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QUALITY ASSURANCE PROJECT PLAN (QAPjP)
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

H.O.D. LANDFILL
ANTIOCH, ILLINOIS

VOLUME 2 of 2

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PREPARED FOR:
WASTE MANAGEMENT OF ILLINOIS, INC.
WESTCHESTER, ILLINOIS

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PREPARED BY:
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QUALITY ASSURANCE PROJECT PLAN (QAPjP)
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
H.O.D. LANDFILL SITE
ANTIOCH, ILLINOIS

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FIELD MEASUREMENT SOPs

APPENDIX C-1

PH

pH

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: EPA 1983, p. 150.1

Sensitivity: 0.01 pH unit

Optimum Range: pH 1.00 to 12.00

Sample Handling: Determine on-site, if possible, otherwise within 24 hours.

Reagents and Apparatus:

1. pH meter
2. Combination pH electrode.
3. Magnetic stirrer and stir bars (for lab use).
4. Beakers or plastic cups.
5. pH buffer solutions, pH 4.00, 7.00, and 10.0.
6. Deionized water

Notes:

1. The pH test is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C of each other. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

2. Interferences in pH measurements occur with presence of weak organic and inorganic salts, and oil and grease. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.

3. Electrode should be stored in pH 7.00 buffer, or electrode storage solution.

4. **Before leaving laboratory for field work:**

   a. Check batteries.

   b. Do quick calibration at pH 7.00 and 4.00 to check electrode response and batteries.

   c. Obtain fresh pH buffer solutions.

5. **Following field measurements:**

   a. Report any problems with meter or electrode.

b. Clean meter and meter case.

c. Make sure electrode is stored in pH 7.00 buffer.

Calibration:

1. Place combination electrode in fresh pH 7.00 buffer solution.
2. After allowing meter to stabilize, turn calibration dial until reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4.00 buffer solution.
4. Wait for reading to stabilize and then turn slope adjustment dial until reading of 4.00 is obtained.
5. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00 + 0.05, follow Steps 2-5 again.
6. Rinse electrode with deionized water and place in pH 10.00 buffer. Reading must be in the range of 9.90-10.10 or calibration must be repeated.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses.
2. Calibrate meter using calibration procedure.
3. Pour the sample into clean beaker or plastic cup.
4. Place stir bar in beaker and put on magnetic stirrer (low speed) for lab measurement of pH. Swirl cup gently for field measurement of pH.
5. Check temperature of sample. It should be ± 2°C of the buffer solutions.
6. Rinse electrode with deionized water.
7. Immerse electrode in sample. The white KCl junction on side or bottom of electrode must be fully immersed in solution. Allow sufficient time for reading to stabilize. Record pH. Rinse electrode with deionized water.
8. Check calibration with pH 7.00 buffer solution initially and after every 10 samples and at the end of the analytical run and record on data sheet. Buffer solution should read ±0.05 of true value.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be within acceptable ranges. Average the results.

Revision Dates

7-22-86

6-28-90

1-3-92

Manager, Operations

Supervisor, Quality Assurance

Kim Johnson

Director, Analytical Services

APPENDIX C-2

CONDUCTIVITY

CONDUCTIVITY YSI METER

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance (Electrical Conductivity), umhos/cm @ 25°C

Reference: EPA 1983, Method 120.1

Detection Limit: 10 umhos/cm @ 25°C

Sample Handling: Determine on-site or within 28 days

Reagents and Apparatus:

1. Conductivity meter, YSI 33 SCT
2. Deionized water
3. conductivity standard, 1000 umhos/cm @ 25°C. Commercially Available

Notes:

1. All conductivity readings must be corrected to 25°C.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized rinsed prior to analysis.

2. With mode switch of the meter in the OFF position. Check the zero setting. If not at zero, use meter adjusting screw to zero (on front of the meter).

3. Plug probe into jack located on side of meter.

4. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if meter cannot be aligned.

5. Analyze the conductivity standard. If the result is within 90-110% of the standard value, analyze samples. A standard should be analyzed after every 10 samples and at the end of the analytical run. Table 1 lists non-temperature compensated values for both of the standards. Use this table as a reference when a calculator is not available to perform temperature correction on the standards.

6. Totally immerse and suspend the probe in the water sample. Do not allow probe to touch the sides of the sample container.

7. Turn mode switch to the appropriate conductivity scale (X100, X10, or X1). Use the scale that produces a mid-range output on the meter.

8. Wait for needle to stabilize (about 15 seconds) and record conductivity. Multiply the conductivity reading by the scale setting.

9. While gently agitating the probe, take sample temperature (°C) to nearest 0.5°C and record.

10. Rinse probe with deionized water.

11. Record specific conductivity and temperature.

12. Store probe in D.I. water between uses whenever possible.

13. Turn mode switch to the OFF position when finished.

Quality Control:

1. A quality control calibration standard of 1000 umhos/cm is to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within 90-110% of the standard value or the samples run after the last acceptable check standard are to be reanalyzed.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

Calculation:

1. **Calculate specific conductivity at 25° C using following formula:**

$$G_{25} = \frac{G_T}{[1 + 0.02\,(T-25)]}$$

G_{25} = Specific conductivity at 25° C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T, umhos/cm

Table 1
NON-TEMPERATURE COMPENSATED CONDUCTIVITY STANDARD READINGS FOR 1,000 AND 10,000 umhos/cm STANDARDS

Conductivity Standard 1,000 umhos/cm			Conductivity Standard 10,000 umhos/cm		
Conductivity Standard Should Read	**at**	**Degrees C°**	**Conductivity Standard Should Read**	**at**	**Degrees C°**
500		0	5000		0
520		1	5200		1
540		2	5400		2
560		3	5600		3
580		4	5800		4
600		5	6000		5
620		6	6200		6
640		7	6400		7
660		8	6600		8
680		9	6800		9
700		10	7000		10
720		11	7200		11
740		12	7400		12
760		13	7600		13
780		14	7800		14
800		15	8000		15
820		16	8200		16
840		17	8400		17
860		18	8600		18
880		19	8800		19
900		20	9000		20
920		21	9200		21
940		22	9400		22
960		23	9600		23
980		24	9800		24
1000		25	10,000		25
1020		26	10,200		26
1040		27	10,400		27
1060		28	10,600		28
1080		29	10,800		29
1100		30	11,000		30
1120		31	11,200		31
1140		32	11,400		32
1160		33	11,600		33
1180		34	11,800		34
1200		35	12,000		35

Revision Date

9-06-86

4-27-87

2-17-88

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1-4-92

Manager, Operations

Supervisor, Quality Assurance

Kim Johnson

Director, Analytical Services

APPENDIX C-3

TEMPERATURE

Effective Date: 3-13-92

FIELD TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater, and groundwater.

