting The Run
 - 11.6.1 Click the lightening bolt on the top toolbar to connect to the system.
 - 11.6.2 The stoplight should be activated – click this to start the run.
 - 11.6.3 The standby window will come up. Select 'shutdown instrument'
 - 11.6.4 The run will start.

12. Calculations

- 12.1 Area counts are plotted against concentration to produce a linear curve. Sample, standards area counts are then compared to the curve to produce concentrations. The curve coefficient must be 0.995 or greater.
 - 12.2 If dilutions are needed, then the analyst will need to apply the correct dilution factor to the concentration read-out. Any results above 200 mg/L need dilution and rerun. Any dilution factors need to be manually calculated.
 - 12.3 When the run is finished, click on the run title and then right click print tables and print sample report. Combine data in a folder and submit for approval.
- 13. Pollution Prevention**
- 13.1 The samples analyzed in this method pose little threat to the environment when recycled and managed properly.
 - 13.2 Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.
- 14.0 Data Assessment and Acceptance Criteria for QC Measures**
- 14.1 See Internal QC Summary
- 15.0 Corrective Actions for Out-of-Control Data**
- 15.1 See Internal Quality Control Summary
- 16.0 Contingencies for Handling Out-of-Control or Unacceptable Data**
- 16.1 See SOP # Q-CORRECT
- 17.0 Waste Management**
- 17.1 All waste is segregated as to type, and put into the appropriate waste container.
 - 17.2 All waste is tested prior to disposal, and the results are reviewed by the waste disposal coordinator. If the results are found to be greater than is allowed for general disposal, the waste is removed by a licensed hauler. This metals waste is disposed of as corrosive liquid waste by an approved hazardous waste vendor. If the results of testing are found to be less than allowable limits for general disposal, the waste is poured into the sewer after neutralization.
 - 17.3 For further information see the Caltest Waste Management Coordinator.

18. Method Performance

18.1 See EPA Methods and Guidance for Analysis of Water, June 1999.
Method 415.1, Section 9.

18.2 Standard Methods for the Examination of Water and Wastewater. 18th Edition Section 6.

19. References

19.1 Methods for Chemical Analysis of Water and Wastes, EPA 415.1, 1983.

19.2 O/I Analytical instrument manuals.

19.3 EPA Methods and Guidance for Analysis of Water, June 1999.
Method 415.1.

19.4 Standard Methods for the Examination of Water and Wastewater. 18th Edition

19.5 SW-846 Method 9060 Revision 0, September 1996

APPENDIX XXIX, Caltest SOP W-TURB-rev7. Turbidity EPA 180.1/SM 2130B (Revision 7, 03/18/09)

CALTEST STANDARD OPERATING PROCEDURE

TURBIDITY

EPA 180.1 / SM 2130B

Reviewed by	Nancy Pacheco	Title:Wet Chemisrty Analyst	Date:	3-18-09
Reviewed by:	Michael Soon	Title:Wet Chemisrty Coordinator	Date:	3-18-09
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Approved by:	Christine Horn	Title: Laboratory Director	Date:	3-18-09

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#5	_____	_____	___/___/___	_____

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TURBIDITY
EPA 180.1 / SM2130B

1. Scope and Application:

- 1.1 This method is applicable to drinking, surface, and saline waters in the range of turbidity from 0 to 40 nephelometric turbidity units (NTU). Turbidity in water is caused by suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, plankton and other microscopic organisms. Higher values may be obtained with dilution to the sample.

2. Method Summary

- 2.1 The Nephelometric method for turbidity is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity.

3. Definitions

- 3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.
- 3.2 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
- 3.3 **Method Blank (MB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 3.4 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.

- 3.5 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
 - 3.6 **Primary Calibration Standard (PCAL)** -- A suspension prepared from the primary dilution stock standard suspension. The PCAL suspensions are used to calibrate the instrument response with respect to analyte concentration.
 - 3.7 **Laboratory Control Sample (LCS)** -- A solution of the method analyte of known concentrations that is used to fortify an aliquot of LRB matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance.
 - 3.8 **Secondary Calibration Standards (SCAL)** -- Commercially prepared, stabilized sealed liquid or gel turbidity standards calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymers.
 - 3.9 **Stock Standard Suspension (SSS)** -- A concentrated suspension containing the analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension is used to prepare calibration suspensions and other needed suspensions.
4. **Interferences**
- 4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles will affect results in a positive manner.
 - 4.2 The presence of true color, that is the color of water that is due to dissolved substances which absorb light, will cause turbidities to be low.
5. **Safety and Precautions:**
- 5.1 Prior to performing this analysis, review the MSDS for all standards and reagents to be used. Observe the recommended safety precautions. Protective clothing and safety glasses should be worn when handling samples or reagents.
 - 5.2 Maintain a clean and uncluttered work space. Return all chemicals, reagents and resultant wastes to their designated storage area at the completion of the test.
6. **Apparatus**
- 6.1 Turbidimeter, VWR Scientific
 - 6.2 Sample tube
 - 6.3 Graduated cylinders (needed for dilutions).

7. Reagents:

7.1 Turbidity free water (D.I.)

7.2 Stock Formazin turbidity suspensions: (prepare monthly)

7.2.1 Solution 1:

Dissolve 1.00g hydrazine sulfate $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ in D.I. water and dilute to 100mLs.

7.2.2 Solution 2:

Dissolve 10.00g hexamethylenetetramine in D.I. and dilute to 100mLs.

7.2.3 In a 100 mL volumetric flask, mix 5.0 mL of solution 1 with 5.0 mL of solution 2. Allow to stand 24 hours at $25 \pm 0.5^\circ\text{C}$, then dilute to 100 mL (400NTU)

7.3 Standard Formazin Suspension: (prepare weekly)

7.3.1 Dilute 2.5, 1.0, 0.1mls stock suspension to 100 mLs. The turbidity of this suspension is defined as 10, 4.0, 0.4 NTU.

7.4 Purchased Standards are also available-we mainly use 0.4, 4.0, 10 NTU Standards. Other concentrations are used depending on availability.

8. Sample Handling and Preservation

8.1 Preservation of sample is not practical; analysis should begin as soon as possible. Refrigeration of the sample at 4°C minimizes bacterial decomposition. Samples must be analyzed within 48 hours.

9. Quality Control

9.1 Run a set of duplicates every batch, not to exceed 20 samples. No spike is required.

9.2 Run one LCS per batch of 20 samples or less. LCS recovery should be 90-110%. If LCS recovery is outside control limits, check reagents and calculations.

10. Calibration and Standardization

10.1 Allow all samples and standards to come to room temperature.

10.2 Adjust meter to zero with DI water using the instrument offset adjust screw, on the side panel. Calibrate the 20 scale with 10NTU, adjust with a screw driver on the side panel.

10.3 For 20 scale, measure 4.0 NTU. A secondary source of 0.4 NTU is also measured. The recovery should be 90-110% and every 10 samples.

10.4 Fill sample to the line when measuring samples and standards.

11. Procedure

11.1 Following the calibration, read 4.0 and 0.4 NTU on 20 scale.

11.2 Run the LCS prior to analysis (Standard Formazin suspension). Use one concentration for each range available on the instrument.

11.3 Run the sample:

11.3.1 For samples with turbidity less than the dynamic range of the instrument, thoroughly invert the sample. Wait until air bubbles disappear, then pour the sample into the sample tube. Wipe the tube and read turbidity on appropriate pre-calibrated scale.

11.3.2 For samples with turbidity greater than the dynamic range of the instrument, invert the sample thoroughly. Pipet a Known volume into a graduated cylinder and dilute to volume with turbidity-free water. Mix well and pour into the sample tube. Wipe the tube and read the turbidity. Record the turbidity and dilution in the logbook.

12. Calculations

12.1 Not applicable

13. Pollution Prevention

13.1 The samples analyzed in this method pose little threat to the environment when recycled and managed properly.

13.2 Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

14. Data Assessment and Acceptance Criteria for QC Measures

14.1 See Internal QC Summary

15. Corrective Actions for Out-of-Control Data

15.1 See Internal Quality Control Summary

16. Contingencies for Handling Out-of-Control or Unacceptable Data

16.1 See SOP # Q-CORRECT

17. Waste Management

17.1 The waste from this analysis can be poured into general lab waste containers.

17.2 All waste is segregated according to type, and tested before disposal. The results are reviewed by the waste disposal coordinator. If they are greater than is allowed for general disposal, the waste is removed by a licensed hauler. If the results of testing are less than allowable limits for general disposal, the waste is poured into the sewer after neutralization.

17.3 For further information, see the Caltest Waste Management Coordinator.

18. Method Performance

18.1 Not available at this time.

19. References:

19.1 Methods for Chemical Analysis of Water and Wastewater, EPA 180.1, EPS 600/4-79-020, March 1983

19.2 Standard Methods for Examination of Water and Wastewater, 18th Edition, 1992

APPENDIX XXX. Caltest SOP B-TSS-rev7. Caltest SOP Residue, Non-Filterable EPA 160.2/SM2540D (Gravimetric, Dried at 103-105°C) (Revision 7, 05/05/09)

CALTEST STANDARD OPERATING PROCEDURE

RESIDUE, NON-FILTERABLE
SM 2540D (Gravimetric, Dried at 103-105°C)

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Reviewed by: Michael Soon	Title: WetChem/Bio Coordinator	Date: 5-5-09
Reviewed by: Carmelita Oliveros	Title: Quality Assurance Officer	Date: 5-5-09
Approved by: Christine Horn	Title: Laboratory Director	Date: 5-5-09

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Residue, Non-Filterable
SM 2540D (Gravimetric, Dried at 103-105°C)

1. Scope and Application

- 1.1 This method is applicable to industrial and domestic wastes, drinking, surface and saline waters.
- 1.2 The practical range of the determination is 3 mg/L to 20,000 mg/L.
- 1.3 A well mixed sample is filtered through a weighed glass fiber filter and the residue retained on the filter is dried to a constant weight at 103-105°C.
- 1.4 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to a constant weight at 103-105°C.

2. Summary

- 2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C.
- 2.2 The filtrate from this method may be used for Residue, Filterable.

3. Definitions

- 3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103-105°C.

4. Interferences

- 4.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
- 4.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes the potential interference

5. Safety

5.1 Prior to performing this analysis, review the MSDS for all standards and reagents to be used. Observe the recommended safety precautions. Protective clothing and safety glasses should be worn when handling samples or reagents.

5.2 Maintain a clean and uncluttered workspace. Return all chemicals, reagents, and resultant wastes to their designated storage area at the completion of the test.

6. Equipment and Supplies

6.1 Glass Microfiber Filters Whatman 934-AH 90 mm and 125mm (required for Anheuser-Busch only); or equivalent.

6.2 Buchner funnels, 70 mm, 100mm.

6.3 Filter Flasks, 1000 mL.

6.4 Drying oven, 103-105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

6.7 Aluminum pans, 70 mm., 100 mm

6.8 Wide bore pipets.

6.9 Graduated cylinders.

6.10 Forceps.

7. Reagents and Standards

Celite, Johns-Manville

8. Sample Handling and Preservation

8.1 Non-representative particulates such as leaves, sticks, insects, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is

not desired in the final result. Any alteration of sample due to nonrepresentative particulates should be appropriately documented in the Analytical Logbook.

- 8.2 Analysis should begin as soon as possible. Refrigeration or icing to just above freezing to 6°C, to minimize microbiological decomposition of solids, is required. Hold time is 7 days. Sample cannot be chemically preserved (i.e. with H₂SO₄ or HNO₃).

9. Quality Control

- 9.1 A blank must be done with each batch of 20 samples analyzed. Follow the same procedure in Section 11.3.

- 9.2 A standard must be done with each batch of 20 samples analyzed. Follow the same procedure as in Section 11.3. Use a 50mL volume of the 500 mg/L Celite Standard.

9.2.1 Preparation of Laboratory Control Standard 500mg/L:

Dry Celite for at least two hours at 120C. Place dried Celite in Desiccator and let cool for 15 to 20 minutes. Weigh 0.5g of dried Celite and transfer to 1000mL Volumetric flask. Bring the flask up to volume with 20°C DI water. Transfer to an amber liter for use and store at 4°C. The standard is good for one month.

- 9.3 A set of duplicates must be done daily or with each batch of 20 samples analyzed, whichever is more frequent. To analyze duplicates, choose a sample (Anheuser-Busch, if available) and follow the procedure in Section 11.3.

10. Calibration and Standardization

Not applicable

11. Procedure

- 11.1 Get samples out of the refrigerator. Set up analytical batch on the computer. Preparations of glass fiber filter:

Insert glass fiber filter into bottom of Buchner funnel with wrinkled surface up. While vacuum is applied, wash the filter with three successive 20mL volumes of reagent-grade water, allowing each washing to filter completely. Remove all traces of water from filter by continuing to apply vacuum after water has passed

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through filter (3-4 minutes). Remove filter from Buchner funnel and place in marked aluminum pan. Handle filter and pan with forceps only from this point on. Dry in an oven at 103-105C for one hour. Place dried filters and pans in a desiccator for at least 15 minutes to balance temperature. Weigh filter and pan, and record weight. Repeat cycle of drying, desiccating, and weighing until a constant weight is obtained (or until weight change is less than 0.5mg) from previous weighing. Store in a desiccator until needed.

11.2 Selection of sample volume:

Choose sample volume to yield between 5 and 200mg dried residue (i.e. AB 50mL, most clear effluent or pond water 5-200mL, most influents and cloudy industrial effluents 50 mL, and sludges 5-25mL). If more than 5 minutes are required to complete filtration, decrease sample volume by approximately half.

11.3 Sample analysis:

Remove filter and pan from desiccator and insert filter into bottom of Buchner funnel. Seat filter in funnel with a small amount of reagent grade water. Mix sample thoroughly by inverting sample container vigorously 20 times. Immediately transfer predetermined sample volume selected in Section 11.2 to the filter using a graduated cylinder or wide bore pipet. After completing filtration, wash the graduated cylinder or wide bore pipet and filter three successive 20mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for 3-4 minutes. Carefully remove filter from Buchner funnel (using forceps), and place in aluminum pan. Dry for one hour at 103-105°C in oven. Cool in desiccator for at least 15 minutes to balance temperature, and weigh. Repeat cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5 mg, whichever is less. Some samples will continue to lose weight. After five weighings, accept weight and footnote report.

11.4 Record date, analyst initials, time in & time out, lab number, client, pan I.D., all weight values, and any observations in the TSS logbook.

12. Calculations

12.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/L} = \frac{(A-B) \times 1000\text{mg/g} \times 1000 \text{ mL/L}}{\text{Sample Volume, mL}}$$

where:

A = weight of filter + dried sample residue + weigh dish, g

B = weight of filter + weigh dish, g

Detection limit: 3mg/L

13. Pollution Prevention

13.1 There are no particular pollution prevention steps taken by the laboratory for this test. The pans that are weighed gravimetrically are re-used.

14. Data assessment and acceptance criteria for quality control measures

14.1 The criteria is referenced in Caltest Internal QC Summary Manual.

15. Corrective actions for out of control data

15.1 The criteria is referenced in Caltest Internal QC Summary Manual

16. Contingencies for handling out of control or unacceptable data

16.1 The contingencies for handling out of control or unacceptable data is addressed in the Caltest SOP: Q-CORRECT.

17. Waste Management

17.1 The filtrate is stored in non-hazardous waste jugs and is not disposed off until it has been analyzed and cleared by the waste coordinator. The pans are cleaned and re-used, and the filters are discarded to municipal disposal.

18. References

18.1 Methods for Chemical Analysis of Water and Wastewater, EPA 600/4-79-020, March 1983.

APPENDIX XXXI. Caltest SOP W-RESIDUE-rev7. TOTAL RESIDUE, SM 2540B / EPA METHOD 160.3, TOTAL VOLATILE RESIDUE, SM 2540 G / EPA METHOD 160.4 (Revision 7, 03/18/09)

CALTEST STANDARD OPERATING PROCEDURE

TOTAL RESIDUE, SM 2540B

TOTAL VOLATILE RESIDUE, SM 2540 G / EPA METHOD 160.4

Reviewed by:	Nancy Pacheco	Title: Wet Chemisrty Anlyst	Date: 3-18-09
Reviewed by:	Michael Soon	Title: Wet Chemisrty Coordinator	Date: 3-18-09
Reviewed by:	Carmelita Oliveros	Title: Quality Assurance Officer	Date: 3-18-09
Approved by:	Christine Horn	Title: Laboratory Director	Date: 3-18-09

This SOP outlines the exact procedure to be followed by all staff of Caltest Laboratory who are performing the indicated method. It is the responsibility of any individual performing the procedure to follow these instructions outlined in this document. Any significant modifications to this method require a revision to this SOP. Any deviations from this SOP require prior authorization from the department Coordinator/Manager and the QAO. In addition, all deviations from the written procedure require complete documentation in the appropriate logbook.

