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ROCKY FLATS PLANT
EMD OPERATING
PROCEDURES MANUAL

Manual No.: 5-21000-OPS-GW
Procedure No.: Table of Contents, Rev 2
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Organization: Environmental Management

THIS IS ONE VOLUME OF A SIX VOLUME SET WHICH INCLUDES:

VOLUME I: FIELD OPERATIONS (FO)
VOLUME II: GROUNDWATER (GW)
VOLUME III: GEOTECHNICAL (GT)
VOLUME IV: SURFACE WATER (SW)
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By [Signature]
Date March 19, 1992

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This is a RED Stamp **FIELD MEASUREMENT OF GROUNDWATER FIELD PARAMETERS**

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EMD MANUAL OPERATION SOP

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TITLE:
FIELD MEASUREMENT OF
GROUNDWATER FIELD PARAMETERS

Approved By:

(Name of Approver)

(Date)

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2.0 PURPOSE AND SCOPE

Chemical and physical data collection on location during the sampling of groundwater will supplement analytical data. Data collected in the field will be referred to as field parameters. This standard operating procedure (SOP) describes measurement procedures that will be used for the collection of the following field parameters: pH, specific conductance, dissolved oxygen, temperature, nitrate and N, and turbidity.

The collection frequency of these field parameters specific to individual projects will be detailed in individual field sampling plans. However, some criteria for the collection of field parameters will be consistent for all programs. Those criteria will be described in this SOP.

3.0 RESPONSIBILITIES AND QUALIFICATIONS

All personnel performing these procedures are required to have 40-hour OSHA classroom training which meets Department of Labor Regulation 29 CFR 1910.120(e)(3)(i). In addition, all personnel are required to have a complete understanding of the procedures described within this SOP and receive specific training regarding these procedures if necessary.

4.0 REFERENCES

4.1 SOURCE REFERENCES

The following is a list of references reviewed prior to the writing of this procedure:

A Compendium of Superfund Field Operations Methods. EPA/540/P-87/001.

Data Quality Objective for Remedial Activities Development Process. EPA/540/G-87/003. 1987.

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HACH DR/2000 Spectrophotometer Handbook. HACH Company. Loveland, CO 1988.

HACH Water Analysis Handbook. HACH Company. Loveland, CO 1989.

RCRA Groundwater Monitoring Technical Enforcement Guidance Document.
EPA, OSWER-9950.1. September 1986.

Standard Methods for the Examination of Water and Wastewater. 16th Edition. Method 212. 1985.

The Environmental Survey Manual. DOE/EH-0053. Appendix E, "Field Protocols and Guidance."
1987.

4.2 INTERNAL REFERENCES

Related SOPs cross-referenced by this SOP are as follows:

- SOP FO.3, General Equipment Decontamination
- SOP FO.7, Handling of Decontamination Water and Wash Water
- SOP GW.6, Groundwater Sampling
- SOP FO.13, Containerizing, Preserving, Handling, and Shipping of Soil and Water Samples

5.0 FIELD MEASUREMENT PROCEDURES

Several of the parameters required to be measured are physically or chemically unstable and must be tested either in the borehole using a probe (in situ) or immediately after collection using a field test kit or instrument (EPA 1986). Examples of unstable parameters include pH, redox potential, dissolved oxygen, and temperature. Although specific conductivity of a substance is relatively stable,

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it is recommended that this characteristic be measured in the field. Most instruments measuring conductivity require temperature compensation; therefore, the temperature of the samples should be measured at the time conductivity is measured.

The spectrophotometric methodology utilized in this SOP is applicable to a wide range of chemical analyses. Turbidity, nitrate/nitrite, and many other parameters may be measured using this methodology. If analyses not addressed in this SOP are needed, and may be performed using this methodology, they will be added as addenda to this SOP. All field analyses will meet the data quality objectives specified in the subcontractor's Quality Assurance Project Plan (QAPjP).