Method: Thermometric

Reference: EPA 1983, Method 170.1

Reagents and Apparatus:

1. Thermometer; mercury-filled, or temperature probe, °C
2. Plastic bottles, 250 mL
3. Deionized water
4. Kimwipes

Notes:

1. The thermometer or temperature probe must be calibrated annually against an ASTM certified thermometer.

2. It is recommended the thermometer or temperature probe have a range of -10 to 100°C with 0.5° sensitivity.

Procedure:

1. Pour approximately 100-150 mL sample into a plastic bottle.

2. Place thermometer or temperature probe in sample and swirl gently for 30-60 seconds.

3. Record the temperature to the nearest 0.5 degree.

4. Rinse thermometer with deionized water and blot dry with a kimwipe.

5. Continue as above with next sample.

Quality Control:

1. Duplicate minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

APPENDIX C-4

DISSOLVED OXYGEN

YSI DISSOLVED OXYGEN METER AND PROBE

Scope and Application: The instructions outlined below are to be followed for the daily calibration and routine operation of the YSI Dissolved Oxygen Meter and Probe.

Reference: Instruction Manual YSI Model 54ARC Dissolved Oxygen Meter and YSI 5700 Dissolved Oxygen Probe.

Reagents and Apparatus:

1. YSI 54ARC Dissolved Oxygen Meter
2. YSI 5720A B.O.D. Bottle Probe
3. Membrane and KCl kit, standard, YSI 5775
4. Replacement "O" ring, YSI Part #5945
5. Beater boot assembly, YSI Part #5486

Operating Procedure:

I. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probe as follows.

ALL PROBES ARE SHIPPED DRY - FOLLOW THESE INSTRUCTIONS
TO PREPARE FOR USE

1. Prepare the electrolyte by dissolving the KCl crystals in a dropper bottle with Milli-Q water. Fill the bottle to the top.

2. Remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.

3. Fill the probe with electrolyte as follows (see Figure 1):

   a. Grasp the probe with your left hand.

   b. Fill the sensor body until no more air bubbles appear. Tap the probe against the countertop to dislodge any air bubbles adhering to the sensor.

   c. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.

   d. With the thumb and forefinger of your other hand, grasp the free end of the membrane.

e. Using a continuous motion stretch the membrane UP, OVER, and DOWN the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to approximately 1 1/2 times its normal size.

f. Secure the end of the membrane under the forefinger of the hand holding the probe.

g. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.

h. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

4. Shake off excess KCl.

5. Store the probe in a BOD bottle containing about 1 inch of water.

6. Membranes average replacement is 2-4 weeks. If the electrolyte in the probe is allowed to evaporate, air bubbles form under the membrane. If air bubbles are noted under the membrane or if the membrane becomes damaged, thoroughly flush the reservoir with fresh KCl and install a new membrane as described above.

7. Replace the membrane if erratic readings are observed or if calibration is not stable.

NOTE: The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or abrasives in an attempt to clean it.

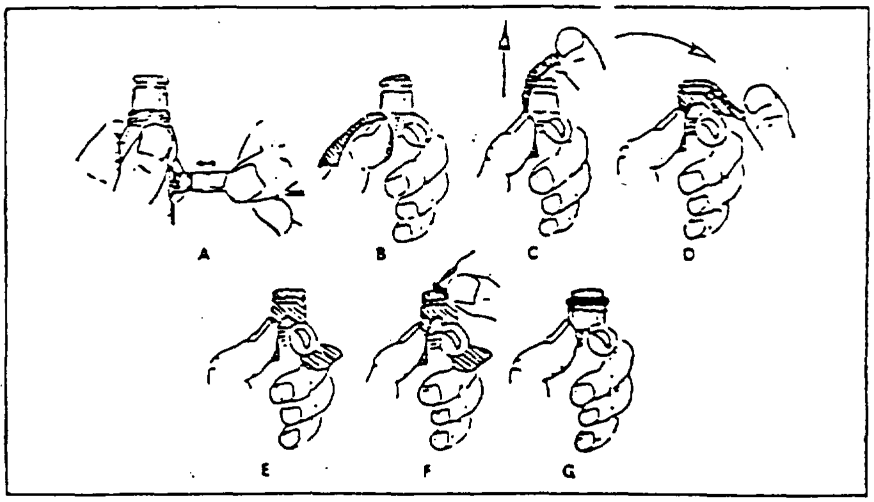

Figure 1

II. Preparing the YSI Instrument

It is important that the instrument be placed in the intended operating position; vertical, tilted, or on its back - before it is prepared for use and calibrated. (See Figure 8). Recalibration may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument operating position is changed.

2. Switch to RED LINE and adjust with the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.

3. Switch to ZERO and adjust to zero with zero control knob.

4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.

5. For optimum probe stabilization, let the meter and probe equilibrate for 15 minutes before calibrating.

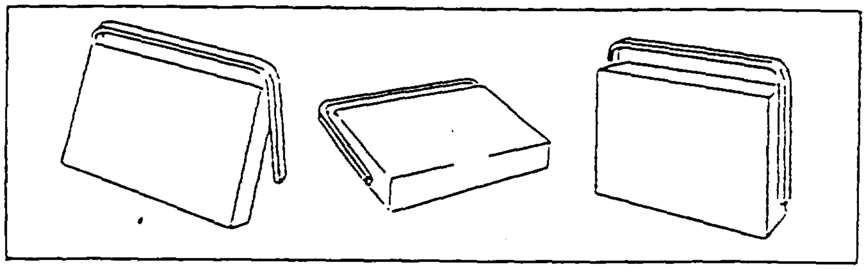

Figure 2

III. Calibration

The operator has a choice of three calibration methods: Winkler titration, Saturated Water, and Air. The three methods are described in the following paragraphs. The Winkler titration is the preferred method of calibration.

Winkler Titration

1. Determine the dissolved oxygen in two samples from the aerated water source using the Winkler titration technique (refer to the Dissolved Oxygen SOP) and average the values. If the values differ by more than 0.5 mg/L, discard the samples and repeat.

2. Place the YSI probe in the third sample and stir.

3. Switch to desired mg/L scale range and adjust with the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability (Readjust if necessary).

Saturated Water Calibration

1. Air saturate a volume of water by aerating for at least 1 hour at a constant temperature (preferably room temperature).

2. Place the probe in the sample and stir. Switch to TEMPERATURE scale. Refer to Calibration Table I for the mg/L value corresponding to the temperature.

3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.

4. Multiply the mg/L value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/L. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is 8.9 mg/L x 0.96 = 8.54 mg/L.

5. Switch to an appropriate mg/L scale range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

Air Calibration - Fresh Water

1. Place the probe in a BOD bottle partially filled with water. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.

2. Switch to TEMPERATURE and read. Refer to Table I - Solubility of Oxygen in Fresh Water, and determine calibration value.

3. Determine altitude or atmospheric correction factor using Table II.

4. Multiply the calibration value from Table I by the correction factor from Table II.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/L. From Table II the correction factor for 1000 feet is about 0.96. Therefore the corrected calibration value is 8.9 mg/L x 0.96 = 8.54 mg/L.

5. Switch to an appropriate mg/L range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability. Readjust if necessary.

IV. Dissolved Oxygen Measurement

1. With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and turn on stirring boot.

2. Allow sufficient time for probe to stabilize to sample.

3. Read dissolved oxygen directly from scale.

V. Maintenance of the Stirrer Boot

1. The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage which may allow leaking into the motor housing, the boot must be replaced.