Amendment	Pages	Summary of Changes	Date	Initials Coordinator, LD, QAO
#1	_____	_____	____/____/____	_____
#2	_____	_____	____/____/____	_____
#3	_____	_____	____/____/____	_____
#4	_____	_____	____/____/____	_____
#5	_____	_____	____/____/____	_____

A maximum of 5 amendments can be added to each SOP, at which point the entire SOP warrants revision.

TOTAL RESIDUE, SM 2540 B (Gravimetric)

TOTAL VOLATILE RESIDUE, SM2540 B / EPA METHOD 160.4

1.0 Scope and Application

- 1.1 This method is applicable to drinking surface waters, saline waters, domestic wastes, industrial wastes, solids and semisolid samples as river and lake sediments or other sludge cakes from vacuum filtration, centrifugation or other sludge dewatering processes.
- 1.2 The practical range of the determination is from 10 mg/L to 20,000 mg/L for aqueous waste and percentage levels for semisolid and solid type matrices.

2.0 Method Summary

- 2.1 A well mixed aliquot of the sample is quantitatively transferred to a pre-weighed evaporating dish and evaporated to dryness at 103-105°C.

3.0 Definitions

- 3.1 Gravimetric Analysis – a type of quantitative analysis involving precipitation of a compound that can be weighed and analyzed after drying.
- 3.2 Residue – the solid matter left after all liquid has been removed from a solution or sludge sample.
- 3.3 Volatility – the tendency of a solid or liquid material to pass into the vapor state at a given temperature.

4.0 Interferences

Caltest Analytical Laboratory

- 4.1 Non-representative particulars such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
- 4.2 Floating oil and grease, if present, should be included and dispersed by a blender device before aliquoting.

5.0 Safety and Precautions

- 5.1 Prior to performing this analysis, review the MSDS for all standards and reagents to be used. Observe the recommended safety precautions. Protective clothing and safety glasses should be worn when handling samples or reagents.
- 5.2 Maintain a clean and uncluttered workspace. Return all chemicals, reagents, and resultant wastes to their designated storage area at the completion of the test.
- 5.3 This method is used to analyze potentially hazardous samples. Care should be taken when handling all samples, standards and reagents.

6.0 Equipment and Supplies

- 6.1 Evaporating dishes or aluminum pans.
- 6.2 Drying oven capable of 103-105 °C
- 6.3 Calibrated analytical balance.
- 6.4 Desiccator with dry desiccant. Desiccant chips must be blue. If chips are pink replace with new desiccant.
- 6.5 Muffle furnace (550±50°C).

7.0 Reagents and Standards

- 7.1 NaCl – granular
- 7.2 Deionized reagent water

8.0 Sample Handling and Preservation

- 8.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigerate or ice from just above freezing to 6°C to minimize microbiological decomposition of solids and analyze within 7 days of sampling.

9.0 QC Requirements – Quality Control

For each sample batch of 20 or less, the following QC samples must be prepared and analyzed:

- 9.1 One method blank - an aliquot of uncontaminated matrix (using DI water) used to demonstrate that there is no source of contamination and that constant weight has been achieved.
- 9.2 One sample duplicate - A replicate aliquot of a sample used to demonstrate the precision of a method. The RPD should be $\leq 20\%$.

10.0 Calibration and Standardization

- 10.1 None other than the QC requirements.

11.0 Procedure

- 11.1 Get samples out of the refrigerator. Set up analytical batch on the computer (see Appendix 1).
- 11.2 For aqueous sample, heat the clean beaker or disposable aluminum dishes (*dish is mainly used, but if sample is low, use beaker*) to 103-105°C for one hour. If volatile residue is to be measured, heat vycor dishes or heat resistant equivalent dishes at 550 +/- 50° for one hour in a muffle furnace. Cool, desiccate, weigh and store in dessicator until ready for use.
- 11.3 Measure 100 mL sample (*for samples with low % solids*) or an aliquot of sample sufficient to contain a residue of at least 2.5mg for aqueous and 10 grams for solids.

- 11.4 Transfer a measured aliquot of sample to the pre-weighed dish and evaporate to dryness in a drying oven at least 98°C but no less than 80 °C for 12-18 hours to prevent boiling and splattering of sample.
- 11.5 Dry the evaporated sample for at least 1 hour at 103-105°C. Cool in a desiccator and weigh. Record results.
- 11.6 Reheat the sample for an additional hour at 103-105°C. Cool in a desiccator, weigh and record results. Weigh dishes and record the weight in the log book. Repeat cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight loss is less than 4% of previous weight or 0.5 mg, whichever is less. If this criteria cannot be met by two successive measurements, additional drying must be performed. Some samples will continually lose weights. After five weighings, accept weight and footnote report.
- 11.7 If volatile solids analysis is requested, place sample in a muffle furnace for one hour at 250°C and one hour at 550°C. Cool in desiccator and weigh.

12.0 Calculation

- 12.1 Calculate total residue as follows:

$$\text{Total Residue mg/L or mg/kg} = \frac{(A - B) * 1000}{C}$$

Where A = mass of dried sample and dish in mg
 B = mass of empty dish in mg
 C = volume of sample in mL or mass in grams

$$\text{Total Residue(\%)} = \frac{(A - B) * 100}{(C - B)}$$

Where A = mass of dried sample and dish in grams, B = mass of empty dish in grams, and C = mass of sample in grams

- 12.2 Calculate volatile residue as follows:

- 12.2.1 Aqueous Samples:

$$\text{Volatile Residue (mg/L)} = \frac{(A - B) * 1000}{C}$$

Where A = mass of sample and dish dried at 550 °C
 B = mass of sample and dish ignited at 550 °C
 C = volume of sample initially used in mL

- 12.2.2 Solid or Semisolid Samples: (This result is expressed in terms of % relative to the total residue as determined in 12.1)

$$\text{Volatile Residue (\%)} = \frac{(A - B) * 100}{A}$$

Where A = mass of sample and dish dried at 550 °C*
B = mass of sample and dish ignited at 550 °C*

* the units for the mass measurements must be the same for both A and B

13.0 Pollution Prevention

- 13.1 The samples analyzed in this method pose little threat to the environment when recycled and managed properly.
- 13.2 Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

14.0 Data Assessment and Acceptance Criteria for Quality Control Measures

- 14.1 See Internal Quality Control Summary

15.0 Corrective Actions for Out-of-Control Data

- 15.1 See Internal Quality Control Summary

16.0 Contingencies for Handling Out-of-Control or Unacceptable Data

- 16.1 See SOP # Q-CORRECT

17.0 Waste Management

- 17.1 The waste from this analysis can be poured into general lab waste containers.

- 17.2 All waste is segregated according to type, and tested before disposal. The results are reviewed by the waste disposal coordinator. If they are greater than is allowed for general disposal, the waste is removed by a licensed hauler. If the results of testing are less than allowable limits for general disposal, the waste is poured into the sewer after neutralization.

17.3 For further information see the Caltest Waste Management Coordinator.

18.0 Method Performance

- 18.1 None available at this time.

19.0 References

- 19.1 Standard Methods for Examination of Water and wastewater, 18th Edition, 1992.
- 19.2 Test Methods for Chemical Analysis of Water and Wastewater, EPA 600/4-79-020, March 1983.
- 19.3 EPA Methods and Guidance for Analysis of Water, June 1999.

APPENDIX 1

GRAVIMETRIC LOGBOOK PROCEDURE

1. Create a batch in the Horizon system.
 - 1.1 Batch may be created by choosing a Blank and an LCS. Note the Batch and Queue.
2. Open Microsoft Excel.
3. Open a new file using the top toolbar under the file tab.
4. Choose appropriate logbook under templates
 - 4.1 Moisture for % M, % S (Results in %)
 - 4.2 Solids for TSS, TDS, TS (Results in mg/L)
 - 4.3 OG for Oil and Grease.
 - 4.4 Vol Solids for VSS and VS
5. Enter LOGIN data
 - 5.1 Initials
 - 5.2 Password
 - 5.3 Queue
 - 5.4 Batch
6. Fill out sample ID#, Pan/Dish ID #, Tare wt.(printed from balance), sample volum if applicable.
7. Perform analysis, print weights from balance.
8. Once the entire sheet is finished, go back to Horizon batch and enter samples in the order they appear on the excel logbook. The Blank, LCS, LCSD, and Duplicate (R1, R2) will be assigned Horizon QC numbers.
9. Enter the numbers into the EXCEL Logbook.
10. Refresh Batch. This will prompt you for your password again. Every thing should turn blue. If not, check that you have all of the sample ID's are correct. Fix and refresh again.
11. Once the batch is complete, post batch.

APPENDIX XXXII. Caltest SOP O-Pyrethroidsncirev1. Caltest SOP Pyrethroids By GCMS Selective Ion Monitoring (Revision 1, 07/2009)

CALTEST STANDARD OPERATING PROCEDURE

PYRETHROIDS BY NCI-GCMS- SIM/MICRO ECD

Reviewed by: Rick Heines	Title: Organics Coordinator	Date: 7/2009
Reviewed by: Sonya Babcock	Title: Quality Assurance Officer	Date: 7/2009
Approved by: Patrick Ingram	Title: Laboratory Director	Date: 7/2009

This SOP outlines the exact procedure to be followed by all staff of Caltest Laboratory who are performing the indicated method. It is the responsibility of any individual performing the procedure to follow these instructions outlined in this document. Any significant modifications to this method require an amendment to this SOP with the approval of the department Coordinator, Laboratory Director (LD) and QAO. All amendments must be identified below, and attached to this document to be considered valid changes. Any deviations from this SOP require prior authorization from the department Coordinator, Lab. Director and QAO. In addition, all deviations from the written procedure require complete documentation in the appropriate logbook.

Amendment	Pages	Summary of Change	Date	Initials
#1	_____	_____	_____	____/____/____
#2	_____	_____	_____	____/____/____
#3	_____	_____	_____	____/____/____
#4	_____	_____	_____	____/____/____
#5	_____	_____	_____	____/____/____

A maximum of 5 amendments can be added to each SOP, at which point the entire SOP warrants revision.

PYRETHROIDS BY NCI-GCMS- SIM/MICRO ECD

1. *Scope and Application*

- 1.1 This document details the procedures necessary for the analysis of extracts by NCI-GCMS SIM. This method is appropriate for extracts of all matrices prepared by EPA Method 3510A, EPA Method 3520C, EPA Method 3535, EPA Method 3540C, EPA Method 3541, EPA Method 3550, EPA Method 3580A.
- 1.2 All soils and sludges are either air dried or freeze dried before extraction. Any residual water in the extracts adversely affects the response.

2. *Summary of Method*

- 2.1 The samples are prepared for analysis by gas chromatography/NCI-mass spectrometry-SIM using the appropriate sample preparation.
- 2.2 The compounds are introduced into the GC/MS through the injection of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with mass spectrometer (MS) connected to the gas chromatograph. If needed to minimize matrix enhancements the analytes can be split onto two detectors, a MSD and micro ECD in a 5:1 split ratio. The micro ECD may not be as affected by matrix enhancements as much as the MSD, when operated in the negative chemical ionization mode. In the dual detector mode, results could be reported from either the micro ECD, which is confirmed by the MSD as long as all data meets method criteria or NCI on its own. Both detectors require sample clean up before analysis. All soils/sludges and waters require extensive clean up before being analyzed (refer to clean up SOP DOC#: O-3-phase clean up-rev.1).
- 2.3 Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a calibration curve of five points or more. On the MSD, the target analytes are identified by comparing their selective mass ion ratios to the mass ion ratios of an authentic standard.

3. *Definitions*

The following terms are defined for use in this document:

- 3.1 ACCURACY: The closeness of agreement between an observed value and

an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

- 3.2 BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.
- 3.3 CONTROL SAMPLE: A QC sample introduced into a process to monitor the performance of the system.
- 3.4 FIELD DUPLICATES: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.
- 3.5 LABORATORY CONTROL A known matrix spiked with compound(s) representative of sample target analytes. This is used to document laboratory performance.
- 3.6 MATRIX: The component or substrate (e.g., surface water, drinking water), which contains the analyte of interest.
- 3.7 MATRIX DUPLICATE: An intralaboratory-split sample, which is used to document the precision of a method in a given sample matrix.
- 3.8 MATRIX SPIKE: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.
- 3.9 MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.
- 3.10 METHOD BLANK: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.
- 3.11 PRECISION: The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used estimates of precision are the

relative standard deviation (RSD) or the coefficient of variation (CV), $RSD = CV = 100 \frac{S}{\bar{x}}$,

where:

\bar{x} = the arithmetic mean of the measurements, and $S^2 = \sum (x_i - \bar{x})^2 / n$ variance; and the relative percent difference (RPD) when only two samples are available.

$RPD = 100 [(x_1 - x_2) / \{(x_1 + x_2) / 2\}]$.

- 3.12 REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
- 3.13 REAGENT WATER: Water that has been generated by any method, which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.
- 3.14 STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards, which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.
- 3.15 SURROGATE: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.
- 3.16 SIM: Selective ion monitoring
- 3.17 PCI: Positive Chemical Ionization
- 3.18 NCI: Negative Chemical Ionization
- 3.19 MSD: Mass selective detector.
- 3.20 GCMS: Gas Chromatography Mass Spectrometry
- 3.21 ECD: Electron Capture Detector
- 3.22 SOURCE: Component of MSD where ionization and ion focusing take place

4. Interferences

- 4.1 Solvents, reagents, glassware and other hardware used during extract and standards preparation can yield interferences. The lot numbers, the Caltest reagent ID number or Caltest source number of all reagents and solvents used for standards preparation should be recorded in the Standards Log Book to trace any sources of contamination that may occur.
- 4.2 Plastics should be avoided during the extract preparation procedure to eliminate sources of phthalate esters. Care should also be taken to limit contact of latex gloves with the sample or reagents. Only Teflon, glass or metal equipment should be used, such as Teflon squirt bottles, and all surfaces that come in contact with the sample should be solvent rinsed with methylene chloride.
- 4.3 Avoid cross-contamination between samples by rinsing any materials used for multiple samples between samples. Do not touch pipettes, graduated cylinders or squirt bottles to any of the glassware.
- 4.4 Matrix interference.
 - 4.4.1 IS Interference - Low or high internal standard areas may be corrected by diluting the sample or by using the external standard calibration technique if target compounds are not affected by the interference.
 - 4.4.2 Carryover - Contamination may occur from a previous injection. If this is suspected, re-analyze the sample with instrument blanks before and after the sample. Dirty extracts may require dilution and/or additional clean up to prevent interference, carryover, or overloading the column and detectors. Document any dilution or cleanup procedure in the Instrument Log Book. Footnote the report as appropriate.
 - 4.4.3 Dirty Injection Port - Maintenance such as replacing the injection port liner, seal, and clipping the front of the column should be done routinely.
 - 4.4.4 Column bleed - Rising baselines late in the chromatogram indicate column bleed. Utilize the column conditioning techniques outlined by the column manufacturer; trim both ends of the column, or solvent rinse the column.
 - 4.4.5 Chemical Ionization source requires more cleaning than the electron impact source, and cannot tolerate any residual water in the extracts or any air leaks. If either air or water is introduced into the chemical ionization source, response and mass signal to noise are severely degraded.

5. *Safety and Precautions*

- 5.1 This method is used to analyze potentially hazardous samples. Use of a hood is required.
- 5.2 Prior to performing this analysis, review the MSDS for all standards and reagents to be used. Observe the recommended safety precautions. Protective clothing and safety glasses should be worn when handling samples or reagents, and all manipulations are to be conducted in a hood.
- 5.3 Maintain a clean and uncluttered workspace. Return all chemicals, reagents and resultant wastes to their designated storage area at the completion of the test.

6. *Equipment and Supplies*

- 6.1 2 mL ALS vials, inserts, and crimp top seals.
- 6.2 Crimper, 12mm
- 6.3 Analytical column: 30 meter x 0.25 mm I.D., 0.25 μ m df, capillary fused silica.
- 6.4 Vials - 8 mL, 4 mL and 2 mL screw-cap vials with Teflon faced septum, clear and amber.
- 6.5 Syringes: 10 μ l, 25 μ l, 100 μ l, 500 μ l, 1000 μ l (Hamilton). Autosampler syringes: 10 μ l (Hamilton).
- 6.6 Volumetric flasks (Class A): 5.0 mL, 10.0 mL, 100.0 mL.
- 6.7 Data System: Agilent MS ChemStation (E.02.00.493) with ThruPut Target Data Analysis Software, Revision 4.12.
- 6.8 GC/MS system: Agilent 7890A-GC with Agilent 5975 inert XL/EI/CI-MSD equipped with Agilent 7683B autosampler.
- 6.9 Misc. Chromatography supplies: ferrules- 0.4mm (85% vespel-15% graphite, and 100% graphite) Restek 4.0mm double gooseneck deactivated injection port liners with glass wool, 1.2 mm inlet seals, 10 mm diameter septa.
- 6.10 Balance -Mettler analytical balance capable of reading to 0.01g.