The standardization/calibration of in situ monitoring equipment or field-test probes and kits will be completed according to the manufacturer's specifications, at the frequency specified in the appropriate work plans or at the minimum frequency specified in Table GW.5-1 of this SOP. Instruments which meet the acceptance criteria specified in Table GW.5-1 or more stringent criteria specified in the work plans are acceptable for use in the field. Instruments not meeting the specified criteria must be calibrated prior to each use, so that the acceptance criteria are met. Samples will be collected for field parameter measurements according to SOP GW.6, Groundwater Sampling.

Solutions used for standardizing, calibrating, or titrating will be checked prior to use in the field to determine if the expiration dates have been exceeded. Any expired solutions will be discarded and replaced with new solutions.

Forms applicable to this SOP are contained in SOP GW.6, Groundwater Sampling.

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TABLE GW.5-1
Calibration/Standardization Frequencies and
Minimum Acceptance Criteria for Field Measurements

<u>Parameter</u>	<u>Minimum Frequency</u>	<u>Procedure</u>	<u>Acceptance Criteria</u>
pH	Each well	Calibrate	Standard Value \pm 0.2 pH units
Specific Conductance	Each well	Calibrate	Standard Value \pm 10%
Temperature	Weekly	Calibrate ^a	\pm 1.0°C (difference between measured value and NIST calibrated thermometer or thermometer calibrated against an NIST traceable thermometer)
Dissolved Oxygen - Electrode Sensor	Each well	Calibrate	Standard Value \pm 10%
Dissolved Oxygen Photometric Baseline ^b	Each sample	Zero instrument	0.0 mg/l
Total Alkalinity	For each new lot of titrant	Standardize	Standard Value \pm 10%
Nitrate/Nitrite as N Baseline ^b	Each sample Each lot	Zero instrument Determine Reagent	0.0 mg/L < 1.0 mg/l
Nitrite	(landfill only)	Calibrate	0.0 mg/l
Blank Value on each new lot of nitra-ver 5 ampuls	Each lot	Standardize each new lot of nitra-ver 5 ampuls ^c	Standard Value \pm 10%
Turbidity Baseline ^b	Each sample each new lot of calibration reagent	Zero Instrument Check Standard Solution	0 FTUs \pm 2 FTUs

^a Instruments that will be utilized to measure temperature sensitive parameters (pH, specific conductance, and D.O.) will also require weekly calibration or standardization if they are utilized to measure temperature to adjust the associated temperature sensitive parameter value.

Some of these instruments may allow for actual field calibration while others will not be capable of temperature adjustment and may therefore only be standardized. Instruments which allow for actual field calibration will be calibrated weekly. Instruments that do not allow for calibration will be standardized. If the standardization is outside the acceptance limits, the instrument should be returned to the manufacturer for maintenance and repair.

^b A chemically untreated portion of the sample will be used as a blank to establish a zero level for the parameter prior to measurement of the chemically treated sample.

^c Follow manufacturer's recommendations for standardizing the reagent lots, to be utilized for field measurements.

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5.1 TEMPERATURE

Temperature measurements will be made with a high quality mercury-filled thermometer or thermistor having an analog or digital readout device that has been standardized by comparison with a thermometer calibrated against a National Institute of Standards and Technology (NIST) calibrated thermometer. Since the temperature on the HACH pH meter is capable of being calibrated by field personnel, instead of only standardized, the pH meter will be utilized to determine water temperatures in the field unless a standardized mercury thermometer is used. All temperature measuring devices will be scaled to indicate degrees Celsius in increments of 1°C or less as appropriate to meet data quality objectives. Thermometers will be the Teflon[®]-coated safety type of thermometer. Glass thermometers will be transported in a protective case to prevent breakage. Field thermometers will also be enclosed in an armored casing to prevent breakage.

Temperature measurements made for the purpose of providing adjustment factors for other field parameters will be conducted simultaneously with those related measurements. Volumes and methods of collection will be determined by the procedural requirements of the primary field measurement taken. Thermometers or thermistors used in the field will be standardized at least weekly against an NIST traceable thermometer. This standardization will be verified weekly. Verification will consist of comparing the temperature measured with the field instrument against an NIST traceable thermometer. If the result of calibration verification shows a variance of more than 1.0 degrees Celsius, the thermometer or thermistor will be taken out of service. Thermometers and thermistors that cannot be calibrated within the variance criteria will be replaced.