2. In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot with deionized water after use in contaminated samples.

3. Boot replacement is as follows:

   a. Pull off old assembly and clean shaft.

   b. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.

   c. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

TABLE I

Solubility of Oxygen in Fresh Water

Temperature °C	mg/L Dissolved Oxygen	Temperature °C	mg/L Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derrived from 16th Edition "Standard Methods for the Examination of Water and Wastewater".

This table shows the amount of oxygen in mg/L that is dissolved in air saturated fresh water at sea level (760 mm Hg atmospheric pressure) as temperature varies from 0° to 45°C.

Table II

Correction for Atmospheric Pressure

Atmospheric Pressure mm/Hg	or	Equivalent Altitude Ft.	=	Correction Factor
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1628		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derrived from 16th Edition "Standard Methods for the Examination of Water and Wastewater".

This table shows the correction factor that should be used to correct calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmoshperic pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

YSI DISSOLVED OXYGEN METER AND PROBE

Detection Limit: 1.00 mg/L Dissolved Oxygen

Working Concentration Range: 0 - 15 mg/L Dissolved Oxygen

YSI 5700 SERIES DISSOLVED OXYGEN PROBES INSTRUCTIONS

he probes described in these instructions are designed or direct use with YSI Models 50, 51B, 54ABP, 54ARC, 56, 57 and 58 Dissolved Oxygen Meters. The probes can also be used with discontinued YSI Models 51A, 54BP and 4RC Dissolved Oxygen Meters when the YSI 5735 Cable dapter is employed.

PRINCIPLES OF OPERATION

YSI 5700 Series Probes are polarographic sensors. A thin permeable membrane stretched over the sensor isolates the electrodes from the environment, but allows gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the difference across it in partial pressure of oxygen. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure under the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the partial pressure of oxygen outside the membrane. As the oxygen partial pressure varies, both the oxygen diffusion through the membrane and the probe current will change proportionally.

SPECIFICATIONS

Cathode: Gold
Anode: Silver
Membrane: .001" FEP Teflon, standard
Electrolyte: Half saturated KCl
Temperature Range: -5° to 45°C
15° to 35°C for the 5760 probe
Temperature Accuracy: ±0.2°C
Temperature Compensation: (see instrument specifications)
Polarizing Voltage: 0.8 Volts (nominal)
Probe Current in Air at 30°C: 19 microamps (nominal)
in Nitrogen at 30°C: 0.15 microamps or less
Response Time: Typical response for dissolved oxygen, using standard membranes, is 90% in 10 seconds at a constant temperature of 30°C.
Response at low dissolved oxygen levels is typically 90% in 30 seconds.

ACCESSORIES AND REPLACEMENT PARTS

YSI 5492A Battery Pack for Models 51B and 54A (Powers the submersible stirrers.)
YSI 5735 Cable Adapter (Mates 5700 Series probes with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters)

Accessories for the 5720A, 5739 and 5750

YSI 5680 Probe Reconditioning Kit. Includes a sanding tool and ten adhesive disks.
YSI 5775 Membrane and KCl Kit, Standard. Includes two 15-membrane packets (.001" thick standard FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo.
YSI 5776 Membrane and KCl Kit, High Sensitivity. Includes two 15-membrane packets (.0005" thick FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo. Used for measurements below 15°C and/or for low oxygen levels
YSI 5793 .001" membranes, 10-membrane packet
YSI 5794 .0005" membranes, 10-membrane packet
YSI 5945 O-ring pack (Contains replacement sensor O-rings)

Accessories for the 5720A Only

YSI 5486 Stirrer Boot Assembly

Accessories for the 5739 Only

YSI 5075A Calibration Chamber
YSI 5986 Diaphragm Kit

YSI 5740-10	detachable 10' cable
YSI 5740-25	detachable 25' cable
YSI 5740-50	detachable 50' cable
YSI 5740-100	detachable 100' cable
YSI 5740-150	detachable 150' cable
YSI 5740-200	detachable 200' cable

YSI 5791A Submersible Stirrer with 50' cable for stirrer only
YSI 5795A Submersible Stirrer with 50' combined probe and stirrer cable

YSI 5720A BOD BOTTLE PROBE

The 5720A bottle probe (Figure 1) is used for measuring dissolved oxygen in standard BOD bottles. It is provided with a stirrer powered by a DC supply available for 115 or 230 VAC input.

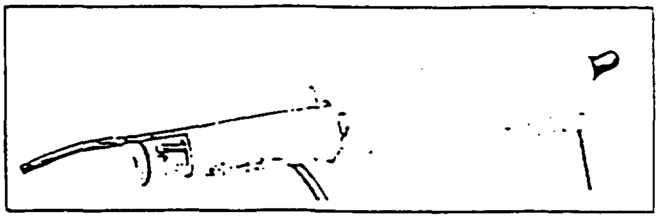

Figure 1. The YSI 5720A Probe

To use the 5720A, plug the stirrer power supply into line power and the probe plug in the instrument. With the stirrer off, place the tapered probe end into a filled the BOD bottle and turn on the stirrer. The probe should be operated with a minimum of trapped air in the bottle. A slight amount of air in the unstirred region at the top may be neglected, but no bubble should be permitted around the sensor. CAUTION: The motor housing is not waterproof; do not submerge this probe beyond the part that is inserted into a BOD bottle.

Stirrer Boot (YSI 5486)

The 5720A uses a flexible stirring boot to transmit motion from the motor housing to the sample. If the boot shows signs of cracking or other damage liable to allow leakage into the motor housing, it must be replaced. Running the 5720A with a damaged stirring boot could cause permanent motor damage. Boot life may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life, rinse the boot after each use. Boots are replaced as follows:

1. Pull off the old assembly and clean the stir rod housing.

2. Slide on the new assembly, making sure the back spring is over the grooved area of the stir rod housing. A drop of alcohol will aid installation by providing lubrication.

3. Do not permit the stir rod to press against the end of the stirrer boot tip or it will bind.

YSI 5739 DISSOLVED OXYGEN PROBE

The 5739 probe system consists of the probe body plus a detachable cable (see Figure 2). The detachable cable is a convenience feature that facilitates changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retainer. The assembly is not intended for casual disconnection; cable and probe should be separated only when necessary.

To detach the cable, unscrew the retainer and slide it down the cable to expose the connector. Pull gently on the connector until it comes away from the probe body. If the O-ring is frayed or damaged, replace it: a replacement O-ring is supplied with each 5740 cable. Reassemble by pushing the connector into the probe body, rotating it until the two halves mate. A light coating of silicone grease on the O-ring will make reassembly easier. Be sure the connector is dry; otherwise, erratic readings may result. Screw on the retainer <u>finger-tight only.</u>

Pressure Compensation

The 5739 probe has a unique pressure compensating system that helps assure accurate readings at great depths. Pressure compensation is effective to 1/2% of reading with pressures up to 100 psi (230 feet of water). The compensating system does not normally require service and should not be taken apart. However, if electrolyte is leaking through the diaphragm, or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Use a coin to unscrew the retaining plug and remove the washer and diaphragm. With distilled water, flush any salt crystals from the reservoir, install a new diaphragm (flat side out), replace the washer and securely screw in the retaining plug.