7. *Reagents and Standards*

- 7.1 Methylene chloride, Burdick & Jackson, High purity solvent.
- 7.2 Acetone, Mallinkrodt, Nanograde.
- 7.3 Hexane, Omnisolve, high purity solvent.
- 7.4 Internal Standard Solution, 2000 ug/mL, 4,4'-Dibromooctafluorobiphenyl; Accustandard M-625-06-10x
Six-month expiration date upon opening ampule.
 - 7.4.1 Working Internal Standard Solution, 0.04 ug/mL.
Six month expiration date.
- 7.5 Calibration Standards: custom Pyrethroid standard, 1000ug/mL, Accustandard S-12458-R4
Six month expiration date upon opening ampules.
- 7.6 Surrogate mix: Decachlorobiphenyl, Accustandard, neat, C-209N One year expiration date upon opening ampule.
- 7.7 Working Standards with Surrogate-0.25 ppm, Six month expiration date.
- 7.8 Second source Standards: Custom Pyrethroid standard, 1000ug/ml. Accustandard, S- 12458-R4 (different lot and gravimetric weighing than the calibration standard)
Six month expiration date upon opening ampule.

8. *Sample Preservation and Storage*

- 8.1 The containers used for sampling and storage should be glass or Teflon and have screw caps with Teflon. All samples and extracts should be stored at not frozen to 6°C.
- 8.2 Aqueous Samples should be extracted within 14days of sampling, with the exception of Cyhalothrin and Permethrin which shows lack of stability after 3days (per USGS and CA Department of Food and Ag).
- 8.3 Soils can be frozen to extend the stability time to 6 months per USGS.

9. *Quality Control Procedures*

- 9.1 A method blank (MB) is included for every 24-hour extraction batch, or every 20 samples, and should be subjected to the same procedures as the samples. The blank is spiked with the same surrogate used in the samples. The blank should be free of contamination. If there are any target hits found in the analysis of the blank, a fresh extract aliquot should be prepped and run to determine if the contamination is a result of extraction or instrument contamination. If instrument contamination is suspected, run a solvent blank to confirm the problem.
- 9.2 Acceptance Criteria for Spiking Compounds in LCS and MS/MSD can be found in Caltest internal QC. Recovery outside of these limits must be checked and noted. The batch may need re-extraction.
- 9.3 Internal Standard QC-An area range of plus 100% and minus 50% is calculated from the average ISTD area from the calibration curve or from the continuing calibration standard. An ISTD area outside of the acceptance range necessitates prepping and analyzing a fresh extract to verify the results. If the problem is due to matrix interference, dilute the sample, inject ISTD and analyze. Repeated ISTD failures within the sequence indicate a problem with the system or ISTD solution. Correct any problems and rerun with a fresh ISTD solution. Re-prep fresh sample extracts if new ISTD solution has been prepared.

9.4 Surrogate QC-The surrogates, spike level, and the acceptance criteria are listed in Caltest internal QC.

9.5 Qualitative Data Analysis

- 9.5.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample quantitation of secondary and tertiary ion ratio. The reference selective mass ion ratios must be generated by the laboratory using the conditions of this method. Compounds should be identified as present when the criteria below are met
- 9.5.2 The intensities of the characteristic ions of a compound maximize at the same scan or within one scan of each other. Selection of a peak by a data system where identification is based on the presence of chromatographic peaks containing ions specific for the target compound at a known retention time will be accepted as meeting this criterion
- 9.5.3 The Relative Retention Time of the sample component is within ± 0.06 RRT units of the RRT of the standard component
$$RRT = \frac{RT \text{ sample}}{RT \text{ Istd}}$$

RT sample = Retention Time of sample component
RT Istd = Retention Time of internal standard.

- 9.5.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference selective mass ion ratios.
- 9.5.5 The GC oven program has been developed so that structural isomers either have partial resolution or elute close together so they can be integrated as one peak and the isomers are reported as a total pesticide residue.
- 9.5.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When a peak obviously represents more than one sample component, appropriate selection of analyte spectra and any necessary background spectra subtraction is important. Examination of extracted ion current profiles of target ions can aid in the selection of spectra and in the qualitative identification of compounds. When analytes co elute, the identification criteria can be met, but each analyte spectrum will contain a portion of the individual compounds co eluting.

10. Calibration and Standardization

- 10.1 Calibration Criteria-The GC/MS system must pass the following criteria prior to the analysis of any samples. Should any criterion not be met, the problem must be corrected before proceeding. (i.e.: instrument maintenance performed and/or standards and samples rerun.)
- 10.1.1 The GC/MS system must be tuned prior to sample analysis.
- 10.1.1.1 Adjust methane reagent gas flow in PCI mode before doing an auto tune. Then do the auto tune in the PCI mode first. Review the PCI auto tune report for air leaks and proper methane flow. Then load the NCI tune method and do a second auto tune in the NCI mode. If the auto tune completes, it passes. There is no tune check standard.
- 10.2 Initial Calibration -An initial calibration is performed prior to the analysis of any samples using a minimum of five points containing all the compounds of interest. The laboratory analyses standards that range from 0.0005ppm to 0.05pp
- 10.3 Using 4µl injections for the split mode or 3µl injections if only the MSD is being used. Each standard is acquired on the appropriate detector. Tabulate area responses against

concentration for each compound of interest including the Internal Standard, and calculate the Response Factor (RF) for each compound using the following equation:

$$RF = (AsCis)/(AisCs) \text{ where:}$$

As = Response for the parameter to be measured.

Ais = Response for the Internal Standard.

Cis = Concentration of the Internal Standard (ug/L).

Cs = Concentration of the parameter to be measured (ug/L).

The results are used to plot a calibration curve of Response Ratios, vs. RF. The linearity of the curve for each compound is checked and adjustments are made as necessary. A calibration curve is considered linear (acceptable) if the average RF is $\leq 15\%$ and/or the grand mean average RF of all the target analytes is $\leq 15\%$. If a quadratic curve is used you must use no less than 6 calibration points and achieve a correlation coefficient of 0.995 or higher (approaching 1.0).

- 10.4 A mid-level secondary standard is injected following the calibration curve to verify the validity of the primary standards. This standard is from a different Lot # and gravimetric weighing than those purchased for the initial calibration. All compounds should meet 30% acceptance criteria. Should any compound not meet the criteria, the samples will be additionally reviewed for that compound. Should the sample prove positive for that compound, the discrepancy between the calibration and the secondary standard will be resolved and the sample re run.

11. Procedure

- 11.1 Proper documentation is essential at all points of sample preparation.

- 11.1.1 Before preparing any sample extract for analysis, check the extraction sheet for any comments concerning extraction and multiplier; check the Semi-volatiles Worklist for any work notes, test requested, age, client ID, matrix, and sample description.

Confirm any anomalies against the Lab Confirmation or the Chain of Custody. Notify the department manager or project manager if there are any discrepancies.

- 11.1.2 The GC/MS Instrument Run Log must be created during the course of the analysis. See Doc. #: Q-LGBK for the correct procedure for logbook entries. The following information is needed to complete the GC/MS Instrument Logbook (See Appendix 1 for a sample of the logbook page).

- 11.1.2.1 Operator - Enter the Initials of the analyst(s) responsible for loading the samples and standards.

- 11.1.2.2 Method - Enter acquisition and data analysis method if separate.
- 11.1.2.3 Column - Analytical column
- 11.1.2.4 Comments-Lot #s for Standards, detailed information about sample dilutions (i.e.: 100µl-500µl=5x), and any other comments.
- 11.1.3 All containers or glassware used to hold the samples or QC should be clearly marked to prevent confusions or switching of samples.
- 11.1.4 ALS vials-Label with sample number, date extract was prepared for analysis, Analytical code, and any dilution made to the sample.
- 11.1.5 The Semi volatiles Standards Log Book must be completed whenever standards or reagents are prepared. See Doc. #: Q-LGBK for the correct procedure for logbook entries.
- 11.1.6 MSDF Maintenance Log Book: Any maintenance; routine or special, column changes, additions or changes performed on the system must be recorded in the MSDF Maintenance Log Book. The result of any maintenance procedures must also be recorded.

11.2 Acquisition

- 11.2.1 Refer to instrument maintenance logbook for run instrument parameters. Update run instrument parameters every time they are changed.

11.3 Analysis

- 11.3.1 The samples are extracted according to Caltest SOP's O-3510PREP, O-3550PREP and O- GCMSPREP. ALS vials are labeled appropriately (See 6.1.3.1). Samples may be diluted with hexane. **The ISTD must have equilibrated at room temperature before use.** A dilution on the sample, or a new curve must be prepared that will properly bracket the concentration of that compound. All dilutions are recorded on the extract vial, in the Instrument Log Book, within the analytical sequence and on the final LIMS fast track worksheet.

- 11.3.2 Sequence - A typical sequence is:

- 11.3.2.1 Mid level Standard Mix.(CCV): Pass = ±15% drift from ICAL RF for each individual analyte and /or grand mean average RF drift of all target analytes is ≤15%. If it does not pass recalibrate.

- 11.3.2.2 Secondary source standard, mid level.
(Only if a new calibration curve is run)
 - 11.3.2.3 QC samples-MB, LCS, LCSD, MS, MSD.
 - 11.3.2.4 Samples
 - 11.3.2.5 Closing response check (0.0005ppm) to check source inertness.
- 11.4 Sample extracts in the refrigerator at not frozen to 6°C until prepped and analyzed. Analyzed extracts should be archived until the data is reviewed for completeness. Neat extracts will be kept for 60 days past the extraction date. All extracts must be disposed in the proper manner.
- 11.5 Data Reporting, and Archival
- 11.5.1 Generate Quant Reports using Target software for samples and QC. IS and surrogate recoveries must pass QC criteria outlined in section 7 of this document. Corrective steps may be taken when necessary to achieve QC requirements (i.e.: manual integration of a peak, dilution of the sample, or other measures
 - 11.5.2 Using the Review feature of the Target software, check any hits that are suspect. Manual integration of a peak may be necessary to accurately quantitate the sample. Regenerate the report after any changes are made.
 - 11.5.3 Taking into account the multiplier for the sample, report concentrations of compounds that meet all of the following criteria:
 - 11.5.3.1 The mass spectral data meets the criteria set forth in section 10.
 - 11.5.3.2 The peak has a retention time that falls within the retention time window. See section 10.
 - 11.5.3.3 The concentration calculated is above the reporting limit taking into account the sample extraction and dilution factors.
 - 11.5.3.4 The experience of the analyst must weigh heavily in the interpretation of chromatograms.
- 11.6 Sample Data Entry - When reporting sample results on the LIMS system, enter the following data as appropriate:
- 11.6.1 DILUTION FACTOR: The detection limits in LIMS assume a dilution factor of 1 reflecting a 1L -> 1mL prep for aqueous samples and a 30g -> 2mL for

soils/sludges. All dilutions entered in LIMS should be rounded to one significant figure.). To calculate dilution factor multipliers, if two or more dilutions were made on a sample, enter the product dilution factor.

- 11.6.2 RESULTS - Report all hits with two significant figures. For non-detected target compounds, enter "0" for Non-Detect.
- 11.7 To report QC results on LIMS, first generate a TARGET QC report. Report all QC data results with three significant figures.
- 11.8 Completed data results are to be given to the coordinator/manager for data approval and filing.
- 11.9 Trouble shooting
- 11.9.1 Low response-Check the autosampler syringe for breakage or partial blockage. If press-tights connectors are being used, check them for leaks first. Check ferrule nuts at injector and detector and tighten if necessary. Check gas supplies and check for leaks. Replace septum. Check for septum particles in the injector liner. Cut several inches of column from the injector end and/or detector end. Low responses may also be caused by a dirty source, poorly performing filaments, or an old multiplier (voltage >2400mV)
- 11.9.2 No peaks-Check the autosampler syringe for breakage and replace if necessary. Check vu-unions or press-tights for leakage. Check the gas supplies and replace tanks as necessary. Verify flow through the column. Check injector liner for septum debris.
- 11.9.3 Large, misshapen peaks-Compounds in the sample may be overloading the detector. Dilute the sample and rerun.
- 11.9.4 Noisy baseline- remove column nuts and check for crushed ferrule and/or column. Remove a few inches of column, replace with a new ferrule and bake out system at 325° C for 30 minutes and check for baseline stability. Further problems may indicate a contaminated column requiring solvent rinsing or replacement.

12. Calculations

- 12.1 The internal standard technique -The Enviroquant GC software automatically calculates the concentrations of analytes using the following equation:

$$\begin{aligned} \text{Concentration, ug/L} &= \frac{(A_s) (I_s)}{(A_{is}) (RF)} \\ &= \end{aligned}$$

As = Quant ion response for the compound to be measured.
Is = Amount of internal standard.
Ais= Quant ion response for the internal standard.
RF = Response factor as determined in section 6.3.3

- 12.2 If the external standard technique is to be employed in cases where there is interference with the IS, calculate the amount of material injected from the peak response using the calibration curve. The concentration can be determined by using the following equation:

$$\text{Concentration } \mu\text{g/L} = \frac{A_{uk} \times C_s}{A_s}$$

Auk = Quant ion response of the compound being measured.
As = Quant ion response of the compound in the standard.
Cs = Concentration of the standard.

This technique should only be used when the target analytes are unaffected by matrix interferences. When used, any such calculations should be clearly annotated and footnoted on the LIMS report.

- 12.3 Multipliers are used to relate the results calculated in the final analysis to the reported units.

12.3.1 Liquid matrices

Analysis units = ug/mL Reporting units = ug/L

Result: on column amount x $\frac{\text{Final Volume}}{\text{Initial Volume}}$ x Dilution Factor

12.3.2 Solid matrices

Analysis units = ug/mL Reporting units = mg/Kg

Result: on column amount x $\frac{\text{Final Volume}}{\text{Initial Volume}}$ x Dilution Factor

13. *Pollution Prevention*

- 13.1 There are no particular pollution prevention steps taken by the laboratory for this test.

14. **Data Assessment and Acceptance Criteria for Quality Control Measures.**

14.1 The data is referenced in Caltest's internal QC summary manual.

15. *Corrective Actions for Out of Control Data.*

15.1 The data is referenced in Caltest's internal QC summary manual.

16. Contingencies for Handling Out-Of-Control or Unacceptable Data.

16.1 The contingencies for handling out-of-control or unacceptable data are addressed in the Caltest SOP: Q-CORRECT.

17. *Waste Management*

17.1 All solvent saturated aqueous waste is collected in a drum. The waste is shipped out for disposal using a hazard waste disposal company. It is profiled as waste corrosive liquid.

17.2 All dichloromethane is collected in a solvent drum. The waste is shipped out for disposal using a hazard waste disposal company. It is profiled as waste Dichloromethane.

17.3 All Acetone waste is collected in a drum. The waste is shipped out for disposal using a hazard waste disposal company. It is profiled as waste flammable liquid.

18. Method Performance

18.1 This is not currently available.

19. *References*

19.1 Analytical Method for the Determination of Synthetic Pyrethroids in Sediment by Gas Chromatography-Negative Chemical Ionization Mass Spectrometry. Neil J. Robinson, Syngenta Ltd.

Appendix 1

Pyrethroid List of Analytes

Pyrethroid Analyte	CAS Number	Ret Time	1°	2°	3°
Bifenthrin (Biphenthrin)	82657-04-3	19.7	386	387	241
Cyfluthrin (Baythroid)	68359-37-5	24.4	207	209	171
Lambda-Cyhalothrin	91465-08-6	21.5	241	205	243
Cypermethrin	52315-07-8	24.9	207	209	171
Fenvalerate / Esfenvalerate (Pydrin)	51630-58-1	26.7	211	213	212
Tau-Fluvalinate	102851-06-9	26.7	294	296	295
Fenpropathrin *(Danitol)	39515-41-8	20.0	141		
Permethrin	52645-53-1	23.3	207	209	
Tralomethrin/ Deltamethrin	66841-25-6	27.8	81	295	297
Allethrin	584-79-2	13.6	167	168	
Tetramethrin	7696-12-0	19.8	331	332	
Phenothrin	26002-80-2	19.8	167	168	

Additional Analyte per request:

Analyte	CAS Number		1°	2°	3°
Chlorpyrifos	2921-88-2	12.3	315	214	313
*Diazinon	333-41-5	9.6	169		

Ret Time = retention time.

1° = Quantitation Ion

2° = Qualifying Ion

3° = Monitoring Ion

* = NCI only produces one ion; the compound qualifies using 1° and retention time.

APPENDIX XXXIII. PTS SOP #3. PARTICLE SIZE BY DRY SIEVE PROCEDURE (Method: ASTM D422-63 Reapproved 1998) (Revision number unknown, 1998)

PARTICLE SIZE BY DRY SIEVE PROCEDURE

(Method: ASTM D422-63 Reapproved 1998)

Scope

Particle size distribution of sediments is determined by sieving which consists of using a set of standard square-mesh, woven-wire cloth sieves that conform to ASTM E-11 in conjunction with a Ro-Tap mechanical shaker unit.