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5.1.1 Temperature Measurement by Thermometer

The following procedure will be used when collecting temperature measurements using a thermometer:

- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing field temperature measurements. The Site Health and Safety Plan will be followed at all times.
- A mercury-filled standardized thermometer will be used.
- Standardize the thermometer at least weekly to the criteria specified in Table GW.5-1. If the acceptance criteria specified in Table GW.5-1 are not met, replace the thermometer so that the acceptance criteria presented in Table GW.5-1 are met.
- The thermometer will be inspected before each field trip to ensure that there are neither cracks in the glass nor air spaces or bubbles in the mercury.
- A portion of well water will be transferred to a beaker previously rinsed with distilled water. The thermometer will be inserted into the sample collection container, and the sample in the container will be swirled. The temperature reading will be taken when the mercury column stabilizes.
- The temperature measurement will be recorded to the nearest 0.5°C.
- The thermometer will be decontaminated in accordance with SOP FO.3, General Equipment Decontamination.

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- Liquids and materials from decontamination operations will be handled in accordance with SOP FO.7, Handling of Decontamination Water and Wash Water.

5.1.2 Temperature Measurement by HACH ONE pH Meter

- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing field temperature measurements. The Site Health and Safety Plan will be followed at all times.
- Standardize the probe at least weekly to the criteria specified in Table GW.5-1. If the acceptance criteria specified in Table GW.5-1 are not met, calibrate the instrument following the manufacturer's instructions so that the acceptance criteria presented in Table GW.5-1 are met.
- A portion of well water will be transferred to a beaker previously rinsed with distilled water. The probe will be inserted into the sample collection container, and the sample in the container will be swirled. The temperature reading will be taken when the digital readout stabilizes.
- The temperature will be recorded on the field logsheet to the nearest $\pm 0.1^{\circ}\text{C}$.
- The probe will be decontaminated in accordance with SOP FO.3, General Equipment Decontamination.
- Liquids and materials from decontamination operations will be handled in accordance with SOP FO.7, Handling of Decontamination Water and Wash Water.

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5.2 DISSOLVED OXYGEN (D.O.) - SPECTROPHOTOMETER

This procedure describes the measurement of D.O. using the High Range D.O. AccuVac Ampul and measuring the color change in a spectrophotometer at 535 nm. The high range D.O. AccuVac Ampul contains a reagent vacuum sealed in a 12-ml ampul. When the ampul is broken open in a sample containing D.O., it forms a yellow color which then turns purple. The method is applicable to D.O. measurements up to 13.0 mg/l O₂. The following method will be used to measure D.O. concentrations in groundwater when using the HACH DR2000 Spectrophotometer:

- Press "445" and then "READ/ENTER" to enter the stored program number for D.O.
- When the display shows "DIAL nm to 535," rotate the wave length dial until the display shows "535 nm."
- Press the "READ/ENTER" key. The display will show "mg/l O₂ HRDO."
- Fill a blank zeroing vial with at least 10 ml of sample, and fill a blue ampul cap with sample.
- Prior to collection of the sample water for D.O. measurement, the sample beaker shall be rinsed with distilled water. Fill a high-range D.O. AccuVac Ampul with sample by holding the ampul with the pointed tip down in a beaker containing 50 to 100 ml of sample and breaking the tip of the ampul with your gloved finger. Be sure to keep the tip of the ampul immersed in the water at all times while the ampul fills completely.
- Immediately after filling and without inverting the ampul, securely place the blue ampul cap that has been filled with the sample over the tip of the AccuVac ampul,

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and shake the ampul for approximately 30 seconds. Securing the cap is important in order to prevent contamination with atmospheric oxygen. If the seal between the ampul and cap is breached, discard the ampul and start again.