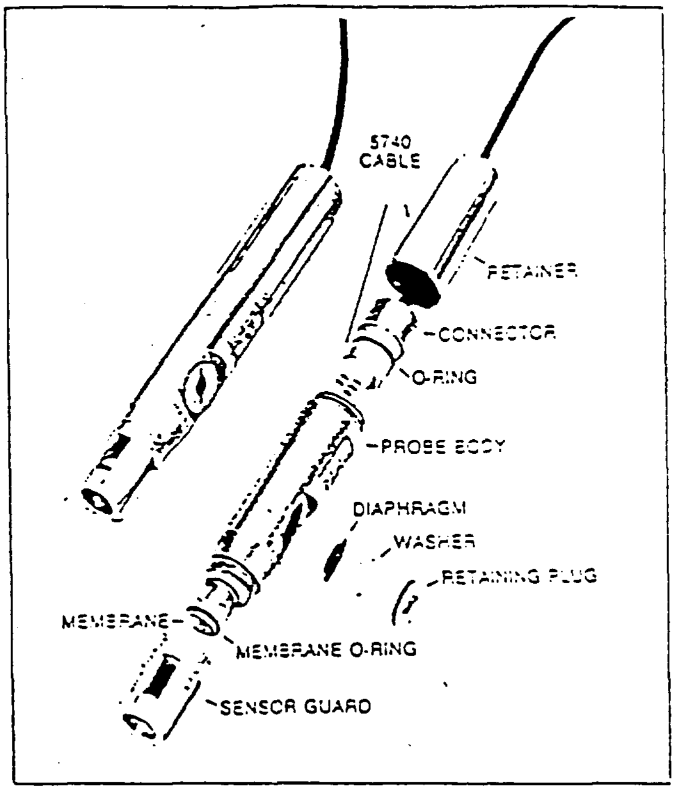

Figure 2. The YSI 5739 Probe

YSI 5750 BOD BOTTLE PROBE

The 5750 (Figure 3) is similar to the 5720A except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer.

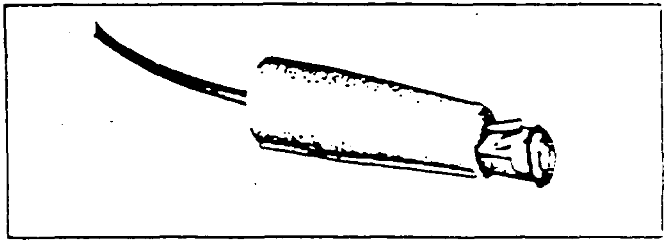

Figure 3. The YSI 5750 Probe

PROBE PREPARATION

All probes are shipped dry. You must follow these instructions when preparing a new probe or when changing membranes. Prepare the electrolyte by dissolving the KCl crystals which are supplied in a dropper bottle that should be filled to the neck with distilled water and shaken until the crystals are dissolved.

1. Unscrew the sensor guard (5739 only). Remove the O-ring and membrane, then thoroughly rinse the sensor with distilled water.

2. To fill the probe with electrolyte and install a new membrane, follow these steps:

a. Grasp the probe in your left hand. (See the sketches in Figure 4.) When preparing the 5739 probe, the pressure compensating port should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or a similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. For ease in preparing the 5720A, the stirring rod should be to the left. When preparing the 5720A or 5750 probes, simply fill the sensor body until no more air bubbles appear.

b. Secure a membrane between your left thumb and the probe body. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, touching it at the ends only.

c. With the thumb and forefinger of your other hand, grasp the free end of the membrane.

d. With a continuous motion, stretch it up, over and down the other side of the sensor. Stretching forms the membrane to the contour of the probe.

e. Secure the end of the membrane under the forefinger of your left hand while holding the probe.

f. Roll the O-ring over the end of the probe, being careful not to touch the membrane surface. For the 5720A, start at the right side of the sensor and roll the O-ring toward the stirring rod. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the O-ring.

g. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

3. Shake off excess KCl. On the 5739, reinstall the sensor guard.

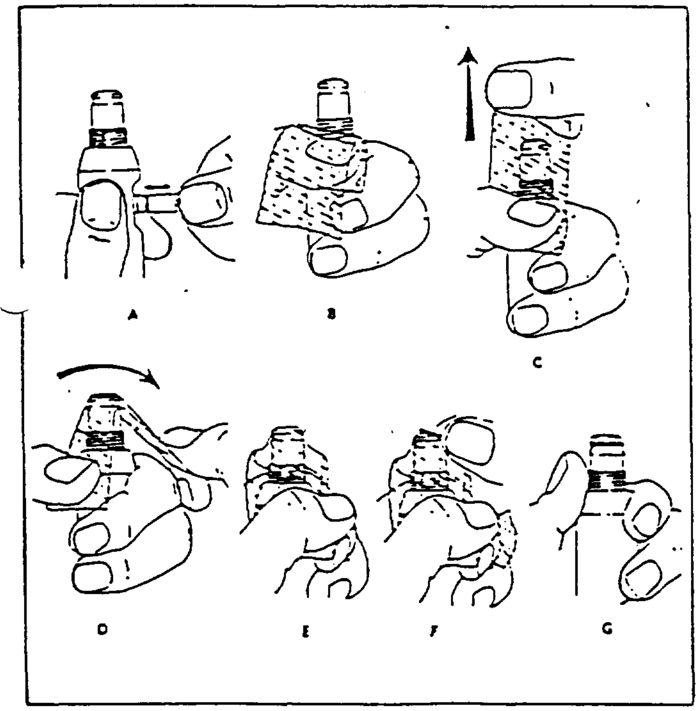

Figure 4. Membrane Application

Probe Storage

A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The 5720A and 5750 probes can be stored in a BOD bottle containing at least 1" of water.

OPERATING PRECAUTIONS, ALL PROBES

1. Membrane life depends on use. Membranes will last a long time if installed properly and treated with care during use. Erratic readings result from loose, wrinkled or fouled membranes, or from large bubbles in the electrolyte reservoir. If erratic readings, or evidence of membrane damage occur, you should replace the membrane and KCL. The average replacement interval is two to four weeks; electrolyte in constant or heavy use will be exhausted in about two weeks.

If the sensor O-ring on any probe is worn or loose, replace it with the O-ring provided in the YSI 5945 O-ring Pack.

2. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), it needs to have its surface restored. Probes may either be returned to the factory, or cleaned with the YSI 5680 Probe Reconditioning Kit; never use chemicals or any abrasive not supplied with this kit.

3. It is also possible that the silver anode may become contaminated, which will prevent successful calibration. Try soaking the probe overnight in a 3% ammonia solution; rinse with deionized water, recharge with electrolyte, and install a new membrane. If still unable to calibrate after several hours, return the probe for service.

4. Hydrogen sulfide, sulfur dioxide, halogens, and neon are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause.

These gases have been tested for response:

100% Carbon Monoxide	less than 1%
100% Carbon Dioxide	around 1%
100% Hydrogen	less than 1%
100% Chlorine	2/3 O_2 response
100% Helium	none
100% Nitrous Oxide	1/3 O_2 response
100% Ethylene	none
100% Nitric Oxide	1/3 O_2 response

5. The correct liquid level in BOD bottles is achieved by overfilling, then inserting a stopper and pouring off the excess. When using a YSI 5760 or a 5720A probe in a filled BOD bottle, be careful to insert it slowly to avoid sample overflow.

