



# STANDARD OPERATING PROCEDURES

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## DETERMINATION OF PERCENT MOISTURE, pH, MELTING POINT AND CLOUD POINT AT THE STANDARD CHLORINE-METACHEM SITE

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### 1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the methods to be used for the on-site determination of percent (%) moisture, pH, melting point and cloud point in pure product and other matrices. On-site field laboratories provide quick turnaround on critical data needed for field decisions concerning site characterization, treatability, separation, and remediation/removal activities.

### 2.0 METHOD SUMMARY

#### 2.1 Percent Moisture by Karl Fischer

In the coulometric Karl Fischer (KF) titration, the end point is determined by electrochemical means. For a liquid sample, the actual sample weight is determined by weighing the syringe with sample and after injecting sample. For a solid sample, a weighed sample is extracted or dissolved into methylene chloride and mixed several times by inverting the sample. Prior to the first sample injection, the titration vessel is preconditioned. A 1-milliliter (mL) aliquot of weighed sample is then injected into the coulometer. After the titration, the result for water (% moisture) in the sample is displayed in parts per million (ppm).

#### 2.2 pH

A weighed sample is placed into a glass vial and mixed with deionized (DI) water. The glass vial is inverted several times to facilitate mixing. The pH is determined using a calibrated pH meter.

#### 2.3 Melting Point

For solid samples, a portion of the sample is placed into a clear glass tube. A thermometer, calibrated against a National Institute of Standards and Technology (NIST)-certified thermometer, is inserted into the tube. For liquid samples, a portion of sample is transferred to a clear glass tube, a calibrated thermometer is inserted into the tube, and the tube is placed into a cooling system until frozen.

The glass tube is then heated until the sample starts to melt. Melting point is defined as the point at which the sample first begins to melt.

#### 2.4 Cloud Point

A thermometer, calibrated against a NIST-certified thermometer, is inserted into a container containing the sample. The sample is heated until the sample is completely melted. The sample is removed from the heating source, and cooled until crystals appear in the sample. Cloud point is defined as the point at which crystals begin to form.

### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples are typically analyzed immediately upon receipt. All unused portions must be maintained in appropriate containers until disposal.



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### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

- Samples with multiple phases may pose difficulties when trying to obtain a representative sample for testing. The sample may not fully dissolve in methylene chloride.
- Temperature fluctuations will cause pH measurement errors.
- Coating on the surface of the pH electrode or the KF coulometer electrodes may necessitate the need for additional cleaning and maintenance.
- Glassware used for these analyses must be scrupulously cleaned with no residual water present.

### 5.0 EQUIPMENT/APPARATUS

- Coulometric titration system, capable of determining water (% moisture) in product samples, equipped with titration vessel setup (Metrohm 756 Karl Fischer Coulometer or equivalent).
- Bottles, glass, 10-mL and 40-mL.
- Balance, capable of weighing 0.1 gram (g) of sample (Ohaus, Inc. or equivalent).
- Thermometers, capable of reading temperatures between -20 and 200 degrees Celsius ( $^{\circ}\text{C}$ ), calibrated against a NIST-certified thermometer (VWR, Inc. or equivalent).
- Thermometer, NIST-certified, for calibrating working thermometers.
- Syringes, standard microliter ( $\mu\text{L}$ ) volume with cemented needles: 10, 1000, and 2500  $\mu\text{L}$  (Hamilton, Inc. or equivalent).
- Refrigerators, explosion-proof for sample and standard storage (Lab-Line or equivalent).
- Hot plate, capable of heating samples up to  $150^{\circ}\text{C}$  (Thermolyne Corp. or equivalent).
- pH Meter, capable of reading pH between 1 to 14 (Orion or equivalent).
- Test tubes, glass, for melting point and cloud point determination.
- Beaker, glass, 500-mL and 1000-mL, for use as a water bath.

### 6.0 REAGENTS

- Deionized water, American Society for Testing and Materials (ASTM) Type II, for pH extraction.
- Dichloromethane (DCM), pesticide grade, for percent moisture extraction (Burdick & Jackson or equivalent).



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- Hydranal water standard, 1000 ppm, commercially available, for KF determination (Brinkman or equivalent).
- Hydranal titration solution, commercially available, for KF determination (Metrohm or equivalent).
- pH 7.0 buffer, commercially available.

### 7.0 PROCEDURES

#### 7.1 Sample Preparation

The preparation procedures outlined below are those typically used by the on-site laboratory. The sample preparation may be modified depending on the type of matrix.