- Press the "SHIFT" and then the "TIMER" keys. A two-minute reaction period will begin in which oxygen, which was degassed during aspiration, is allowed to redissolve and react. Wipe the outside of the ampul with a paper towel to clean off finger prints and any liquid present, and then place the sample ampul into the AccuVac vial adapter. Place the vial adapter containing the sample into the cell holder. The grip tab on the vial adapter is placed to the rear of the cell holder.
- When the timer beeps and the display shows "mg/l O₂ HRDO," remove the adapter from the cell holder and shake the ampul for 30 seconds by continuing to invert the ampul.
- Place the blank in the cell holder and close the lid.
- Press "zero" and the display will show "wait" then "0.0mg/L O₂ HRDO."
- Remove the blank and place the AccuVac vial adapter containing the sample into the cell holder with the grip tab on the vial adapter to the rear of the cell holder.
- Close the lid, and after waiting approximately 30 seconds for the air bubbles to disperse from the light path, press the "READ/ENTER" key.
- Record the displayed result as mg/l of D.O.

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5.3 pH

This procedure describes the method to be utilized to measure pH in the field using a HACH ONE (model 43800-00) pH meter. If an instrument other than the HACH ONE pH meter is used, the manufacturer's instructions will be followed for calibration and use. All pH meters used for field measurements will be temperature compensating.

Measurements in the field will be performed in the following manner:

- Meters will be calibrated daily prior to the start of field activities following the manufacturer's instructions. The automatic calibration mode will be used with buffers of pH 7 and pH 4.
- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing field pH measurements.
- Before each field activity, the meter will be checked for cracked or fouled electrodes and battery condition in accordance with the manufacturer's recommendations.
- Calibration will be verified immediately before the pH measurement is taken. Verification will consist of recording the instrument reading of a pH 7 standard solution.
- The probe and sample beaker will be thoroughly rinsed with distilled water and the excess water removed.
- The probe will be placed into the beaker containing 100 ml of sample, the dispenser button pressed, and the electrode swirled at a constant rate until the meter reading

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stabilizes. The stirring rate should be maintained so as to minimize the surface disturbance of the sample.

- The pH will be read and recorded to the nearest ± 0.01 pH unit.
- The probe will be rinsed thoroughly with distilled water and stored in accordance with the manufacturer's recommendations.
- Sampling tools, instruments, and equipment will be protected from sources of contamination before use and will be decontaminated after use as specified in SOP FO.3, General Equipment Decontamination.
- Calibration will be verified at the end of each work day. Verification will consist of recording the instrument reading of a pH 7 standard solution. If the instrument reading varies from the standard by more than $\pm .2$ pH units, the instrument will be checked for a malfunction. If the variance continues for two consecutive days, the frequency of calibration of that instrument will be increased. Calibration will then be performed prior to use at each site.

5.4 SPECIFIC CONDUCTANCE

Conductance is the reciprocal of resistance, and, therefore, is often reported in units of reciprocal ohms or mhos. The international system of units, the siemen (S), will be used to report conductivity for this program. Most waters have a specific conductance much less than 1 siemen; therefore, data will be reported in microsiemens (μ S)/cm. Measurements should be made prior to conducting the pH measurement sequence.

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This procedure describes the method to be utilized to measure specific conductance in the field using a HACH Conductivity/TDS Meter (model 44600). If an instrument other than the HACH meter is used, the manufacturer's instructions will be followed for calibration and use. All conductivity meters used for field measurements will be temperature compensating. This will allow for the recording of specific conductance measurements directly from the meter. All conductivities will also have adjustable readings which will allow for accurate calibration to a known standard.