6. When using the 5720A in samples containing heavy particulate solids, additional stirring may be needed. Inverting the stoppered bottle immediately before use will usually provide adequate mixing.

CALIBRATION

Daily calibration is generally appropriate. Calibration can be disturbed by physical shock, touching the membrane, fouling of the membrane or drying out of the electrolyte. Check calibration after each series of measurements, and in time you will develop a realistic schedule for recalibration. When probes are not in use, store them as recommended in Probe Preparation.

Probes may be calibrated by Winkler Titration or by the Water Saturated Air method. Experience has shown that air calibration is quite reliable, yet far simpler than titration. Both methods are described here. Consult the manual for your particular instrument for more complete instructions.

Winkler Titration

1. Draw a volume of water from a single source and carefully divide it into four samples. Determine the oxygen in three of the samples using the Winkler Titration technique and average the three values. If one of the values differs from the other two by more than 0.5 mg/L, discard it and average the two values remaining.

2. Using the probe-meter system you are calibrating, place the probe into the fourth sample and stir.

3. Switch to the desired mg/L range and adjust the CALIBRATION control to the average value determined in step 1. Allow the probe to remain in the sample for at least 5 minutes before setting the calibration value, then leave it in the sample for an additional two minutes to verify stability. Readjust if necessary.

Air Calibration

1. Place the probe in a BOD bottle containing about 1 inch of water. Wait approximately ten minutes for temperature stabilization.
The 5739 probe can be placed in the YSI 5075A Calibration Chamber or in the small calibration bottle supplied with the probe (the one with the hole in the bottom) along with a few drops of water, or a moistened towel or cloth.

2. Read the temperature and refer to the instrument Calibration Table to determine the calibration value. NOTE: To achieve the stated accuracy of measurement, the probe must be stabilized before calibrating. The calibration temperature should be within 5 degrees of the sample temperature.

3. Determine the atmospheric correction factor (see Instrument instructions).

4. Multiply the calibration value by the correction factor.

5. Switch your instrument to an appropriate mg/L range and adjust the CALIBRATE control until the meter reads the corrected calibration value from step 4. Without changing the calibration setup, monitor the readings for an additional 3 minutes to verify calibration stability. Readjust if necessary.

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer. Electrode cleaning is not covered by warranty.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing on all its products. This warranty is limited to repair or replacement (YSI's option) at no charge.

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YSI Inc,
attn: Product Service
Billing address
name & ph no.
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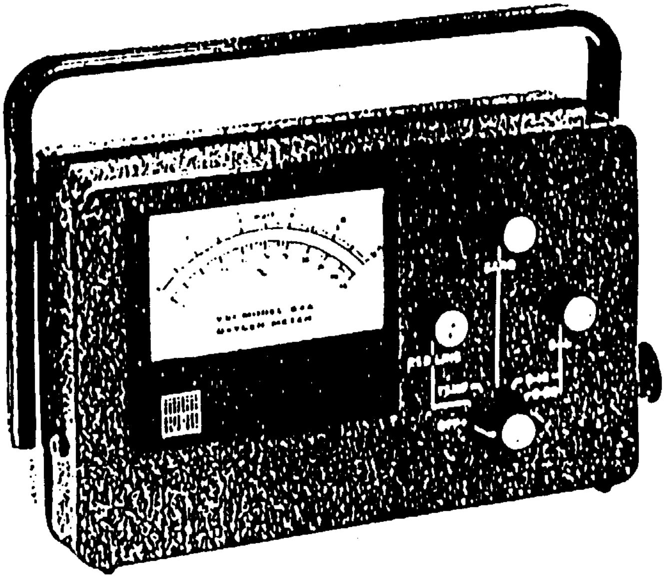
ITEM 066001 P/N A05467AJ FEBRUARY 1986

INSTRUCTION MANUAL
YSI MODELS 54ARC AND 54ABP
DISSOLVED OXYGEN METERS



Scientific Division
Yellow Springs Instrument Co., Inc.
Yellow Springs, Ohio 45387, U.S.A. • Phone 513-767-7241

PRICE INCLUDING HANDLING $5.00

SUMMARY OF OPERATING INSTRUCTIONS

1. CALIBRATION

A. Switch instrument to OFF and adjust meter mechanical zero.

B. Switch to RED LINE and adjust.

C. Prepare probe for operation, connect to instrument, wait up to 15 minutes for probe to stabilize. Probe can be in calibration chamber or ambient air.

D. Switch to ZERO and adjust to "0" on mg/l scale.

E. Switch to TEMP and read on °C scale.

F. Use probe temperature and true local atmospheric pressure (or feet above sea level) to determine calibration values from Tables I and II. (See pages 14 and 15).

EXAMPLE: Probe temperature = 21°C; Altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the altitude factor for 1000 feet is approximately .96. The correct calibration value, then, is:

8.9 mg/l X .96 factor = 8.54 mg/l

G. Switch to 0-10 or 0-20 mg/l range and adjust meter with CAL control to calibration value determined in Step F.

NOTE: It is desirable to calibrate probe in a high humidity environment. (See calibration section for more detail).

2. MEASUREMENT

A. Place probe in sample and stir.

B. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.

C. Read dissolved oxygen on appropriate range (1-10 or 0-20 mg/l)

D. We recommend the instrument be left on between measurements to avoid the necessity to repolarize the probe.

3. GENERAL CARE

A. Recharge batteries in the YSI Model 54ARC when the instrument can no longer be red lined. Recharge 16-20 hours. Replace with Burgess CD-6 or equivalent. Replace batteries in the YSI Model 54ABP when red line cannot be set with Panasonic UM-2N or equivalent.

B. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. Probe should be stored in humid environment to prevent drying out.

C. Calibrate daily.

TABLE OF CONTENTS

GENERAL DESCRIPTION

The YSI Models 54ARC and 54ABP Dissolved Oxygen Meters are intended for dissolved oxygen and temperature measurement in water and wastewater applications, but are also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/l (milligrams per liter) on 0-10 and 0-20 mg/l scales. Temperature is indicated in °C on a -5° to +45°C scale. Both dissolved oxygen ranges are automatically temperature compensated for solubility of oxygen in water and permeability of the probe membrane.

The probes use Clark-type membrane covered polarographic sensors with built-in thermistors for temperature measurement and compensation. A thin, permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

Power to operate the system is provided by internal batteries in the instruments, rechargeable batteries in the YSI Model 54ARC and disposable batteries in the YSI Model 54ABP.

SPECIFICATIONS

I. Instrument

Oxygen Measurement

Ranges: 0-10 and 0-20 mg/l (0-5 and 0-10 mg/l with YSI 5776 High Sensitivity Membrane)

Accuracy: ±1% of full scale at calibration temperature (±0.1 mg/l and 0-10 scale).

Readability: .05 mg/l on 0-10 scale; 0.1 mg/l on 0-20 scale.

Temperature Measurement

Ranges: -5° to +45°C

Accuracy: ± 0.7°C, including probe

Readability: 0.25°C

Temperature Compensation

± 1% *of D.O. reading for measurements made within* ± 5°C of calibration temperature.

± 3% of D.O. reading over entire range of -5 to +45°C Probe temperature.