Weighed particles can range from 0.25 to 0.0012 inches and are determined as the fraction of each sample is collected and weighed in each standard sieve. The complete set of sieves is:

<u>U.S. Sieve Number</u>	<u>Opening, Inches</u>	<u>Opening, mm</u>	<u>Phi of Screen</u>
1	0.9844	25.002	-4.64
1/2	0.4922	12.501	-3.64
3/8	0.3740	9.500	-3.25
1/4	0.2500	6.351	-2.67
4	0.1875	4.757	-2.25
6	0.1324	3.364	-1.75
10	0.0787	2.000	-1.00
14	0.0557	1.414	-0.50
18	0.0394	1.000	0.00
25	0.0278	0.707	0.50
35	0.0197	0.500	1.00
40	0.0166	0.420	1.25
45	0.0139	0.354	1.50
60	0.0098	0.250	2.00
80	0.0070	0.177	2.50
120	0.0049	0.125	3.00
200	0.0029	0.074	3.75
270	0.0021	0.053	4.25
400	0.0015	0.037	4.75

Method Summary

The soil sample is dried and gently disaggregated using a wooden mortar and pestle. Samples that contain heavy concentrations of hydrocarbons are extracted by Dean-Stark distillation (API RP40) using either toluene or a chloroform-methanol azeotrope as the solvent prior to disaggregation. A representative portion of the sample, 50-1000 grams, is introduced into a stack of sieves beginning with U.S. Standard Sieve number 1/4 and is continuously shaken on the Ro-Tap shaker unit for a period of no less than 20 minutes and no greater than 30 minutes. Weights of the sample retained on each standard sieve number are recorded.

Quality Control

Calibration is determined by standards created by PTS Laboratories, Inc. by multiple size separation runs. Each screen is inspected prior to use for signs of excessive wear, distortion of the wire cloth or other aberrations. Sieves suspected of having a flaw are replaced and given to the laboratory supervisor for microscopic inspection, after which they are tested with standards, repaired or discarded. Duplicate samples are run for each batch of twenty samples and blind mixes of standards are run weekly. Duplicate sample acceptance ranges are 80-120 %, blind sample acceptance ranges are 90-110%.



PARTICLE SIZE BY DRY SIEVE PROCEDURE

(Method: ASTM D422-63 Reapproved 1998)

Reporting

Data is reported in tabular and graphical formats corresponding to the ASTM/USCS Classification system and can be presented in an EDD format. Statistical data of mean, median, standard deviation, sorting, skewness and kurtosis are included.

SAMPLE PREPARATION FOR PARTICLE SIZE ANALYSIS PROCEDURE

(ASTM D421/422M Method)

Sampling

1. Sub-sample approximately 20-30 grams of representative material at the requested interval.
2. Break up aggregations using a wood or rubber-covered pestle.
3. Dry sample at room temperature until weight is stable.
 - a. If RUSH turnaround time is required, dry sample at 150°F until weight is stable.
 - b. If sample is contaminated with petroleum hydrocarbons, sample must be cleaned.
 - i. Package sample in thimble or cellulose envelope for Dean-Stark or Soxhlet extraction.
 - ii. Extract for 4-8 hours or until sample is clean.
 - iii. Dry sample at 150°F in vacuum oven until weight is stable.
4. For LPSA analysis, material passing a No. 10 (2.00-mm) sieve is required.
5. **Note:** For non-sediment (soil) samples, contact client to develop procedure.

ATTACHMENTS

ASTM D422 Method

PARTICLE SIZE BY LASER LIGHT SCATTERING PROCEDURE

(Method: ASTM D4464M-85)

Scope

Particle size distribution of sediments is determined by using laser light diffraction to measure the amount and patterns of light scattered by a particle's surface. This is done using laser light with a 750 nm wavelength, a Fourier lens focusing system and an array of 126 detectors. Light is diffracted around a particle at angles inversely proportional to the size of the particle; the smaller the particle the larger the angle of diffraction.

Measured particles can range from 0.4-2000 μm and are determined as the sample, dispersed in a transport fluid, is circulated through the analyzer. Extended range analyses, 0.4 - >2000 μm is accomplished by combining the >2000 μm fraction from screen sieving with the <2000 μm fraction from light scattering.

The procedure is a modification of ASTM D4464-85 (reapproved in 2000) to measure the particle size of catalytic material. The modification is that the procedure has been extended to include the measurement of unconsolidated soils and sediments. The procedure is in current use by the USGS, City of Los Angeles, University of Florida and major petroleum companies. Local, state and federal regulatory agencies have recognized the method as an alternative to ASTM D422 (hydrometer) and the pipette method.

Method Summary

For soils the sample is dried and gently disaggregated using a wooden mortar and pestle. Samples that contain heavy hydrocarbons are extracted by Dean-Stark distillation (API RP40) using either toluene or a chloroform-methanol azeotrope as the solvent prior to disaggregation. A representative portion of the sample, 5-10 grams, is introduced into the fluid module of the analyzer and is continuously circulated through the laser beam. Water samples are introduced directly into the fluid module. The circulation fluid is filtered to 0.2 μm and contains a dispersant to prevent coagulation along with an internal sonification unit to further separate the particles.

The laser beam passes through the sample cell where the suspended particles scatter the incident light. Fourier optics collect the diffracted light and focus it on to three sets of detectors, one for low-angle scattering, the second for mid-angle scattering and the third for high-angle scattering. The composite, time-averaged diffraction pattern is measured by 126 detectors placed at angles to $\sim 35^\circ$ from the optical axis. Sizes are computed by the Fraunhofer Model for Light Scattering and summed into normal ASTM, USCS or Wentworth distribution classifications for 0.4-2000 μm .

Quality Control

Calibration is determined by the optical design. Therefore, no calibration is required. All necessary adjustments are made by measuring electrical offsets and aligning the laser beam. Quality control samples of traceable diameter are run at regular intervals or as required on a project basis. Repeatability is <1% about the mean size (repeat runs of the same sample).

Reporting

Data is reported in tabular and graphical formats corresponding to the ASTM/USCS Classification system and can be presented in an EDD format. Statistical data of mean, median, standard deviation, sorting, skewness and kurtosis are included.

SAMPLE PREPARATION FOR PARTICLE SIZE ANALYSIS PROCEDURE (ASTM D421/422M Method)

Sampling

1. Sub-sample approximately 20-30 grams of representative material at the requested interval.
2. Break up aggregations using a wood or rubber-covered pestle.
3. Dry sample at room temperature until weight is stable.
 - a. If RUSH turnaround time is required, dry sample at 150°F until weight is stable.
 - b. If sample is contaminated with petroleum hydrocarbons, sample must be cleaned.
 - i. Package sample in thimble or cellulose envelope for Dean-Stark or Soxhlet extraction.
 - ii. Extract for 4-8 hours or until sample is clean.
 - iii. Dry sample at 150°F in vacuum oven until weight is stable.
4. For LPSA analysis, material passing a No. 10 (2.00-mm) sieve is required.
5. **Note:** For non-sediment (soil) samples, contact client to develop procedure.

LPSA Sample Preparation

1. Disaggregate sample material to grain size using wood or rubber-covered pestle being careful not to break individual grains.
2. Separate test sample by sieving with a No. 10 (2-mm) sieve.
 - a. Record weight of any retained material and passing material weight.
3. By use of a micro sample splitter, select a portion of sample suitable for occlusion (8-12 percent occlusion) of the Laser Particle Size Analyzer (LPSA).
4. Place the sample in a 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution.
 - a. A solution of sodium hexametaphosphate is prepared at a rate of 40 grams of sodium hexametaphosphate per liter of distilled or demineralized water.
 - b. Stir or agitate until the soil is thoroughly wetted.
 - c. Allow to soak for at least 16 hours.
5. Following soaking period, disperse sample prior to introduction into Laser Particle Size Analyzer.
6. Stir with stainless steel or glass stirrer or use sonicator.
7. **Note:** For non-sediment (soil) samples, contact client to develop procedure.

LPSA Analysis

1. Wash sample into LPSA fluid module using distilled or demineralized water making sure all sample is washed from beaker.
2. Allow to circulate for at least 20 seconds or until the sample is completely dispersed before measuring.
3. Measure sample particle size distribution per Method ASTM D4464M.

Test results are presented in tabular and graphical formats.

PARTICLE SIZE ANALYSIS PROCEDURE
(ASTM D421/422M Method, ASTM D4464M)

Additional

Dry Sieve data may be mathematically combined with Laser Diffraction data to create a combination report presenting grain size distribution from 1" to 0.375um.

ATTACHMENTS

ASTM D4464M Method



Standard Test Method for Particle-Size Analysis of Soils¹

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Note 1 and Note 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425- μm), or No. 200 (75- μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (1) a high-speed mechanical stirrer, and (2) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20- μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm .

2. Referenced Documents

2.1 ASTM Standards:

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

E 100 Specification for ASTM Hydrometers⁴

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated

stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than $\frac{3}{4}$ in. (19.0 mm) nor more than $1\frac{1}{2}$ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup⁵ (Note 3) conforming to the general details shown in Fig. 3 (Note 4 and Note 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft³/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chut and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and $2\frac{1}{2}$ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

¹ This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 – 62.

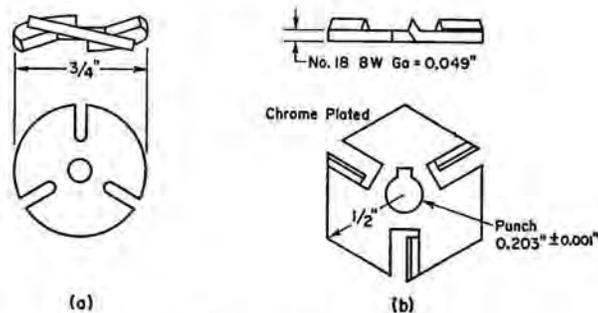
² Annual Book of ASTM Standards, Vol 04.08.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vol 14.03.

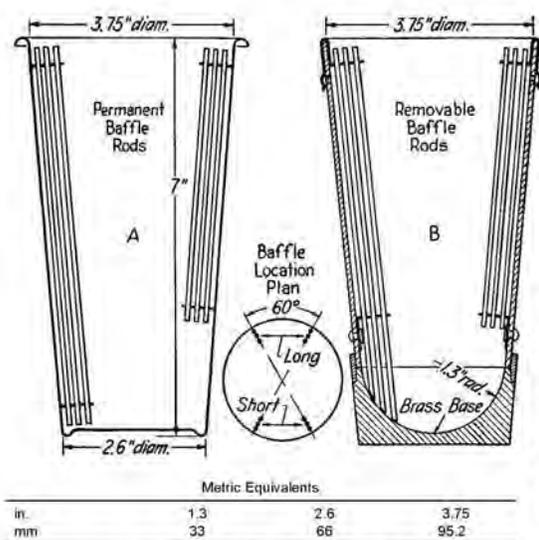
⁵ Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19105. Order Adjunct No. 12-404220-00.

D 422



Metric Equivalents					
in.	0.001	0.049	0.203	1/2	3/4
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles



Metric Equivalents			
in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

3-in. (75-mm)	No. 10 (2.00- μ m)
2-in. (50-mm)	No. 20 (850- μ m)
1 1/2-in. (37.5-mm)	No. 40 (425- μ m)
1-in. (25.0-mm)	No. 60 (250- μ m)
3/4-in. (19.0-mm)	No. 140 (106- μ m)
3/8-in. (9.5-mm)	No. 200 (75- μ m)
No. 4 (4.75-mm)	

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1 1/2-in. (37.5-mm)	No. 30 (600- μ m)
3/4-in. (19.0-mm)	No. 50 (300- μ m)
3/8-in. (9.5-mm)	No. 100 (150- μ m)
No. 4 (4.75-mm)	No. 200 (75- μ m)
No. 8 (2.36-mm)	

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure

D 422

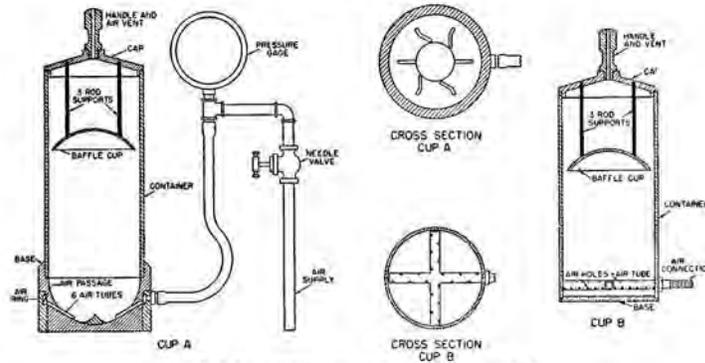
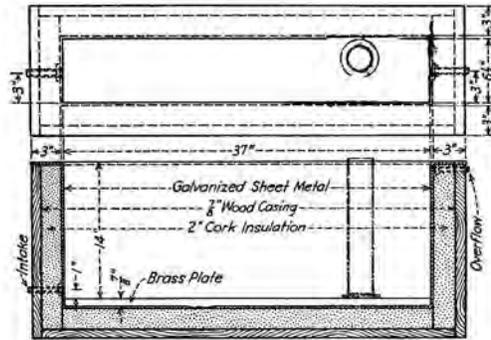


FIG. 3 Air-Jet Dispersion Cups of Apparatus B



Metric Equivalents						
in.	3/8	1	3	6 1/4	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
3/8 (9.5)	500
1/4 (19.0)	1000
1 (25.4)	2000
1 1/2 (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in.

(50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅝-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-MM) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus

formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil-water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil-water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil-water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil-water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil-water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75- μ m) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the $\frac{3}{8}$ -in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the $\frac{3}{8}$ -in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hygroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100 000/W) \times G(G - G_s)](R - G_s) \quad (1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100 \quad (2)$$

where:

- a = correction fraction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1).
- P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,
- R = hydrometer reading with composite correction applied (Section 7),
- W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,
- G = specific gravity of the soil particles, and
- G_s = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_s .

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law: see Table 2

TABLE 1 Values of Correction Factor, α , for Different Specific Gravities of Soil Particles^a

Specific Gravity	Correction Factor ^a
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

^aFor use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

$$D = \sqrt{[30m/980(G - G_j)] \times L/T} \quad (3)$$

where:

- D = diameter of particle, mm,
- n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),
- L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (see Table 2)),
- T = interval of time from beginning of sedimentation to the taking of the reading, min,
- G = specific gravity of soil particles, and
- G_j = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows: see Table 3

$$D = K\sqrt{L/T} \quad (4)$$

where:

- K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A - and B -scales, the square root being indicated on the D -scale. Without ascertaining the value of the square root it may be multiplied by K , using either the C - or CT -scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes^a

Hydrometer 151H		Hydrometer 152H	
Actual Hydrometer Reading	Effective Depth, L , cm	Actual Hydrometer Reading	Effective Depth, L , cm
1.000	16.3	0	16.3
1.001	16.0	1	16.1
1.002	15.8	2	16.0
1.003	15.5	3	15.8
1.004	15.2	4	15.6
1.005	15.0	5	15.5
1.006	14.7	6	15.3
1.007	14.4	7	15.2
1.008	14.2	8	15.0
1.009	13.9	9	14.8
1.010	13.7	10	14.7
1.011	13.4	11	14.5
1.012	13.1	12	14.3
1.013	12.9	13	14.2
1.014	12.6	14	14.0
1.015	12.3	15	13.8
1.016	12.1	16	13.7
1.017	11.8	17	13.5
1.018	11.5	18	13.3
1.019	11.3	19	13.2
1.020	11.0	20	13.0
1.021	10.7	21	12.9
1.022	10.5	22	12.7
1.023	10.2	23	12.5
1.024	10.0	24	12.4
1.025	9.7	25	12.2
1.026	9.4	26	12.0
1.027	9.2	27	11.9
1.028	8.9	28	11.7
1.029	8.8	29	11.5
1.030	8.4	30	11.4
1.031	8.1		
1.032	7.8		
1.033	7.6		
1.034	7.3		
1.035	7.0		
1.036	6.8		
1.037	6.5		
1.038	6.2		

^aValues of effective depth are calculated from the equation:
 $L = L_1 + 1/2 [L_2 - (V_h/A)]$ (5)

where:

- L = effective depth, cm,
- L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
- L_2 = overall length of the hydrometer bulb, cm,
- V_h = volume of hydrometer bulb, cm³, and
- A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table 2 are as follows:

- For both hydrometers, 151H and 152H:
 - L_2 = 14.0 cm
 - V_h = 67.0 cm³
 - A = 27.8 cm²

- For hydrometer 151H:
 - L_1 = 10.5 cm for a reading of 1.000
 - = 2.3 cm for a reading of 1.031

- For hydrometer 152H:
 - L_1 = 10.5 cm for a reading of 0 g/litre
 - = 2.3 cm for a reading of 50 g/litre

test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature, ^a C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01496	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01225	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

- (1) Gravel, passing 3-in. and retained on No. 4 sieve %
- (2) Sand, passing No. 4 sieve and retained on No. 200 sieve %
 - (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve %
 - (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve %
 - (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve %
- (3) Silt size, 0.074 to 0.005 mm %
- (4) Clay size, smaller than 0.005 mm %
 - Colloids, smaller than 0.001 mm %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS	
Sieve Size	Percentage Passing
3-in. %
2-in. %
1½-in. %
1-in. %
¾-in. %
½-in. %
No. 4 (4.75-mm) %
No. 10 (2.00-mm) %
No. 40 (425-µm) %
No. 200 (75-µm) %
HYDROMETER ANALYSIS	
0.074 mm %
0.005 mm %
0.001 mm %

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-µm) sieves may be substituted for No. 10 and No. 40 sieves.