The following method will be used to measure specific conductance in the field using the HACH conductivity meter:

- The meter will be calibrated at the start of each day prior to any field activities. Calibration will be performed according to manufacturer's instructions and the guidance given in Table GW.5-1. All three instrument ranges will be calibrated with one standard solution. A solution of 1.99 mS/cm NaCl is preferred.
- Before each field activity, the meter will be checked for damage to the probe and for weak batteries in accordance with manufacturer's recommendations.
- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing field conductivity measurements.
- The probe and sample beaker will be thoroughly rinsed with distilled water, and excess water will be removed.
- The probe will be immersed in the sample to a depth of at least 1 inch below the surface of the sample. The probe will be agitated vertically to dislodge any trapped air bubbles and to allow the meter reading to stabilize.

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- The temperature and the temperature-compensated conductance reading will be recorded on the field form.
- The probe will be thoroughly rinsed with distilled water after use.
- Sampling tools, instruments, and equipment will be protected from sources of contamination before use and will be decontaminated after use as specified in SOP FO.3, General Equipment Decontamination.
- Calibration will be verified at the end of each day. Verification will consist of recording the instrument reading of a standard solution. If the instrument reading varies by more than 10 percent of the standard, the instrument will be checked for malfunction. If the variance continues for two consecutive days, the calibration frequency for that instrument will be increased. Calibration will then be performed prior to each sampling event.

5.5 TOTAL ALKALINITY- ORION TOTAL ALKALINITY TEST KIT

This section describes the procedure that will be utilized to measure total alkalinity in the field using an ORION Total Alkalinity Test Kit and HACH ONE. The ORION test kit includes a reagent composed of several acids and a conversion wheel. After the pH of a solution is recorded, the reagent is added to the solution, and the pH is recorded again. Using the conversion wheel, the pH difference is converted to total alkalinity as ppm CaCO₃. The procedure for determining total alkalinity is as follows:

- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing total alkalinity measurements.

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- The pH meter will be calibrated as described in Subsection 5.3. During the daily calibration of the pH meter, verification of proper alkalinity measurement will be performed following manufacturer's instructions. If the verification measurement varies by more than 10 percent, the reagent and standard will be replaced.
- At the appropriate point during a sampling event, transfer 100 ml of sample into a clean beaker.
- Measure and record the pH of the sample using the HACH ONE pH meter as described in Subsection 5.3.
- Dispense 10 ml of Total Alkalinity Reagent into the sample and stir well using the pH probe.
- Measure and record the resulting pH of the sample as described in Subsection 5.3.
- Using the gray side of the Total Alkalinity Conversion Wheel, find the resulting pH value and record the total alkalinity of the sample.
- If the alkalinity is off scale, the measurement will be repeated using a fresh sample. The sample and reagent volume will be adjusted according to the manufacturer's instructions.
- Upon completion of all alkalinity measurements, the probe will be thoroughly rinsed with distilled water after use.

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- Sampling tools, instruments, and equipment will be protected from sources of contamination before use and will be decontaminated after use as specified in SOP FO.3, General Equipment Decontamination.

5.6 NITRATE/NITRITE AS N - CADMIUM REDUCTION SPECTROPHOTOMETRIC METHOD

This procedure describes the measurement of nitrate/nitrite nitrogen using the HACH DR2000 Spectrophotometer and HACH AccuVac Ampuls. The method is a cadmium reduction method in which cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product which is measured with the spectrophotometer at 500 nanometers. Nitrate/nitrite nitrogen from 0 to 30 mg/l may be measured with this method. Tests with standard solutions and a single operator have yielded a standard deviation of ± 1.0 mg/l nitrate/nitrite nitrogen. The procedure for measuring nitrate/nitrite as N using HACH AccuVac Ampuls is as follows:

- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing nitrate/nitrite measurements.
- Press "360" and the "READ/ENTER" key to enter the stored program number for high range nitrate nitrogen AccuVac Ampuls.
- When the display shows "Dial nm to 500," rotate the wave length dial until the display reads "500 nm."
- Press "READ/ENTER."
- After the display reads "mg/l N NO₃ H AV," collect at least 40 ml of sample in a beaker and fill a NitraVer 5 Nitrate AccuVac Ampul with the sample. Fill the ampul

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by holding the ampul with the pointed tip down into the water sample in the beaker and breaking the tip of the ampul against the bottom of the beaker. Keep the tip immersed until the ampul fills completely.