System Response Time

Typical response for temperature and D.O. readings is 90% in 10 seconds at constant temperature of 30°C with YSI 5775 Membranes. D.O. response at low temperature and low D.O. is typically 90% in 30 seconds. YSI 5776 High Sensitivity Membranes can be used to improve response at low temperature and low D.O. concentrations. If response time under any operating conditions exceeds two minutes, probe service is indicated.

Operating Temperature Range

Instrument and probe operating range is -2° to +45°C. Large ambient temperature changes will result in 2% loss of accuracy unless Red Line and Zero are reset.

Recorder Output

0 to 114-136 mV. Recorder should have 50,000 ohms minimum input impedance.

Power Supply

YSI Model 54ABP: (4) 1.5 volt carbon zinc batteries provide approximately 1000 hours operation. Replace with Panasonic UM-2N or equal.

YSI Model 54ARC: (4) 1.25 volt Ni-Cad rechargeable cells (Burgess CD-6 or equal) provide approximately 100 hours of operation between charges.

II. Probe

Cathode: Gold
Anode: Silver
Membrane: .001" FEP Teflon (.0005" FEP Teflon available)
Electrolyte: Half Saturated KCl
Temperature Compensation: (See SPECIFICATIONS, I. Instrument)
Pressure Compensation: Effective 1/2% of reading to pressures of 100 psi (230 ft. water)
Polarizing Voltage: 0.8 volts nominal
Probe Current: Air at 30°C = 19 microamps nominal
Nitrogen at 30°C = .15 microamps or less

III. Accessories and Replacement Parts

YSI 5720A — Self Stirring B.O.D. Bottle Probe
YSI 5750 — Non Stirring B.O.D. Bottle Probe
YSI 5739 — Oxygen Temperature Probe for field use. Combine with one *of the following cables for desired lead length:*
YSI 5401 — Battery Charger Eliminator 115V
YSI 5402 — Battery Charger Eliminator 230V

Detachable leads for use with YSI 5739:

YSI 5740-10	10' cable
YSI 5740-25	25' cable
YSI 5740-50	50' cable
YSI 5740-100	100' cable
YSI 5740-150	150' cable
YSI 5740-200	200' cable

YSI 5492A — Battery Pack Operates YSI 5791A and 5795A Submersible Stirrers

YSI 5791A — Submersible Stirrer for field use

YSI 5795A — Submersible Stirrer for field use

YSI 5075A — Calibration Chamber for use with field probe

YSI 5890 — Carrying Case

YSI 5775 — Membrane and KCl Kit, Standard — includes 2 each 15-membrane packets (.001" thick standard membranes) and a 30 ml bottle KCl with Kodak Photo Flo.

YSI 5776 — Membrane and KCl Kit, High Sensitivity — includes 2 each 15-membrane packets (.0005" thick membranes) and a 30 ml bottle KCl with Kodak Photo Flo.

YSI 5945 — "O" Ring Pack — includes (6) "O" rings for each YSI D.O. Probe.

YSI 5486 — Beater Boot Kit — includes (1) A-05486 Boot, (1) A-05484 Tip, (2) A-05485 Spring. Used only on 5720A and discontinued 5420A and 5720.

YSI 5986 — Diaphragm Kit for use only with YSI 5739 D.O. Probe.

YSI 5734 — Adaptor makes it possible to use discontinued YSI 5400 Series Probes with YSI Models 54ARC and 54ABP.

YSI 5735 — Adaptor makes it possible to use YSI 5739, 5720A and 5750 Probes with discontinued YSI Models 54RC and 54BP.

OXYGEN PROBES AND EQUIPMENT

There are three oxygen probes for use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. Descriptions of where they are used are contained in the following paragraphs.

I. YSI 5739 D.O. Probe

The YSI 5739 probe, with built-in lead weight and pressure compensation, is an improved design that replaces the discontinued YSI 5418, 5419, 5718 and 5719 probes. (See Figure 1)

For user convenience the probe is equipped with a disconnecting cable to facilitate changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retaining nut. The connection is *not* designed for casual disconnection and should only be disconnected when necessary.

To disconnect the cable unscrew the retaining nut and slide it down the cable to expose the connector. Pull gently on the cable and connector until the connector comes away from the probe body.

To reassemble, inspect the connector and "O" ring for cleanliness. If the "O" ring is frayed or damaged remove it by squeezing it in the groove causing it to bulge, then roll it out of the groove and off the connector. A replacement "O" ring is supplied with the cable.

Push the connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves which may cause them to spring apart slightly, is normal. Screw on the retaining nut, *hand tight only*. NOTE: If erratic readings are experienced, disconnect the cable and inspect for water. If present, dry out and reconnect, replacing the "O" ring, if necessary.

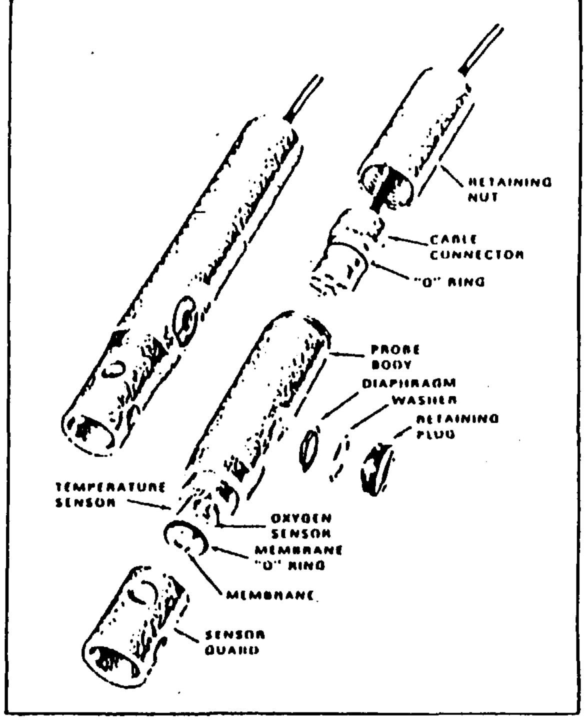


Figure 1

Pressure Compensation

The vent on the side of the probe is part of a unique pressure compensating system that helps assure accurate readings at great depths of water. Pressure compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft. water). The quantity of air bubbles trapped under the membrane determines how serious the pressure error will be, which is why proper preparation of the probe is essential. (See OPERATING PROCEDURES.) The system is designed to accommodate a small amount of trapped air and still function properly, but the amount should be kept to a minimum

The compensating system normally does not require servicing and should not be taken apart. However, if electrolyte is leaking through the diaphragm or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Using a coin unscrew the retaining plug and remove the washer and the diaphragm, flush any salt crystals from the reservoir, install the new diaphragm (convolution side in), replace the washer, and screw in the retaining plug.