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

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Standard Test Method for Particle Size Distribution of Catalytic Material by Laser Light Scattering¹

This standard is issued under the fixed designation D 4464; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the particle size distribution of catalyst and catalyst carrier particles and is one of several found valuable for the measurement of particle size. The range of particle sizes investigated was 30 to 300 μm equivalent spherical diameter. The technique is capable of measuring particles above and below this range. The angle and intensity of laser light scattered by the particles are selectively measured to permit calculation of a volume distribution using light-scattering techniques.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 3766 Terminology Relating to Catalysts and Catalysis²
- E 105 Practice for Probability Sampling of Materials³
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³
- E 456 Terminology Relating to Quality and Statistics³
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³
- E 1617 Practice for Reporting Particle Size Characterization Data³

3. Terminology

3.1 Definitions and recommended nomenclature pertaining to catalysts and to materials used in their manufacture can be found in Terminology D 3766.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *background*—extraneous scattering of light by material present in the dispersion fluid other than the particles to be measured. It includes scattering by contamination in the measurement path.

3.2.2 *Fraunhofer Diffraction*—the optical theory that de-

scribes the low-angle scattering of light by particles that are large compared to the wavelength of the incident light.

3.2.3 *Mie Scattering*—the complex electromagnetic theory that describes the scattering of light by spherical particles. It is usually applied to particles with diameters that are close to the wavelength of the incident light. The real and imaginary indices of light refraction of the particles are needed.

3.2.4 *multiple scattering*—the re-scattering of light by a particle in the path of light scattered by another particle. This usually occurs in heavy concentrations of a particle dispersion.

4. Summary of Test Method

4.1 A prepared sample of particulate material is dispersed in water or a compatible organic liquid and is circulated through the path of a laser light beam or some other suitable source of light. The particles pass through the light beam and scatter it. Photodetector arrays collect the scattered light which is converted to electrical signals to be analyzed using Fraunhofer Diffraction, or Mie Scattering, or both. Scattering information, typically, is analyzed assuming a spherical geometry for the particles. Calculated particle sizes are, therefore, presented as equivalent spherical diameters.

5. Significance and Use

5.1 It is important to recognize that the results obtained by this test method or any other method for particle size determination utilizing different physical principles may disagree. The results are strongly influenced by physical principles employed by each method of particle size analysis. The results of any particle sizing method should be used only in a relative sense and should not be regarded as absolute when comparing results obtained by other methods.

5.2 Light scattering theories (Fraunhofer Diffraction⁴ and Mie Scattering⁵) that are used for determination of particle size has been available for many years. Several manufacturers of testing equipment now have units based on these principles. Although each type of testing equipment utilizes the same basic principles for light scattering as a function of particle size, different assumptions pertinent to application of the theory and different models for converting light measurements

¹ This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.02 on Physical-Mechanical Properties.

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² Annual Book of ASTM Standards, Vol 05.05.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Born, M., and Wolf, E., *Principles of Optics*, Chptr 8, Pergamon Press, Oxford, 1957.

⁵ van Hulst, H.C., *Light Scattering by Small Particles*, Chptr 9, John Wiley & Sons, New York, 1908.

to particle size, may lead to different results for each instrument. Furthermore, any particles which are outside the size measurement range of the instrument will be ignored, causing an increase in the reported percentages within the detectable range. A particle size distribution which ends abruptly at the detection limit of the instrument may indicate that particles outside the range are present. Therefore, use of this test method cannot guarantee directly comparable results from different types of instruments.

5.3 This test method can be used to determine particle size distributions of catalysts and supports for materials specifications, manufacturing control, and research and development work.

6. Interferences

6.1 Air bubbles entrained in the circulating fluid will scatter light and then be reported as particles. Circulating fluids, typically, do not require degassing, but should be bubble-free on visual inspections.

6.2 Contaminants, such as non-aqueous solvents, oil or other organic coatings on the sample may emulsify in an aqueous carrier, scatter light, and be reported as part of the particle size distribution. Samples containing such contaminants may be analyzed in a non-aqueous carrier solvent to dissolve the contaminants or washed free of the contaminant with a compatible aqueous solvent.

6.3 Reagglomeration or settling of particles during analysis will cause erroneous results. Dispersions shall be prepared so a stable dispersion is maintained throughout the analysis.

6.4 Insufficient sample loading may cause electrical noise interference and poor data reproducibility. High sample loading may cause excessive light attenuation and multiple scattering, resulting in erroneous particle size distributions.

7. Apparatus

7.1 *Particle Size Analyzer*, based on Fraunhofer Diffraction or Mie Scattering, or both, light scattering analysis techniques. Ensure that the analyzer system or subsystem is optimum for the range of the powder being tested.

7.2 *Micro Sample Splitter*, used in accordance with MNL 32,⁶ to obtain the test portion of sample.

7.3 *Ultrasonic Probe or Bath*, if needed, to ensure dispersion of agglomerates prior to analysis.

8. Reagents and Materials

8.1 The selected liquid carrier shall:

8.1.1 Be compatible with the construction materials of the sample delivery system.

8.1.2 Not cause dissolution or clumping of the particles.

8.1.3 Be sufficiently clean to achieve acceptable background levels.

8.2 The use of surfactant(s) is often recommended by equipment manufacturers. However, agents such as surfactants, antifoams, and viscosity modifiers should be used with caution. An interlaboratory study of this test method showed that the

use of different types and concentrations of surfactant can significantly affect the results. In calculating the precision of this test method, results obtained using surfactants were excluded because they contributed disproportionately to the scatter in results. Comparisons between laboratories should be performed with liquid carriers which are identical in all respects.

9. Sampling and Sample Size

9.1 A representative test sample shall be obtained according to Practice E 105. The test portion shall be extracted from the test sample using a micro sample splitter according to Manual 32. Quartering shall not be used.

9.2 Refer to the equipment manufacturer's recommendation to ensure that the amount of the test portion is acceptable to achieve optimum light scattering conditions. A wide range of sample portions is acceptable depending upon median particle size, particle density, and the sample delivery system.

9.3 For liquid dispersed materials, redisperse as necessary to ensure representative samples.

10. Preparation of Apparatus

10.1 Allow the instrument to warm up according to the manufacturer's recommendations.

10.2 Install and fill the desired sample delivery system and select applicable instrument range as indicated by the instrument manufacturer's instructions.

10.3 Establish correct optical alignment and calibration at a frequency in accordance with the manufacturer's requirements.

11. Calibration and Standardization

11.1 Performance of the instrument is defined by the geometry of the optical components. (Refer to the manufacturer's instruction manual.)

11.2 Spherical particle standards are available. Diagnostic powders are available from some equipment manufacturers to ensure consistent instrument function. (Some instruments may permit the use of reticles for calibration.)

NOTE 1—A partial list of standards, powders, and reticles can be found in the D32 research report for this test method.

12. Procedure

12.1 Measure the background in the mode in which the analysis will be carried out. Be sure that the carrier is flowing through the light path while measuring the background. Background values shall not exceed the manufacturer's specifications. If background values exceed manufacturer's recommendations, perform the necessary procedures as specified by the manufacturer to bring the background values within acceptable limits.

12.2 Obtain representative sample according to Section 9.

12.3 Select appropriate run time for the sample. This procedure is very specific to the application and is generally gauged by the run-to-run repeatability.

12.4 Select the desired output parameters according to the requirements set forth by the instrument manufacturer.

12.5 Transfer a representative aliquot to the sample delivery system and allow it to circulate for at least 20 s or until the solid is uniformly dispersed before measuring. (Determine that

⁶ASTM Manual Series: MNL 32, "Manual on Test Sieving Methods," Pope, L.R. and Ward, C.W., eds., 4th ed, 1998.

the sample is not settling out in the circulation system. This can be checked by repeated runs at higher circulation rates.)

12.6 Perform the sample analysis according to the manufacturer's instructions.

12.7 Drain and fill the sample dispersion system in preparation for the next sample analysis. Drain and clean, as necessary, to avoid contamination of the subsequent sample.

13. Report

13.1 Information shall be reported as agreed between supplier and user, in accordance with Practice E 1617. The basis of the reported results is percent volume distribution calculated as equivalent spherical diameter. If all particles have the same density, this is the same as percent weight distribution.

14. Precision and Bias ⁷

14.1 *Test Program*—An interlaboratory study was conducted in which particle size was measured as three points in seven separate laboratories on three materials. Each laboratory conducted a single determination on each of three subsamples of each material, Practice E 691, modified for nonuniform data sets, was followed for the data reduction.

14.2 *Precision*—Pairs of test results obtained by a procedure similar to that described in the study are expected to differ in absolute value by less than the 95 % probability interval, which is equal to 2.772*S, where S is the estimate of standard deviation.

⁷ Use of the terms repeatability, reproducibility, precision and bias is in accordance with Terminology E 456 and Practice E 177. Supporting data are available from ASTM Headquarters. Request RR: D32-1013.

14.3 *Summary of Precision Results*—The test results are shown in micrometres as Fraction Smaller Than (FST) at the indicated diameter at three selected points on the cumulative particle size distribution. Repeatability is the within laboratory agreement and reproducibility is the agreement between laboratories, expressed both in micrometres and as percent of consensus mean. See Table 1.

14.4 *Bias*—The test method is without known bias.

15. Keywords

15.1 catalyst; Fraunhofer Diffraction; light scattering; Mie Scattering

TABLE 1 Precision

Fraction Smaller Than (FST)	Consensus Median Diameter, μm	Repeatability	Reproducibility
<i>D3295001</i>			
10 volume %	37.11	0.81 (1.65 %)	4.68 (12.5 %)
50 volume %	68.04	1.18 (1.74 %)	2.85 (4.19 %)
90 volume %	120.8	3.69 (2.98 %)	9.66 (9.01 %)
<i>D3291003</i>			
10 volume %	39.71	1.91 (4.81 %)	8.59 (21.6 %)
50 volume %	83.28	3.13 (3.76 %)	7.49 (8.99 %)
90 volume %	153.2	6.82 (4.45 %)	16.31 (10.7 %)
<i>D3295003</i>			
10 volume %	57.79	5.53 (9.57 %)	12.30 (21.3 %)
50 volume %	140.4	9.70 (6.91 %)	16.38 (11.7 %)
90 volume %	254.6	17.53 (6.88 %)	53.82 (21.1 %)

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APPENDIX XXXIV. PTS SOP #4. TOTAL ORGANIC CARBON PROCEDURE (Method: Walkley-Black) (Revision number unknown, date unknown)

TOTAL ORGANIC CARBON PROCEDURE

(Method: Walkley-Black)

Scope

The Walkley-Black method is a common acid digestion method for the analysis of organic matter in soils. The method in use since the 1930's uses chromic acid to measure the oxidizable organic carbon in the soil. The Walkley-Black method is very accurate and precise on soils with less than 2% organic matter. The Walkley-Black method may result in low-test results for soils with excessively high concentrations of organic matter due to the incomplete oxidation of organic carbon in the sample. The upper limit for the method is approximately 6% organic matter. The loss on ignition method should be used for soils containing >6% organic matter.

Method Summary

The Walkley-Black method involves a known volume of acidic dichromate solution reacting with an aliquot of soil in order to oxidize the organic carbon. The oxidation step is then followed by titration of the excess dichromate solution with ferrous sulfate. The organic carbon is calculated using the difference between the total volume of dichromate added and the volume titrated after reaction.

Laboratory Procedure

1. Obtain 15 grams of representative soil.
2. Extract samples by Dean-Stark method (if there is evidence of hydrocarbon) and dry for 16 hours in a vacuum drying oven at 150°F.
3. Grind soil using a mortar and pestle (no steel or iron) until it can pass through a #40 sieve (< 0.5 mm) making sure to clean the mortar and pestle between samples with methanol.
4. Weigh out 0.5-2 grams of soil. 0.5-1.5 grams for samples that appear to have organic material, 1-2 grams for clean samples. Record sample weight in TOC logbook along with ID, client, file number, date and flask number.
5. Transfer weighed material to a 500 ml Erlenmeyer flask.
6. Add 10 ml EXACTLY of fluid #1 (0.5N $K_2Cr_2O_7$) to each flask and agitate for 30 seconds making sure of complete mixing.
7. Add 20 ml EXACTLY of sulfuric acid, H_2SO_4 , and increase the speed on the shaker to 2 on the speed setting. Agitate for one minute.
8. Set lab timer for 30 minutes and allow flasks to cool.
9. Add EXACTLY 200 ml of deionized water to each flask.
10. Remove flask from the shaker unit and add 3-4 drops of fluid #2 (Ferrioin, α -Phenanthroline-ferrous complex 0.025M).
11. Drop a small clean stir bar into the flask. Place flask on stir plate and spin at low speed.
12. Record the starting volumetric reading for fluid #3 (0.5N Ferrous sulfate heptahydrate solution) burette in the TOC logbook. Always start the burette at the 25 ml mark or lower.
13. Slowly add fluid #3 until the mixture turns dark green, and then titrate drop by drop until the color changes sharply from blue to red.
14. Record the final burette reading in the TOC logbook.
15. Using the excel file TOC.xls, calculate the TOC value for each sample according to the following formula:

Organic Carbon, % = $[(\text{meq } K_2Cr_2O_7 - \text{meq } FeSO_4) \times 0.003 \times 100] / \text{soil weight, g} \times \text{response factor, rf}$.

TOTAL ORGANIC CARBON PROCEDURE

(Method: Walkley-Black)

Reporting

Data is reported in tabular format and can be presented in an EDD form at.

Quality Control

Blank Acceptance Range (RF): 97-103%, 1.261-133.9

Standard Acceptance Range: 70-130%

Method Detection Limit: 100 mg/kg (ppm)

- (1) As listed in **METHODS OF SOIL ANALYSIS, Part 2, Chemical and Microbiological Properties, Second Edition:**
American Society of Agronomy, Inc.; Soil Science Society of America, Inc. 1982.

ATTACHMENTS

Walkley-Black procedure.

TOTAL ORGANIC CARBON
WALKLEY-BLACK METHOD

GENERAL INFORMATION							0.5N FeSO ₄ Titration			CALCULATED DATA					
Run Date	PTS File Number	Client	Project Number	Sample ID	Depth, ft	Flask Number	Soil Weight, g	Start Burette	End Burette	Total	K ₂ Cr ₂ O ₇ meq	FeSO ₄ meq	Response factor	TOC, %	TOC, mg/kg
20-Jan	33999	Client		MW-1		26	0.471	0.000	18.750	18.75	10.002	9.442	1.30	0.46	4636
20-Jan				SQC Blank		21	0.000	0.000	19.750	19.75	10.002	9.946	1.30	0.00	0
20-Jan				SQC Standard		22	0.460	0.000	17.850	17.85	10.002	8.989	1.30	0.86	8590

Blank Acceptance Range (RF): 97-103%
Standard Acceptance Range: 70-130%
Method Detection Limit: 100 mg/kg (ppm)

Rounding:
100-999: Nearest 10
1000-9999: Nearest 50
>10000: Nearest 100

All values between -999 and 99 are listed as <100 mg/kg

obtained if dichromate methods are applied to soils containing significant amounts of carbonized materials. Dry combustion methods are most appropriate for soils containing large amounts of elemental C.

29-3.5.2 WALKLEY-BLACK PROCEDURE (Walkley, 1946; Peck et al., 1947; Greveling & Peck, 1960)

29-3.5.2.1 Reagents

1. Potassium dichromate ($K_2Cr_2O_7$), 1N: Dissolve 49.04 g of reagent-grade $K_2Cr_2O_7$ (dried at $105^\circ C$) in water, and dilute the solution to a volume of 1,000 ml.
2. Sulfuric acid (H_2SO_4), concentrated (not less than 96%): If Cl^- is present in soil, add Ag_2SO_4 to the acid at the rate of 15 g/liter.
3. Phosphoric acid (H_3PO_4), concentrated.
4. *o*-Phenanthroline-ferrous complex, 0.025M: Dissolve 14.85 g of *o*-phenanthroline monohydrate and 6.95 g of ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) in water. Dilute the solution to a volume of 1,000 ml. The *o*-phenanthroline-ferrous complex is available under the name of Fer-rolin from the G. Frederick Smith Chemical Co. (Columbus, Ohio).
5. Barium diphenylamine sulfonate: Prepare a 0.16% aqueous solution. This reagent is an optional substitute for no. 4.
6. Ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) solution, 0.5N: Dissolve 140 g of reagent-grade $FeSO_4 \cdot 7H_2O$ in water, add 15 ml of some sulfuric acid (H_2SO_4), cool the solution, and dilute it to a volume of 1,000 ml. Standardize this reagent daily by titrating it against 10 ml of 1N potassium dichromate ($K_2Cr_2O_7$), as described below.