##### 7.1.1 Percent Moisture

For solid samples, dissolve approximately 0.5 to 1.0 g of sample into one mL of methylene chloride. Invert the mixture several times. For liquid samples, draw approximately 1.0 mL of sample into a pre-weighed 1000 uL syringe and record the weight. Refer to Section 7.2 for analysis.

##### 7.1.2 pH

Place approximately five g of sample into a 40-mL glass vial. Add 15 to 20 mL of DI water to the sample and shake for approximately 1 minute. Let the sample settle for 1 to 4 minutes.

#### 7.2 Percent Moisture Analysis

1. Perform pre-conditioning of the coulometer after instrument setup to dry the titration vessel. This step is only required thereafter if the unit is turned off or the drift will not stabilize. When the titration vessel is dry, the "COND" light emitting diode (LED) shows a steady light.
2. Monitor the drift to ensure that it is stable.
3. Before any sample determination, check the KF Coulometer using a 1000 ppm hydranal water standard. Press "START", inject 1 mL of the 1000 ppm hydranal water standard, and record the reading. The reading must be within  $\pm 30\%$  (700 to 1300 ppm).
4. For solid samples, press "START" and run a DCM solvent check prior to any analyses.
5. Press "START" and inject the sample prepared in Section 7.1.1.
6. Enter the sample size and press "ENTER". NOTE: As the titration progresses, a curve of water (H<sub>2</sub>O) against time will be displayed on the unit.



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7. Record the result in the laboratory notebook.

#### 7.3 pH Analysis

1. For each day of pH analysis, calibrate the pH meter with pH 7.0 buffer solution prior to sample analysis.
2. Using a fresh portion of buffer, insert the electrode into the buffer and gently stir the buffer solution. Once the reading has stabilized, record the reading. The buffer must read between 6.95 and 7.05 pH units.

Note: Check the pH meter periodically using the pH 7.0 buffer, especially if there are temperature fluctuations in the laboratory that will affect pH readings.

3. Check the pH of the DI water used for extraction as a background check and record the reading.
4. Using the sample prepared in section 7.1.2, insert the pH probe into the liquid for 1 to 2 minutes.
5. Once the pH reading is stabilized, record the reading in a laboratory log book.

#### 7.4 Melting Point Determination

1. For a solid sample, place a portion of the sample into a glass tube with a calibrated thermometer.
2. For a liquid sample, place a portion of the sample into a glass tube with a calibrated thermometer. Place the tube containing the sample into a cooling system capable of freezing or solidifying the sample.
3. Heat the sample using a water bath and heat slowly until the sample starts to melt.
4. Record the temperature at which the sample first begins to melt in a laboratory log book.

#### 7.5 Cloud Point Determination

1. Heat the sample in the sample container until the entire sample is melted.
2. Place a calibrated thermometer into the liquid/melted sample.
3. Stir the sample and allow the sample to slowly cool.
4. Record the temperature at which crystals appear in the sample container in a laboratory notebook.



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### 8.0 CALCULATIONS

The percent moisture is calculated by the KF Coulometer unit. Melting point and cloud point are directly read from the calibrated thermometer, and pH is read directly from the pH meter display.

### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following quality assurance/quality control procedures apply:

5. An acceptable 1000-ppm hydranal standard reference material (SRM) must be run for each 24-hour shift during which percent moistures are determined. The reading must be within  $\pm 30\%$  of the theoretical concentration.
6. The blank solvent check must be run before any percent moisture determinations are made on solid samples.
7. The reading for the pH 7.0 buffer must be within 6.95 to 7.05 pH units and must be run for each 24 hour shift during which pH are determined at a minimum.

### 10.0 DATA VALIDATION

The data generated will be reviewed on site by SERAS field chemists according to the Quality Assurance/Quality Control considerations listed in Section 9.0.

### 11.0 HEALTH AND SAFETY

Field laboratory instrumentation and analytical methods must meet all relevant United States Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT), SERAS, and Occupational Safety and Health Administration (OSHA) regulations to ensure the safety of personnel working in the laboratory. All applicable U.S. EPA and U.S. Department of Transportation (DOT) regulations regarding handling, accumulation, storage and removal of hazardous wastes must be met. More specifically, refer to SERAS SOP #1501, *Hazardous Waste Management* and SERAS SOP #3013, *SERAS Laboratory Safety Program*.

### 12.0 REFERENCES

Metrohm, Ltd. 1998. *KF Coulometer Instructions for Use*, 8.756.1003.

### 13.0 APPENDICES

This section is not applicable to this SOP.