II. YSI 5720A B.O.D. Bottle Probe

The YSI 5720A B.O.D. Bottle Probe replaces the discontinued YSI 5420A B.O.D. Bottle Probe for measuring dissolved oxygen and temperature in standard B.O.D. bottles. It is provided with an agitator for stirring the sample solution, available in models for 117VAC (95-135VAC, 50-60 Hz) or 230VAC (190-250VAC, 50-60 Hz) operation. (See Figure 2)

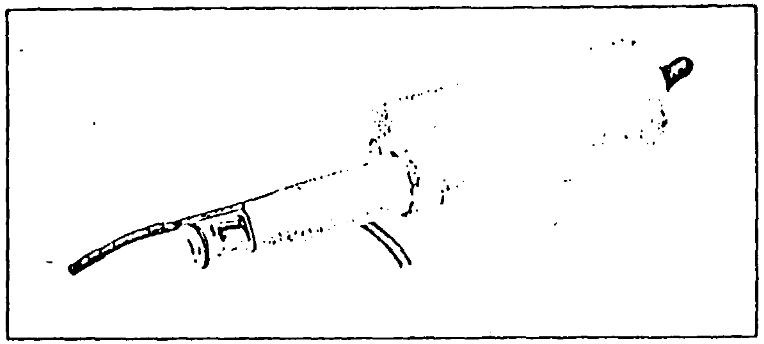


Figure 2

When using the probe, plug the agitator power supply into line power and the probe plug into the instrument. With the agitator turned off place the tapered probe end into the B.O.D. bottle and switch agitator "ON" with switch on top of probe. The probe should be operated with a minimum of trapped air in the B.O.D. bottle. A slight amount of air in the unstirred region at the top of the bottle may be neglected, but no bubbles should be around the thermistor or oxygen sensor.

Stirrer Boot

The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage likely to allow leaking into the motor housing, the boot must be replaced.

In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot after use in contaminated samples. (See Figure 3)

Boot replacement is as follows:

1. Pull off old assembly and clean shaft.
2. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
3. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

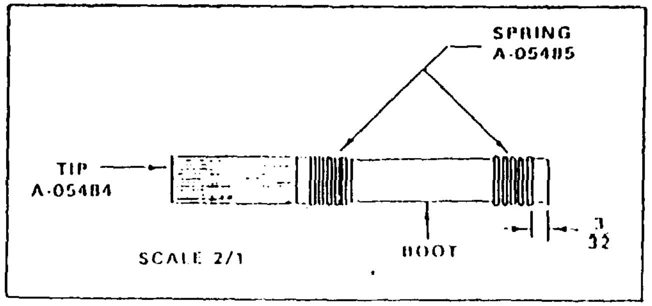


Figure 3

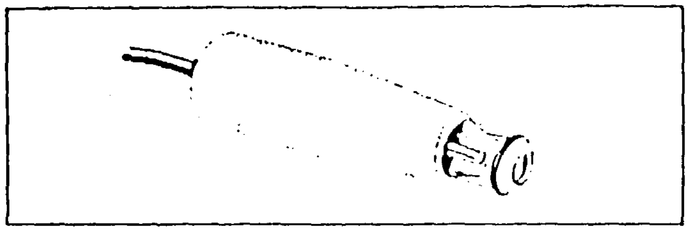


Figure 4

III. YSI 5750 B.O.D. Bottle Probe

The YSI 5750 B.O.D. Bottle Probe replaces the discontinued YSI 5450 B.O.D. Bottle Probe. It is similar to the YSI 5720A B.O.D. Bottle Probe, except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 4)

IV. Cable Adaptors

All YSI 5700 Series Probes are designed for direct use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. However, to use YSI 5700 probes with the discontinued YSI Models 54RC and 54BP, cable adaptor YSI 5735 is required.

V. YSI 5791A and 5795A Submersible Stirrers

The YSI submersible stirrers are accessories that perform the function of stirring the sample being studied when making dissolved oxygen measurements in the field. The YSI 5791A stirrer can be used with the following dissolved oxygen probes: YSI 5418, 5419, 5718, 5719, and 5739. The YSI 5795A stirrer is only for use with the YSI 5739 Probe. (See Figure 6)

When a stirrer and probe are assembled, the stirrer agitates the sample directly in front of the sensor by means of a rotating eccentric weight which causes the spring-mounted hermetically sealed motor housing to vibrate. An impeller on the end of the motor housing flushes the media across the oxygen sensor. (See sales literature and instruction sheets for further information).

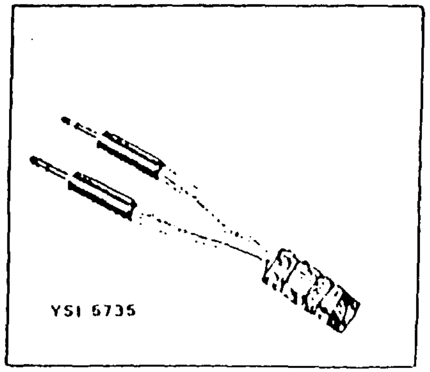


Figure 5

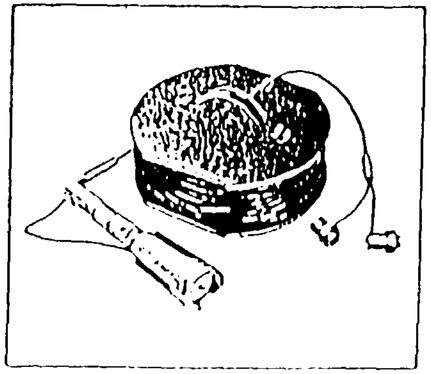


Figure 6

VI. YSI 5492A Battery Pack

The YSI 5492A Battery Pack is designed to attach to the case of all YSI Model 54 Dissolved Oxygen Meters to provide power for operating the submersible stirrers. (See sales literature and instruction sheets for further information).

OPERATING PROCEDURES

I. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probes as follows. (See Figure 7)

ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS

1. Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle with distilled water. Fill the bottle to the top.
2. Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.
3. Fill the probe with electrolyte as follows:
   A. Grasp the probe in your left hand. When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes, simply fill the sensor body until no more air bubbles appear.
   B. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.
   C. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
   D. Using a continuous motion *stretch* the membrane *UP, OVER,* and *DOWN* the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to *approximately 1-1/2 times its normal length.*
   E. Secure the end of the membrane under the forefinger of the hand holding the probe.
   F. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.
   G. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
4. Shake off excess KCl and reinstall the sensor guard.
5. A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe [illegible] from drying out. The YSI 5720A and 5750 probes can be stored in a B.O.D. bottle containing about 1" of water.
6. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. However, should the electrolyte be allowed to evaporate and an excessive amount of bubbles form under the membrane, or the membrane become damaged, thoroughly flush the reservoir with KCl and install a new membrane.
7. Also replace the membrane if erratic readings are observed or calibration is not stable.
8. "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCl and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.
9. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or any abrasive.
10. H_2S, SO_2, Halogens, Neon, Nitrous Oxide and CO are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause. These gases have been tested for response.