29-3.5.2.2 Procedure. Grind the soil to pass through a 0.5-mm sieve, avoiding Fe or steel mortars. Transfer a weighed sample, containing 10 to 25 mg of organic C, but not in excess of 10 g of soil, into a 500-ml wide-mouth Erlenmeyer flask. Add 10 ml of 1N $K_2Cr_2O_7$, and swirl the flask gently to disperse the soil in the solution. Then rapidly add 20 ml of conc H_2SO_4 , directing the stream into the suspension. Immediately swirl the flask gently until soil and reagents are mixed; then more vigorously for a total of 1 min. Allow the flask to stand on a sheet of asbestos for about 30 min. Then add 200 ml of water to the flask, and filter the suspension if experience shows that the endpoint of the titration cannot otherwise be clearly discerned. Add 3 to 4 drops of *o*-phenanthroline indicator, and titrate the solution with 0.5N $FeSO_4$. As the endpoint is approached, the solution takes on a greenish cast and then changes to a dark green. At this point, add the ferrous sulfate heptahydrate drop by drop until the color changes sharply from blue to red (maroon color in reflected light against a white background). Make a blank determination in the same manner, but without soil, to standardize the $Cr_2O_7^{2-}$. Repeat the determination with less soil if $>75\%$ of the dichromate is reduced.

Calculate the results according to the following formula, using a correction factor $f = 1.30$ or a more suitable value found experimentally:

$$\text{Organic C, \%} = \frac{(\text{meq } K_2Cr_2O_7 - \text{meq } FeSO_4)(0.003)(100)}{\text{g water-free soil}} \times f \quad [13]$$

29-3.5.2.3 Comments. Ferrous ammonium sulfate is also a suitable titrant for excess $Cr_2O_7^{2-}$ in conjunction with the Walkley-Black method. The Smith and Weldon (1941) modification involving complete reduction of $Cr_2O_7^{2-}$ with Fe^{2+} , and subsequent back-titration of excess Fe^{2+} with MnO_2 , solution may also be used to estimate unreacted $Cr_2O_7^{2-}$. Other oxidation-reduction indicators that have provided satisfactory results include barium diphenylamine sulfonate and *N*-phenylanthranilic acid. The amounts of $Cr_2O_7^{2-}$ reduced to Cr^{3+} by reaction with soil organic matter may also be estimated colorimetrically.

29-3.5.3 MODIFIED MIERHUS PROCEDURE

29-3.5.3.1 Special Apparatus

1. Erlenmeyer flasks (125 ml) fitted with female standard-taper 24/40 ground-glass joints (Coring 5000 or Kimble 26510).
2. West condensers (30 cm) fitted with male standard-taper 24/40 ground-glass joints at the lower end (Coring 2800 or Kimble 18190).
3. Electric hot plate extraction unit (six plates per unit) fitted with individual rheostat controls (Labconco 60300, Precision 65500, Lab-Linc Multi-Unit Extraction Heater, or equivalent).

29-3.5.3.2 Reagents

1. Potassium dichromate solution ($K_2Cr_2O_7$), 0.5N: Dissolve 24.5125 g of $K_2Cr_2O_7$ (oven-dry) in 200 ml of deionized water, and dilute to 1 liter.
2. Sulfuric acid (H_2SO_4), concentrated, not less than 96%.
3. Ferrous ammonium sulfate hexahydrate solution [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$], 0.2N: Dissolve 78.390 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in 50 ml of conc H_2SO_4 , and dilute to 1 liter with deionized water (must be standardized daily because of slow oxidation).
4. Indicator solution: Dissolve 0.100 g of *N*-phenylanthranilic acid and 0.107 g of sodium carbonate (Na_2CO_3) in 100 ml of water.

29-3.5.3.3 Procedure. Weigh an amount of <100 -mesh soil (≤ 0.5 g) containing not greater than 8 mg of organic C into a 125-ml Erlenmeyer flask. Add exactly 10 ml of 0.5N $K_2Cr_2O_7$ solution and 15 ml of conc H_2SO_4 (H_2SO_4 may be added by buret). Attach the flask to the West condenser, and place on a preheated electric hot plate. Include a blank in each group of five soil samples to be heated and at least two unboiled blanks (unboiled blanks are unheated mixtures of 10 ml of 0.5N $K_2Cr_2O_7$ and 15 ml of conc H_2SO_4) for each day that analyses are performed. The normality of the $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ solution is determined by titrating the unboiled blank. Gently boil each sample for 30 min, and then insert an asbestos pad between the hot plate and bottom of the Erlenmeyer flask. Allow the flask to cool for about 15 min, and rinse the inside of the condenser with de-

WALKLEY-BLACK METHOD

Equipment:

1. 500-mL Erlenmeyer flasks.
2. 10-mL pipette.
3. 10- and 20-mL dispensers.
4. 50-mL burette.
5. Analytical balance.
6. Magnetic stirrer.
7. Incandescent lamp.

Reagents:

1. H_3PO_4 , 85%.
2. H_2SO_4 , concentrated (96%).
3. NaF, solid.
4. Standard 0.167M $\text{K}_2\text{Cr}_2\text{O}_7$: Dissolve 49.04 g of dried (105°C) $\text{K}_2\text{Cr}_2\text{O}_7$ in water and dilute to 1 L.
5. 0.5 M Fe^{2+} solution: Dissolve 196.1 g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4) \cdot 6\text{H}_2\text{O}$ in 800 mL of water containing 20 mL of concentrated H_2SO_4 and dilute to 1 L. The Fe^{2+} in this solution oxidizes slowly on exposure to air so it must be standardized against the dichromate daily.
6. Ferrous indicator: Slowly dissolve 3.71 g of o-phenanthroline and 1.74 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 250 mL of water.

Procedure:

1. Weigh out 0.10 to 2.00 g dried soil (ground to <60 mesh) and transfer to a 500-mL Erlenmeyer flask. The sample should contain 10 to 25 mg of organic C (17 to 43 mg organic matter). For a 1 g soil sample, this would be 1.2 to 4.3% organic matter. Use up to 2.0 g of sample for light colored soils and 0.1 g for organic soils.
2. Add 10 mL of 0.167 M $\text{K}_2\text{Cr}_2\text{O}_7$ by means of a pipette.
3. Add 20 mL of concentrated H_2SO_4 by means of dispenser and swirl gently to mix. Avoid excessive swirling that would result in organic particles adhering to the sides of the flask out of the solution.
4. Allow to stand 30 minutes. The flasks should be placed on an insulation pad during this time to avoid rapid heat loss.
5. Dilute the suspension with about 200 mL of water to provide a clearer suspension for viewing the endpoint.
6. Add 10 mL of 85% H_3PO_4 , using a suitable dispenser, and 0.2 g of NaF. The H_3PO_4 and NaF are added to complex Fe^{3+} which would interfere with the titration endpoint.
7. Add 10 drops of ferrous indicator. The indicator should be added just prior to titration to avoid deactivation by adsorption onto clay surfaces.

8. Titrate with 0.5 M Fe²⁺ to a burgundy endpoint. The color of the solution at the beginning is yellow-orange to dark green, depending on the amount of unreacted Cr₂O₇²⁻ remaining, which shifts to a turbid gray before the endpoint and then changes sharply to a wine red at the endpoint. Use of a magnetic stirrer with an incandescent light makes the endpoint easier to see in the turbid system (fluorescent lighting gives a different endpoint color). Alternatively use a Pt electrode to determine the endpoint after step 5 above. This will eliminate uncertainty in determining the endpoint by color change. If less than 5 mL of Fe²⁺ solution was required to backtitrate the excess Cr₂O₇²⁻ there was insufficient Cr₂O₇²⁻ present, and the analysis should be repeated either by using a smaller sample size or doubling the amount of K₂Cr₂O₇ and H₂SO₄.
9. Run a reagent blank using the above procedure without soil. The blank is used to standardize the Fe²⁺ solution daily.

10. Calculate %C and % organic matter:

a. % Easily Oxidizable Organic C

$$\%C = \frac{(B-S) \times M \text{ of Fe}^{2+} \times 12 \times 100}{\text{g of soil} \times 4000}$$

where:

B = mL of Fe²⁺ solution used to titrate blank
 S = mL of Fe²⁺ solution used to titrate sample
 12/4000 = milliequivalent weight of C in g.

To convert easily oxidizable organic C to total C, divide by 0.77 (or multiply by 1.30) or other experimentally determined correction factor. To convert total organic C to organic matter use the following equation:

b. $\% \text{ Organic Matter} = \frac{\% \text{ total C} \times 1.72}{0.58}$

ROUTINE COLORIMETRIC DETERMINATION OF SOIL ORGANIC MATTER

Equipment:

1. Standard 1 g scoop.
2. Glass marbles with a diameter slightly larger than the mouth of a 50 mL Erlenmeyer flask.
3. 50 mL Erlenmeyer flasks.
4. Digestion oven, capable of temperatures to 90°C, with air circulation fan and fume exhaust.
5. 10 and 25 mL pipettes or dispensers.
6. Standard organic matter samples.

Reagents:

1. Digestion solution:
 (0.5 M Na₂Cr₂O₇ • 2H₂O in 5 M H₂SO₄):
 Dissolve 140 g Na₂Cr₂O₇ • 2H₂O in 600 mL of distilled water. Slowly add 278 mL of concentrated H₂SO₄. Allow to cool and dilute to 1 L with deionized water.

Procedure:

1. Scoop 1 g of soil into a 50 mL Erlenmeyer flask. See Chapter 2 for details on proper scooping techniques.
2. Pipette 10 mL of dichromate-sulfuric acid digestion solution. Include a reagent blank without soil.

**APPENDIX XXXV. SOP for Data Verification, Validation and Loading to the ILRP SWAMP
Comparable Database. (Revision 2, 04/22/10)**

Standard Operating Procedure for Data Verification, Validation and Loading to the ILRP SWAMP Comparable Database

(Revision 2 April 22, 2010 AS)

1.0 Purpose

This standard operating procedure (SOP), based on Moss Landing Marine Laboratory's SWAMP Standard Operating Procedure for Data Loading and Verification of SWAMP database v1.1, describes the process used by the Michael L. Johnson, LLC Data Management Team (MLJ-LLC DMT) to verify, validate and load chemistry and bacteria data into the ILRP SWAMP comparable database. The data verification will be performed in accordance with the requirements of the ILRP QAPP (September 25, 2008).

2.0 Receipt of New Laboratory Data Files

1. Multiple laboratories send data for MLJ-LLC chemistry and bacteria analysis in SWAMP comparable Excel spreadsheets. These data files are delivered to the MLJ-LLC DMT who files the electronic data to be processed.

2.1 Filing and Storage of Laboratory Data Files

1. Data are stored on the "X" drive on the MLJ-LLC server within one of three main folders (New, Working, and Loaded). All new data are stored under the folder *Database\EDDs\New* in the appropriate event category folder. In addition, new data are also stored under the Lab folder in the appropriate coalition, event, agency, and "Original EDDs" category folders and renamed with a "_OR" after the file name. For example, an inorganics file from coalition Y, event W, and analyzed by laboratory X would be filed in *Database\EDDs\New\EventW* as well as *Lab\Lab EDD\CoalitionY\CoalitionY_EventW\Laboratory X\Original EDDs*.
2. While a file is being prepared for loading, it is moved from the specific *New* folder into the *Working* folder.
3. After all necessary corrections have been made and the data file is loaded, the file is moved into the *Database\EDDs\Loaded* folder beneath the appropriate analysis, agency and event category. A copy of the loaded file is also saved in the *Final EDDs* subfolder located on the common drive and renamed with "_Loaded" and the date after the file name (e.g. *_Loadedmmddyy*). For example, data from laboratory X and coalition Y for event W would be filed in *Lab\Lab EDD\CoalitionY\CoalitionY_EventW\LaboratoryX\Final EDDs*.

3.0 Loading Laboratory Data Files

Verification checklists exist for each lab's electronic data files, listing the specific formatting and data verification needed prior to uploading that lab's data. Checklists are updated regularly and are used to ensure quality control. Listed below are all manual data formatting and verification checks needed prior to loading any data file into the Data Loader Program.

3.1 Manual Verification of Unloaded Laboratory Data Files

1. Verify sample integrity
 - a. Verify all results, MDL and RL values with the hardcopy data report.
 - b. Verify result qualifier code (*ResultQualCode* field).
 - c. Verify dates (*Prep Date*, *DigestExtractDate*, and *AnalysisDate* fields) with the hardcopy data report.

- d. Verify the following fields have been entered correctly: *SampleCode*, *EventType*, *ProtocolCode*, *SampleDate*, *CollectionTime*, *SampleTypeCode*, *Replicate*, *CollectionDepth*, *UnitCollectionDepth*, *ProjectCode*, and *AgencyCode* with the appropriate Sample Details table (see Sample Details SOP).
 - e. Verify that the analytes listed in the appropriate Sample Details table have been analyzed for.
 - f. Verify that the holding times have been met as required by the method or the QAPP.
 - g. Verify the *QACode*.
 - h. Verify that batch requirements are met (as per QAPP).
2. Verify method blanks
- a. Verify the method blanks have met the frequency requirements as stated in the QAPP.
 - b. Verify the *SampleTypeCode* is "*LabBlank*".
 - c. Verify that the method blank is within control limits.
 - d. Verify the *QACode* (data outside of control limits must be flagged with the appropriate code).
3. Verify surrogates
- a. Verify surrogates were reported for all samples, blanks, and QC samples as required by the methods. Surrogates are reported as percents with an expected value of 100.
 - b. Verify percent recovery (PR) values were entered in the *Result* field and "%" was entered into the *UnitName* field.
 - c. Verify the surrogates were within the QC acceptance limits as stated in the QAPP.
 - d. Verify the *QACode* (data outside of control limits must be flagged with the appropriate code). If surrogate is flagged, all samples in the batch receive the QA flag as well.
4. Verify the laboratory QC samples (e.g. MS/MSDs, DUPs, LCSs, and CRMs).
- a. Verify the laboratory QC samples met the frequency requirements of the QAPP.
 - b. Verify the codes in the *SampleTypeCode* field against the "*SampleTypeCode LookUp*" table.
 - c. Verify a value was entered in the *ExpectedValue* field for spiked samples.
 - d. Verify the units in the *UnitName* field.
 - e. Verify the QC samples were within the QC acceptance limits as stated in the QAPP.
 - f. Verify the *QACode* (data outside of control limits must be flagged with the appropriate code).
 - g. Verify that all lab replicates of 2 have an RPD value and a corresponding lab replicate 1 result.
 - h. Verify that all LCS samples have a *MatrixName* of "blankwater."
5. Verify the field QC samples (e.g. travel blanks, equipment blanks, field blanks, field duplicates).
- a. Verify the field QC samples meet the frequency requirements of the QAPP.
 - b. Calculate the FieldDup relative percent difference (FD RPD) and verify the FieldDup results were within the expected range of the native sample as stated in the QAPP (RPD<25).
 - c. Verify the *QACode* is correct if the FieldDup RPD was greater than 25. Both FieldDup and environmental sample receive the flag.
 - d. Verify that the FieldBlanks are within control limits as stated in the QAPP (<RL or <1/5 environmental sample).
 - e. Verify the *QACode* is correct if the FieldBlanks were outside the control limits.
 - f. Verify that the *MatrixName* for FieldBlanks, EquipBlanks and TravelBlanks is "blankwater."
6. Verify the target analytes and target reporting limits (TRLs).
- a. Verify against the QAPP that the required target analytes were reported for each requested method for every sample, QC sample, and blank.

- b. Verify the TRLs in the *RL* field are consistent with the required TRLs in the QAPP.
 - c. Verify that the TRLs have been adjusted to reflect all sample dilutions, concentrations and splits.
 - d. Verify the method detection limit (MDL) results in the *MDL* field are at or below the RL values in the *RL* field.
 - e. Verify the units in the *UnitName* field were correct based on the matrix reported.
7. Verify formatting
- a. Verify that the two worksheets *Results* and *LabBatch* exist and are properly named.
 - b. Verify that the Results and LabBatch header rows are correctly formatted (See Chem Analysis Template).
 - c. Date formats should be dd/mmm/yyyy.
 - d. Time formats should be xx:xx.
 - e. Stations, analytes, methods and qualifiers match values in lookup lists
 - f. Results, MDL and RL values should not be calculated values and only contain numerical values.
 - g. LABQA records have the appropriate format for sample information.
8. Verify record completeness
- a. All required fields are complete in *Results* sheet for all records.
 - b. All required fields are complete in *LabBatch* sheet for all records.
9. Verify that all Lab Batch names are unique and are identified in the LabBatch sheet.
- a. Verify that the *LabSubmissionCode* reflects the batch (i.e. missing QC, no QC or minor deviations). Batches with any flags other require a batch comment.