- Immediately press the "SHIFT" and then the "TIMER" keys. This begins a one-minute mixing period. After pressing the "SHIFT" and "TIMER" keys, invert the ampul repeatedly to mix the sample, until the timer beeps.
- When the timer beeps, press the "SHIFT" and then the "TIMER" keys. This begins a five-minute reaction period. During this reaction period, wipe off liquid or fingerprints on the ampul, place the ampul into the AccuVac Vial Adapter, and place the adapter containing the sample ampul into the cell holder.
- Fill a blank zeroing vial, previously rinsed with distilled water, with at least 10 ml of sample.
- When the timer beeps and the display shows "mg/l N NO₃H AV," remove the sample and place the blank vial into the cell holder.
- Close the lid and press the "CLEAR/ZERO" key. The display will show "Wait" and then "0.0 mg/l N NO₃ H AV."
- Remove the blank vial; place the AccuVac vial adapter containing the sample into the cell holder with the grip tab positioned at the rear of the cell holder and close the lid.
- Press the "READ/ENTER" key and record the result displayed as mg/l of nitrate/nitrite as nitrogen.

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- A reagent blank value must be determined on each new lot of Nitraver 5 Ampuls. To determine the reagent blank value, repeat the measurement procedure by substituting distilled water as the sample. Repeat the measurement for 3 ampuls from the lot and take the average of the three values as the reagent blank value. Use this value as the reagent blank value for all nitrate/nitrite measurements performed with this lot of Nitraver 5 Ampuls.
- Subtract the value of the reagent blank from the recorded value to obtain the actual (corrected) nitrate/nitrite-nitrogen concentration in mg/l, and record.

5.7 TURBIDITY

This procedure describes the measurement of turbidity using the HACH DR2000 Spectrophotometer absorptometric method. The turbidity test measures an optical property of the water sample which results from the scattering and absorbing of light by the particulate matter present. The amount of turbidity registered is dependent on such variables as the size, shape, and refractive properties of the particles. This procedure is calibrated using formazin turbidity standards, and the readings are in terms of formazin turbidity units (FTU).

- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performingng turbidity measurements.
- Enter the stored program number for turbidity; press "750 Read/Enter." The display will show "Dial nm to 450."
- Rotate the wave length dial until the small display shows "450 nm."
- Press "Read/Enter." The display will show "FTU Turbidity."

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- Pour 25 ml of deionized water (blank) into a sample cell.
- Place the blank into the cell holder and close the light shield.
- Press "zero" and the display will show "wait" and then "0 . FTU Turbidity."
- Agitate the sample designated for turbidity measurement so that all sediments are suspended. Pour 25 ml of the sample into another clean sample cell; place into the cell holder and close the light shield.
- Press "Read/Enter" and the display will show "wait" and then the result in FTUs will be displayed.

5.7.1 Accuracy Check: Standard Solution Method

The stored program has been calibrated using a milky white suspension of a polymer called formazin. The turbidity of this stock solution is 400 FTU and it should be prepared monthly. Standard formazin solutions for checking the spectrophotometer accuracy can be prepared as follows:

- Dissolve 1.000 gram of hydrazine sulfate in deionized water and dilute to 100-ml volumetric flask.
- Dissolve 10.00 grams of hexamethylenetetramine in deionized water and dilute to 100-ml volumetric flask.
- Mix 5.0 ml of each solution in a 100-ml volumetric flask and allow to stand undisturbed for 24 hours at $25^{\circ} \pm 3^{\circ}\text{C}$.

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- Dilute to the 100-ml mark and mix.

6.0 DOCUMENTATION

All records of field measurements will be recorded on the appropriate forms contained in SOP GW.6, Groundwater Sampling.

All instrument calibration/standardization activities will be recorded on a calibration/standardization logsheet or in a bound field notebook specific to each instrument. Records will be maintained in a locked filing cabinet and will be reviewed periodically by the project QA/QC officer.