3.2 Corrective Action/Resolution

When errors are found either in the data file which prevent it from being loaded into the database or the hardcopy report the following corrective actions will be performed:

1. The appropriate lab will be contacted regarding the issue(s) requiring resolution and sent a copy of the data file to use as a reference if needed.
2. If the issue requires a resubmission, per the direction of the MLJ-LLC DMT, a revised data file and/or hardcopy report will be requested from the laboratory.
3. All other minor issues will be revised by the DMT and the lab notified of these changes prior to loading the data.

3.3 Chemistry Loader Protocol

1. Once verification checks have been completed, the data file is manually loaded into the database following the Chemistry Loader Protocol (file name: Chemistry Loader Protocol_AS_MT111609).

Chemistry Loader Protocol

Name of excel file: _____ copy of database

Date: _____

Username: _____

#Results Sheet _____

#Batch Sheet _____

Link loader to current database: In the switchboard loader form type the UserName of the person loading data and add the suffix "-DL" for data loader (i.e. "mturner-DL"). Then click on the BROWSE DATABASE button under Step 2 on the form. Browse to the current database where data will be entered and click OPEN. Click RE-LINK on the switchboard form.

Import data: Click on the BROWSE DATA FILE button under Step 3 on the switchboard form. Browse to the excel file that contains the data to be loaded. Click OPEN. Click IMPORT DATA button. A message confirming all data currently in the two static tables will be overwritten, Click YES. Click OK.

- The Results and LabBatch data from the Excel file have now been imported into two static tables named:
 - 1tblBatch
 - 1tblResults
- Open each table to check that the data looks correct and that the number of records in each matches the original excel file counts.

	Before	After
#LabBatch	_____	_____
#LabCollection_Entry	_____	_____
#LabResult_Entry	_____	_____
#Location_Entry	_____	_____
#Sample_Entry	_____	_____

Unique queries:

- zBatch Unique
- zResults1 1Samples Unique
- zResults1 2Locations Unique
- zResults1 3Collections Unique
- zResults1 4Results Unique

These queries check the number of unique values in the file to load. Run queries and check that the data looks correct.

FileChecker Report: Check the imported data using the switchboard form. Click the RUN REPORT button under Step 4 of the form. A new window will open with the report findings. Review this information and make any necessary changes to the Excel file. If any changes are needed, save the Excel file and re-import the data. Re-run the report until all errors are either corrected or expected.

- Expect to see all LabBatches
- Expect all LABQA and NONPJ to have unmatched Locations, LabCollections, and Results

Load LabBatches:

- Run select query zBatch. Displays all batches in the static table 1tblBatch, check that the numbers are correct. Close this query.
 - zBatch records _____.
- Run select query zBatch WOMatch LabBatch. Displays all batches not currently in the database. Should equal number of batches in zBatch.
- Open append query zBatch WOMatch LabBatch Append in **design view – DO NOT RUN**. **See NOTE below. Click on the datasheet view to display unique batches that will be appended. The number of batches should match the number from zBatch WOMatch LabBatch. Click on the design view and run this query. A window will appear confirming the number of batches to append, click YES if it is the correct number.

#Batches appended_____

**NOTE: The query automatically adds a BatchVerificationCode of "Vac". Use this default ONLY if data has undergone all standard data checking and quality control procedures (usually this applies to all data). IF for some reason the data was not subject to all standard procedures, you MUST change the BatchVerificationCode to Not Recorded (NR). Go to design view, in the Field row of the BatchVerificationCode column change the text from [BatchVerificationCode: "Vac"] to [BatchVerificationCode: "NR"].

- Run query zBatch WOMatch LabBatch again. Query should be blank if all batches were appended properly.

Load Samples:

- Run select query zResults1 1Samples. Displays all samples in the static table 1tblResults, check that the numbers are correct. Close this query.
 - zResults1 1Samples Records: _____.
- Run query zResults1 1Samples WOMatch Sample Entry. Displays all samples not currently in the database. Most often LABQA and NONPJ samples will be the only ones needed to be appended. Close this query.
- Open append query zResults1 1Samples WOMatch Sample Entry Append in **design view – DO NOT RUN**. Click on the datasheet view to display number of unique samples that will be appended. The number should match the number from zResults1 1Samples WOMatch Sample Entry. Click on the design view and run this query. A window will appear confirming the number of samples to append, click YES if it is the correct number.

#Samples appended_____

- Run query zResults1 1Samples WOMatch Sample Entry again. Query should be blank if all samples were appended properly.

Load Locations:

- Run select query zResults1 2Locations. Displays all samples in the static table 1tblResults, check that the numbers are correct. Close this query.
 - zResults1 2Locations Records: _____.
- Run query zResults1 2Locations WOMatch Location Entry. Displays all locations not currently in the database. Most often LABQA and NONPJ locations will be the only ones needed to be appended, and number of results will be the same as the number of Samples previously appended. Close this query.
- Open append query zResults1 2Locations WOMatch Location Entry Append in design view. Click on the datasheet view to display number of unique locations that will be appended. The number should match the number from zResults1 2Locations WOMatch Locations Entry. Click on the design view and run this query. A window will appear confirming the number of locations to append, click YES if it is the correct number.

#Locations appended_____

- Run query zResults1_2Locations_WOMatch_Location_Entry again. Query should be blank if all locations were appended properly.

Load Collections:

- Run select query zResults1_3Collections. Displays all samples in the static table 1tblResults, check that the numbers are correct. Close this query.
 - zResults1_3Collections Records:_____.
- Run query zResults1_3Collections_WOMatch_Collection_Entry. Displays all collections not currently in the database. Most often LABQA and NONPJ collections will be the only ones needed to be appended. Close this query.
- Open append query zResults1_3Collections_WOMatch_Collection_Entry_Append in **design view – DO NOT RUN**. Click on the datasheet view to display number of unique collections that will be appended. The number should match the number from zResults1_3Collections_WOMatch_Collection_Entry. Click on the design view and run this query. A window will appear confirming the number of collections to append, click YES if it is the correct number.

#Collections appended_____

- Run query zResults1_3Collections_WOMatch_Collection_Entry again. Query should be blank if all collections were appended properly.

Load Collection Comments:

- Run select query zResults1_4CollectionsComments. Displays all collection comments in the static table 1tblResults. **If there are no collection comments to append, skip ahead to Load Results.**

Load Results:

- Open make table query zResults1_5Collections_MakeTable in **design view – DO NOT RUN**. This query creates a static table of unique collections in the database. Run this query. Click YES to the first window confirming that the data in the table tblSampleCollectionTemp will be deleted, and then click YES to the window confirming that the certain number rows will be added to the static table. Close the query.
 - zResults1_5Collections_MakeTable Records:_____.
- Run select query zResults1_5Results. Displays all results in the static table 1tblResults, check that the numbers are correct. Close this query.
 - zResults1_5Results Records:_____.
- Run query zResults1_5Results_WOMatch_Results. Displays all results not currently in the database. The number of results in query zResults1_5Results and this query **should** be identical. Close this query.
- Open append query zResults1_5Results_WOMatch_Results_Append in **design view – DO NOT RUN**. **See NOTE below. Click on the datasheet view to display the number of unique results that will be appended. The number should match the number from zResults1_5Results_WOMatch_Results. Click on the design view and run this query. A window will appear confirming the number of results to append, click YES if it is the correct number.

#Results appended_____

**NOTE: The query automatically adds a ComplianceCode of compliance (Com). Use this default ONLY if data has undergone all standard data checking and quality control procedures (usually this applies to all data). IF for some reason the data was not subject to all standard procedures, you MUST change the ComplianceCode to Not Recorded (NR). Go to design view, in the Field row of the ComplianceCode column change the text from [ComplianceCode: "Com"] to [ComplianceCode: "NR"].

- Run query zResults1_5Results_WOMatch_Results again. Query should be blank if all results were appended properly.

Check Loaded Data: Use *qry4SampleResults_Entry* to compare the records in the Excel file to those in the database. Filtering by the LastUpdateDate will allow you to see the number of records that were uploaded on any particular day. Check that the number of records in the excel file equal the number of records uploaded.

Record number of records uploaded in excel data tracker

APPENDIX XXXVI. SOP for Creating Sample Detail Excel Files (Revision 2, 05/04/10)

Standard Operating Procedure for Creating Sample Detail

Excel Files

(Revision 2 May 04, 2010 AS)

1.0 Purpose

This standard operating procedure (SOP) describes the process used by the Michael L. Johnson, LLC Data Management Team (MLJ-LLC DMT) to create excel files for laboratories that outline the details of the samples collected in the field. The sample detail files aid the laboratories in formatting their electronic deliverable data (EDDs) to be SWAMP comparable and are verified against the Chain of Custody (COC) forms.

2.0 Retrieval of Data from the Database

1. After all the desired field data has been entered into the Access database (see Field Data Entry SOP), open qrySampleDetailsExpanded in the most recent version of the Report_QryDatabase_121608_AS.
 - a. In the Design View set the "SampleDate" criteria to the desired sampling event date, and the "SamplePurposeCode" to FieldMeasure.
 - b. Switch back to Table View.
 - c. Copy and paste the table from Access into the SampleDetails_Template_2.5 "Database" tab, found in the *Sample Details* folder on the "X" drive on the MLJ-LLC server.
2. To obtain the data for the individual lab tabs open qrySampleDetails in the most recent version of the Report_QryDatabase_121608_AS.
 - a. In the Design View set the "SampleDate" criteria to the desired sampling event date.
 - b. Switch back to Table View.
 - c. Copy and paste the table from Access into the SampleDetails_Template_2.5 "APPL" tab.

3.0 Verify Field Data Entry

1. Copy and paste all the sampled site rows from the APPL worksheet into each of the individual lab worksheets.
 - a. Delete station names and replace with the SampleIDs found on the Chain of Custody forms (COCs).
2. For each lab, verify that the *StationCode* of the sites in the database match those listed on their respective COCs.
3. Verify the *EventCode* is "WQ" for all samples.
4. Verify the *ProtocolCode* is "MLJ_FieldSOP_031309", unless using a different protocol or technique (i.e. clean hands/dirty hands), for all samples.
5. Verify the location that each site was sampled by checking the *LocationCodes* against the locations listed on the field sheets.
6. Verify that the *CollectionTime* of the sites in the database match those listed on the COC, and that the time has been formatted as text.
7. Verify *CollectionMethodCode*.
 - a. The code should be "Water_Grab" for all water samples and "Sed_Grab" for sediment samples.
 - b. The code should be "Not Applicable" for all blank samples.

8. Verify *SampleTypeCode*.
 - a. All field duplicate samples are now listed as “Grab” with a *Replicate* of “2”.
 - b. MS are now “MS1”.
9. Verify that all *Replicates* are “1”, with the exception of field duplicate Grab samples which are “2”.
10. Verify *CollectionDepth*.
 - a. “0.1” for all water samples and “2” for sediment samples.
11. Verify *UnitCollectionDepth*.
 - a. “m” for all water samples and “cm” for sediment samples.
12. Verify that the correct project is listed in the *ProjectCode* column (“ESJWQC_08” for the East San Joaquin Water Quality Coalition, “SJCDWQC_08” for the San Joaquin County and Delta Water Quality Coalition, and “TMDL_SJR_OP” for the San Joaquin River TMDL sampling).
13. Verify that “MLJ-LLC” is listed as the *AgencyCode*.

4.0 Laboratory Analysis

1. Following *AgencyCode*, each laboratory worksheet lists the constituents or group of constituents analyzed for at their respective labs.
 - a. From the COC, mark with an “X” the constituents each lab is supposed to analyze for at each station. (Note: not every constituent or group of constituents is analyzed for at every station).

5.0 Sample Detail Submission/Data Tracking

1. When each lab's sample details worksheet has been completed, save the excel file in the SampleDetails folder under the “X” MLJ-LLC common drive in the appropriate coalition and group folder. Name the file “*CoalitionY_SampleDetails_GroupX_date*.” For example an ESJWQC, Irrigation1 sampling event that occurred on April 23, 2008 would have a sample details file named “ESJWQC_SampleDetails_Irrigation1_042308.”
2. After saving, submit the excel file by email to the appropriate laboratories.
3. The final three columns in the lab sample detail worksheets are filled out once the associated EDD has been received by the MLJ-LLC DMT. The dates the EDD is received, uploaded, and filed in pdf format should be noted in the *Received Data in SWAMP format*, and *Uploaded into Database* columns respectively.

**APPENDIX XXXVII. SOP for Toxicity Data Verification, Validation and Loading to the ILRP SWAMP
Comparable Database (Revision 2, 04/22/10)**

Standard Operating Procedure for Toxicity Data Verification, Validation and Loading to the ILRP SWAMP Comparable Database

(Revision 2 April 22, 2010 AS)

1.0 Purpose

This standard operating procedure (SOP), based on Moss Landing Marine Laboratory's SWAMP Standard Operating Procedure for Toxicity Data Verification of the SWAMP database v3.1, describes the process used by the Michael L. Johnson, LLC Data Management Team (MLJ-LLC DMT) to verify, validate and load toxicity data into the ILRP SWAMP comparable database. The data verification will be performed in accordance with the requirements of the most recent project specific QAPP.

2.0 Receipt of New Laboratory Data Files

1. Toxicity data are delivered electronically in SWAMP approved Excel spreadsheets to the MLJ-LLC DMT who file and process the data.

2.1 Filing and Storage of Laboratory Data Files

4. Data are stored on the "X" drive on the MLJ-LLC server within one of three main folders (New, Working, and Loaded). All new data are stored under the folder *Database\EDDs\New* in the appropriate event category folder. In addition, new data are also stored under the Lab folder in the appropriate coalition, event, agency, and "Original EDDs" category folders and renamed with a "_OR" after the file name. For example, a toxicity file from coalition Y, event W, and analyzed by laboratory X would be filed in *Database\EDDs\New\EventW* as well as *Lab\Lab EDD\CoalitionY\CoalitionY_EventW\Laboratory X\Original EDDs*.
5. While a file is being prepared for loading, it is moved from the specific *New* folder into the *Working* folder.
6. After all necessary corrections have been made and the data file is loaded, the file is moved into the *Database\EDDs\Loaded* folder beneath the appropriate analysis, agency and event category. A copy of the loaded file is also saved in the *Final EDDs* subfolder located on the common drive and renamed with "_Loaded" and the date after the file name (e.g. *_Loadedmmddyy*). For example, data from laboratory X and coalition Y for event W would be filed in *La\Lab EDD\CoalitionY\CoalitionY_EventW\LaboratoryX\Final EDDs*.

3.0 Verifying and Loading Laboratory Data Files

Verification checklists exist for each labs electronic data files, listing the specific formatting and data verification needed prior to uploading that labs data. Checklists are updated regularly and are used to ensure quality control. Listed below are manual data formatting and verification checks needed prior to loading any toxicity data file.

3.1 Manual Verification of Unloaded Laboratory Data Files

1. Verify sample integrity
 - a. Verify *Mean*, *PctControl*, and *SigEffect* values in the *Summary* sheet with the hardcopy report.
 - b. Verify the following fields have been entered correctly: *StationCode*, *EventType*, *ProtocolCode*, *LocationCode*, *SampleDate*, *CollectionTime*, *CollectionMethodCode*,

- SampleTypeCode, Replicate, CollectionDepth, UnitCollectionDepth, ProjectCode* and *AgencyCode* with the appropriate Sample Details table (see Sample Details SOP).
- c. Verify that the correct toxicity species have been analyzed for at each sample site based on the Sample Detail sheet.
 - d. Verify that the holding times have been met as required by the method or the QAPP.
 - e. Verify the *TestQACode*.
 - f. Verify that the *SummaryComments* include an explanation for the following if they occur: Resample, or dilution.
 - g. Verify that the *TIENarrative* includes an explanation for a TIE if one is needed.
 - h. Verify that each batch has one control (CNEG) sample.
2. Verify toxicity and water quality measurement results
 - a. Verify values in the *Results* sheet with the hardcopy report.
 - b. Verify the correct number of replicates is reported as required by the QAPP.
 - c. Verify that the following fields have been entered correctly: *StationCode, EventType, ProtocolCode, LocationCode, SampleDate, CollectionTime, CollectionMethodCode, SampleTypeCode, Replicate, CollectionDepth, UnitCollectionDepth, ProjectCode* and *AgencyCode*.
 - d. Verify the correct water quality measurements are reported as required by the QAPP.
 - e. Verify that the water quality measurements meet the QC limits required by the QAPP.
 - f. Verify the values for *TimePoint*.
 - g. Verify that the evaluation thresholds meet QAPP requirements.
 - h. Verify the units in the *UnitAnalyte* field are correct based on the matrix reported.
 3. Verify laboratory QC samples (i.e. Control samples)
 - a. Verify that the laboratory QC samples meet the frequency requirements of the QAPP.
 - b. Verify the codes in the *SampleTypeCode* field against the *SampleTypeCode LookUp* table.
 - c. Verify the *Mean, PctControl, and StdDev* fields based on the hardcopy report.
 - d. Verify the units in the *UnitAnalyte* field.
 - e. Verify that the QC samples are within the QC acceptance limits as stated in the QAPP.
 - f. Verify the *TestQACode* which should reflect any QC limit exceedances.
 4. Verify field QC samples (i.e. Field duplicates)
 - a. Verify that the field QC samples meet the frequency requirements of the QAPP.
 - b. Verify the codes in the *SampleTypeCode* field against the *SampleTypeCode LookUp* table.
 - c. Calculate the FieldDup relative percent difference (RPD) and verify the FieldDup results are within the expected range of the native sample as stated in the QAPP (RPD<25).
 - d. Verify that the *TestQACode* is correct if the FieldDup RPD was greater than 25. Both FieldDup and environmental sample receive the flag.
 5. Verify formatting
 - a. Verify that the three worksheets *Results, Summary* and *ToxBatch* exist and are properly named.
 - b. Verify that the *Results, Summary* and *ToxBatch* header rows are correctly formatted (See Toxicity Template).
 - c. Date formats should be dd/mmm/yyyy.
 - d. Time formats should be xx:xx.
 - e. Station IDs, sample types, matrix, methods, species name, and qualifiers should match values in lookup lists.
 - f. *Result* and *PctControl* values should not be calculated values.
 - g. LABQA records have the appropriate format for sample and toxicity summary information.

6. Verify record completeness
 - a. All required fields are complete in *Results* sheet for all records.
 - b. All required fields are complete in *Summary* sheet for all records.
 - c. All required fields are complete in *ToxBatch* sheet for all records.

7. Verify that all Tox Batches are unique and are identified in the *ToxBatch* sheet.
 - a. Verify that if *TestQACode* in the *Summary* sheet is anything other than "None", the batch has a batch comment to explain the minor deviation (MD) and the *LabSubmissionCode* is "A,MD."
 - b. Verify that all *ToxBatchComments* which have no comments are labeled as "NR."
 - c. Verify that all *ToxBatchComments* are less than 250 characters.

3.2 Corrective Action/Resolution

When errors are found either in the data file which prevent it from being loaded into the database or the hardcopy report the following corrective actions will be performed:

4. The appropriate lab will be contacted regarding the issue(s) requiring resolution and sent a copy of the data file to use as a reference if needed.

5. If the issue requires a resubmission, per the direction of the MLJ-LLC DMT, a revised data file and/or hardcopy report will be requested from the laboratory.

6. All other minor issues will be revised by the DMT and the lab notified of these changes prior to loading the data.

3.3 Toxicity Loader Protocol

2. Once verification checks have been completed, the data file is manually loaded into the database following the Tox Loader Protocol (file name: Toxicity Loader Protocol_041910SH).

Tox Loader Protocol

Name of excel file: _____
of database

□ copy

Date: _____

Username: _____

#Summary Sheet _____

#Results Sheet _____

#ToxBatch Sheet _____

Link loader to current database: In the switchboard loader form type the UserName of the person loading data and add the suffix "-DL" for data loader (i.e. "mtturner-DL"). Then click on the BROWSE DATABASE button under Step 2 on the form. Browse to the current database where data will be entered and click OPEN. Click RE-LINK on the switchboard form.

Import data: Click on the BROWSE DATA FILE button under Step 3 on the switchboard form. Browse to the excel file that contains the data to be loaded. Click OPEN. Click IMPORT DATA button. A message confirming all data currently in the three static tables will be overwritten, Click YES. Click OK.

- The Summary, Results and ToxBatch data from the Excel file have now been imported into three static tables named:
 - 1tblToxBatch
 - 1tblToxResults
 - 1tblToxSummary
- Open each table to check that the data looks correct and that the number of records in each matches the original excel file counts.
- Go through the 1tblToxResults and **delete all -88 Result values** (if there are any), cells should be left blank.
- Import Summary data from Excel file into 1tblSummary_import. Go to design view and **change the CollectionTime data type from TEXT to DATE/TIME & the PctControl data type from NUMBER to TEXT.**

	Before	After
#LabCollection_Entry	_____	/ _____
#Location_Entry	_____	/ _____
#Sample_Entry	_____	/ _____
#ToxBatch	_____	/ _____
#ToxPoint_Entry	_____	/ _____
#ToxPointSummary_Entry	_____	/ _____
#ToxResult_Entry	_____	/ _____
#ToxTest_Entry	_____	/ _____

Unique queries, Part I of II:

- o zTox00Batch Unique

This query checks the number of unique values in the file to load. Run the unique batch query and check that the data looks correct. (The other unique queries will be run after imported data constituents are linked with rowIDs.)

FileChecker Report: Check the imported data using the switchboard form. Click the RUN REPORT button under Step 4 of the form. A new window will open with the report findings. Review this information and make any necessary changes to the Excel file. If any changes are needed, save the Excel file and re-import the data. Re-run the report until all errors are either corrected or expected.

- o Expect to see all LabBatches
- o Expect all LABQA to have unmatched Samples, Locations, and Collections.

Load ToxBatches:

- o Run select query zTox00Batch. Displays all batches in the static table 1tblToxBatch, check that the numbers are correct. Close this query.
 - o zTox00Batch records _____.
- o Run select query zTox00Batch WOMatch ToxBatch. Displays all batches not currently in the database. Should equal number of batches in zTox01Batch.
- o Open append query zTox00Batch WOMatch ToxBatch Append in **design view – DO NOT RUN**. Click on the datasheet view to display unique batches that will be appended. The number of batches should match the number from zTox00Batch WOMatch ToxBatch. Click on the design view and run this query. A window will appear confirming the number of batches to append, click YES if it is the correct number.

#Batches appended _____

- o Run query zTox00Batch WOMatch ToxBatch again. Query should be blank if all batches were appended properly.

Link Imported Data Constituents with RowIDs:

- o Open the make table query zTox00Result xWalk MethodDetail MT in **design view – DO NOT RUN**. This query creates a static table that helps to link the constituents to the needed rowIDs for the data you are trying to import. Run this query. Click YES to the first window confirming that the data in table tblXLResultsRecordID MethodDetailRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResults). Close the query.
 - o zTox00Result xWalk MethodDetail MT Records: _____.
- o Open the make table query zTox00Result xWalk ConstituentRowID MT in **design view – DO NOT RUN**. This query creates a static table that helps to link the constituents to the needed rowIDs for the data you are trying to import. Run this query. Click YES to the first window confirming that the data in table tblXLResultRecordID ConstituentRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResults). Close the query.
 - o zTox00Result xWalk ConstituentRowID MT Records: _____.
- o Open the make table query zTox00Summary xWalk ConstituentRowID MT in **design view – DO NOT RUN**. This query creates a static table that helps to link the constituents to the needed rowIDs for the data you are trying to import. Run this query. Click YES to the first window confirming that the data in table tblXLSummaryRecordID ConstituentRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxSummary). Close the query.
 - o zTox00Summary xWalk ConstituentRowID MT Records: _____.
- o Open the make table query zTox00Summary xWalk MethodDetail MT in **design view – DO NOT RUN**. This query creates a static table that helps to link the constituents to the needed rowIDs for the data you are trying to import. Run this query. Click YES to the first window confirming that

the data in table tblXLSummaryRecordID MethodDetailRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxSummary). Close the query.

- o zTox00Summary xWalk MethodDetail MT Records: _____.

Unique queries, Part II of II:

- o zTox02Results 1Samples Unique
- o zTox02Results 2Locations Unique
- o zTox02Results 3Collections Unique
- o zTox02Results 4ToxTest Unique
- o zTox02Results 5ToxPoint Unique
- o zTox02Results 6ToxResult Unique
- o zTox02Summary 7ToxSummary Unique

These queries check the number of unique values in the file to load. Run queries and check that the data looks correct.

Load Samples:

- o Run select query zTox03Results Samples. Displays all samples in the static table 1tblToxResults, check that the numbers are correct. Close this query.
 - o zTox03Results Samples Records: _____.
- o Run query zTox03Results Samples WOMatch Sample Entry. Displays all samples not currently in the database. Most often LABQA samples will be the only ones needed to be appended. Close this query.
- o Open append query zTox03Results Samples WOMatch Sample Entry Append in **design view – DO NOT RUN**. Click on the datasheet view to display number of unique samples that will be appended. The number should match the number from zTox03Results Samples WOMatch Sample Entry. Click on the design view and run this query. A window will appear confirming the number of samples to append, click YES if it is the correct number.

#Samples appended _____

- o Run query zTox03Results Samples WOMatch Sample Entry again. Query should be blank if all samples were appended properly.
- o Open the make table query zTox03Results Samples xWalk SampleRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the SampleRowID from the database to the data in the 1tblToxResults table and makes comparison of data in the database quicker. Run this query. Click YES to the first window confirming that the data in table tblXLResultsRecordID SampleRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResults). Close the query.
 - o zTox03Results Samples xWalk SampleRowID MT Records: _____.

Load Locations:

- o Run select query zTox04Results Locations. Displays all samples in the static table 1tblToxResults, check that the numbers are correct. Close this query.
 - o zTox04Results Locations Records: _____.
- o Run query zTox04Results Locations WOMatch Location Entry. Displays all locations not currently in the database. Most often LABQA locations will be the only ones needed to be appended, and number of results will be the same as the number of Samples previously appended. Close this query.
- o Open append query zTox04Results Locations WOMatch Location Entry Append in **design view-DO NOT RUN**. Click on the datasheet view to display number of unique locations that will be appended. The number should match the number from zTox04Results Locations WOMatch Locations Entry. Click on the design view and run

this query. A window will appear confirming the number of locations to append, click YES if it is the correct number.

#Locations appended

- Run query zTox04Results Locations WOMatch Location Entry again. Query should be blank if all locations were appended properly.
- Open the make table query zTox04Results Locations xWalk LocationRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the LocationRowID from the database to the data in the 1tblToxResults table and makes comparison of data in the database quicker. Run this query. Click YES to the first window confirming that the data in table tblXLResultsRecordID LocationRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResults). Close the query.
 - zTox04Results Locations xWalk LocationRowID MT Records: _____.

Load Collections:

- Run select query zTox05Results Collections. Displays all samples in the static table 1tblToxResults, check that the numbers are correct. Close this query.
 - zTox05Results Collections Records: _____.
- Run query zTox05Results Collections WOMatch Collection Entry. Displays all collections not currently in the database. Most often LABQA collections will be the only ones needed to be appended. Close this query.
- Open append query zTox05Results Collections WOMatch Collection Entry Append in **design view – DO NOT RUN**. Click on the datasheet view to display number of unique collections that will be appended. The number should match the number from zTox05Results Collections WOMatch Collection Entry. Click on the design view and run this query. A window will appear confirming the number of collections to append, click YES if it is the correct number.

#Collections appended

- Run query zTox05Result Collections WOMatch Collection Entry again. Query should be blank if all collections were appended properly.
- Open the make table query zTox05Results Collections xWalk LabCollectionRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the LabCollectionRowID from the database to the data in the 1tblToxResults table and makes comparison of data in the database quicker. Run this query. Click YES to the first window confirming that the data in table tblXLResultsRecordID LabCollectionRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResult). Close the query.
 - zTox05Results Collections xWalk LabCollectionRowID MT Records: - _____.

Load Collection Comments:

- Run select query zTox06Results CollectionsComments. Displays all collection comments in the static table 1tblToxResults. **If there are no collection comments to append, skip ahead to Load ToxTests.**

Load ToxTests:

- Run select query zTox07Results ToxTest. Displays all results in the static table 1tblToxResults, check that the numbers are correct. Close this query.
 - zTox07Results ToxTest Records: _____.
- Run query zTox07Results ToxTest WOMatch ToxTest Entry. Displays all results not currently in the database. The number of results in query 1tblToxSummary and this query **should** be identical. Close this query.

- Open append query zTox07Results ToxTest WOMatch ToxTest Entry Append in **design view – DO NOT RUN**. Click on the datasheet view to display the number of unique results that will be appended. The number should match the number from zTox07Summary ToxTest WOMatch ToxTest. Click on the design view and run this query. A window will appear confirming the number of results to append, click YES if it is the correct number.

#ToxTests appended

- Run query zTox07Results ToxTest WOMatch ToxTest Entry again. Query should be blank if all results were appended properly.
- Open the make table query zTox07Results ToxTest xWalk ToxTestRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the ToxTestRowID from the database to the data in the 1tblToxResults table and makes comparison of data in the database quicker. Run this query. Click YES to the first window confirming that the data in table tblXLResultsRecordID ToxTestRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResults). Close query.
 - zTox07Results ToxTest xWalk ToxTestRowID MT Records: _____.
- Open the make table query zTox07Summary ToxTest xWalk ToxTestRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the ToxTestRowID from the database to the data in the 1tblToxSummary table and makes comparison of data in the database quicker. Run this query. Click YES to the first window confirming that the data in table tblXLSummaryRecordID ToxTestRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxSummary). Close the query.
 - zTox07Summary ToxTest xWalk ToxTestRowID MT Records: _____.
- Open the make table query zTox07Summary ToxTest QACode Update in **design view – DO NOT RUN**. This query creates a static table that links the QACodes from the database to the data in the 1tblToxSummary table and makes comparison of data in the database quicker. Run this query. Click YES to the window confirming that the certain number of rows will be updated in the static table (The number should match the number from 1tblToxSummary). Close the query.
 - zTox07Summary ToxTest QACode Update Records: _____.

Load ToxPoints:

- Run select query zTox08Results ToxPoint. Displays all results in the static table 1tblToxResults, check that the numbers are correct. Close this query.
 - zTox08Results ToxPoint Records: _____.
- Run query zTox08Results ToxPoint WOMatch ToxPoint. Displays all results not currently in the database. Note: "Survival" and "Total Cell Count" tox points will only be counted once for each collection. Close this query.
- Open append query zTox08Results ToxPoint WOMatch ToxPoint Append in **design view – DO NOT RUN**. Click on the datasheet view to display the number of unique results that will be appended. Click on the design view and run this query. A window will appear confirming the number of results to append, click YES if it is the correct number.

#ToxPoints appended

- Run query zTox08Results ToxPoint WOMatch ToxPoint again. Query should be blank if all results were appended properly.
- Open the make table query zTox08Results ToxPoint xWalk ToxPointRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the ToxPointRowID from the database to the data in the 1tblToxResults table and makes comparison of data in the database quicker. Run this query. Click YES to the window

confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxResults). Close the query.

- zTox08Results ToxPoint xWalk ToxPointRowID MT Records: _____.
- Open the make table query zTox08Summary ToxPoint xWalk ToxPointRowID MT in **design view – DO NOT RUN**. This query creates a static table that links the ToxPointRowID from the database to the data in the 1tblToxSummary table and makes comparison of data in the database quicker. Run this query. Click YES to the first window confirming that the data in table tblXLSummaryRecordID ToxPointRowID will be deleted, and then click YES to the window confirming that the certain number of rows will be added to the static table (the number should match the number from 1tblToxSummary). Close the query.
 - zTox08Summary ToxPoint xWalk ToxPointRowID MT Records: _____.

Load ToxResults:

- Run select query zTox09Results ToxResult. Displays all results in the static table 1tblToxResults, check that the numbers are correct. Close this query.
 - zTox09Results ToxResult Records: _____.
- Run query zTox09Results ToxResult WOMatch ToxResult Entry. Displays all results not currently in the database. The number of results in query zTox09Results ToxResult and this query **should** be identical. Close this query.
- Open append zTox09Results ToxResult WOMatch ToxResult Entry Append in **design view – DO NOT RUN**. **See NOTE below. Click on the datasheet view to display the number of unique results that will be appended. The number should match the number from zTox09Results ToxResult WOMatch ToxResult Entry. Click on the design view and run this query. A window will appear confirming the number of results to append, click YES if it is the correct number.

#ToxResults appended _____

NOTE: The query automatically adds a ComplianceCode of compliance (Com). Use this default ONLY if data has undergone all standard data checking and quality control procedures (usually this applies to all data). IF for some reason the data was not subject to all standard procedures, you MUST change the ComplianceCode to Not Recorded (NR). Go to design view, in the Field row of the ComplianceCode column change the text from [ComplianceCode: "Com"] to [ComplianceCode: "NR"].

- Run query zTox09Results ToxResult WOMatch ToxResult Entry again.

Load ToxPointSummary:

- Run select query zTox10Summary ToxSummary. Displays all results in the static table 1tblToxSummary, check that the numbers are correct. Close this query.
 - zTox10Summary ToxSummary Records: _____.
- Run query zTox10Summary ToxSummary WOMatch ToxPointSum Entry. Displays all results not currently in the database. The number of results in query zTox10Summary ToxSummary and this query **should** be identical. Close this query.
- Open append query zTox10Summary ToxSummary WOMatch ToxPointSum Entry Append in **design view – DO NOT RUN**. Click on the datasheet view to display the number of unique results that will be appended. The number should match the number from zTox10Summary ToxSummary WOMatch ToxPointSum Entry. Click on the design view and run this query. A window will appear confirming number of results to append, click YES if it is correct number.

#ToxPointSummary appended _____

- Run query zTox10Summary ToxSummary WOMatch ToxPointSum Entry again. Query should be blank if all results were appended properly.

Record number of records uploaded in excel data tracker