100% Carbon Monoxide-Less than 1%
100% Carbon Dioxide-Around 1%
100% Hydrogen-Less than 1%
100% Chlorine-2/3 O_2 response
100% Helium-none
100% Nitrous Oxide-1/3 O_2 response
100% Ethylene-none
100% Nitric Oxide-1/3 O_2 response

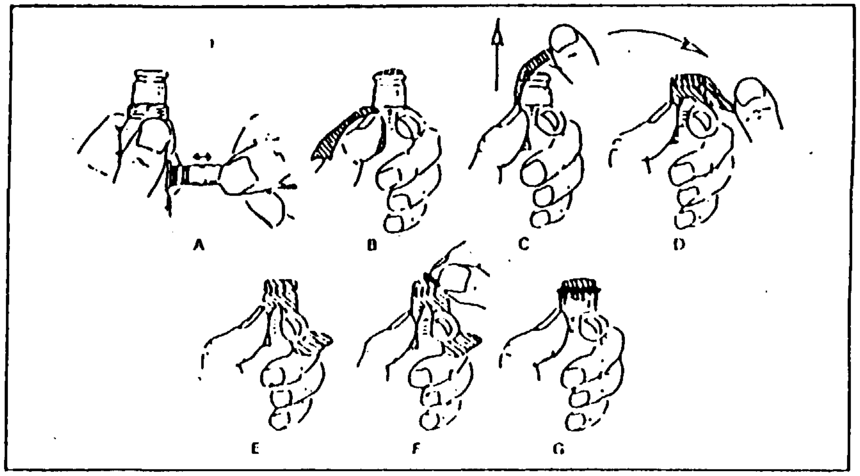


Figure 7

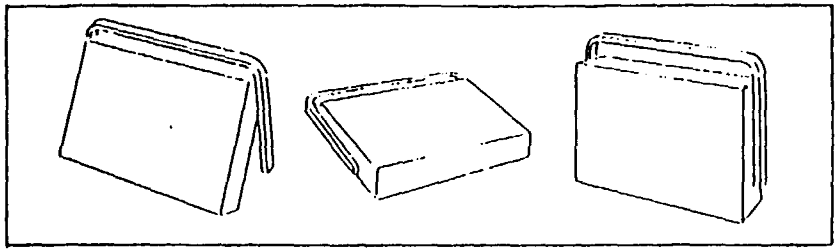


Figure 8

II. Preparing the Instrument

It is important that the instrument be placed in the intended operating position vertical, tilted, or on its back — before it is prepared for use and calibrated. (See Figure 8). Readjustment may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed.
2. Switch to RED LINE and adjust the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.
3. Switch to ZERO and adjust to zero with zero control knob.
4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
5. Before calibrating allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been OFF or the probe has been disconnected.

III. Calibration

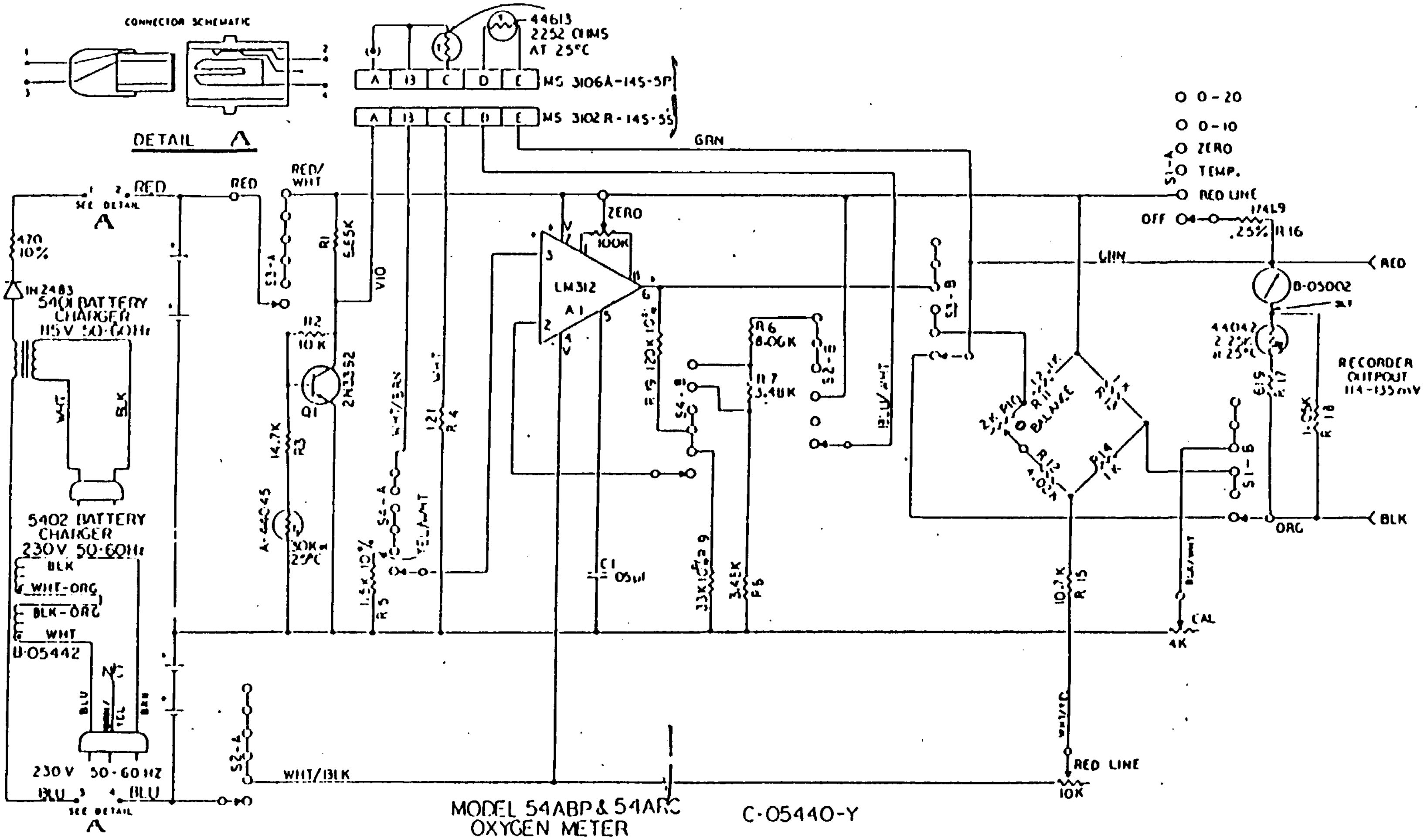
The operator has a choice of three calibration methods — Winkler Titration, Saturated Water, and Air. Experience has shown that air calibration is quite reliable, yet far simpler than the other two methods. The three methods are described in the following paragraphs.

Winkler Titration

1. Draw a volume of water from a common source and carefully divide into four samples. Determine the oxygen in three samples using the Winkler Titration technique and average the three values. If one of the values differs from the other 2 by more than 0.5 mg/l, discard that value and average the remaining two.
2. Place the probe in the fourth sample and stir.
3. Switch to desired mg/l range and adjust the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability. (Readjust if necessary).

Saturated Water

1. Air saturate a volume of water (300-500 cc) by aerating or stirring for at least 15 minutes at a relatively constant temperature.



MODEL 54ABP & 54ARC
OXYGEN METER

C-05440-Y

GENERAL NOTES:

1. ALL RESISTOR VALUES ARE IN OHMS; K=1,000, M=1,000,000. UNLESS OTHERWISE SPECIFIED, RESISTORS ARE 1/4W, 1% METAL FILM.

2. FOR 54ABP BATTERIES ARE 4 EA 1.5V PANASONIC UM-2N OR EQUIV.
FOR 54ARC BATTERIES ARE 4 EA 1.25V BURGESS CD6 OR EQUIV. NICads.

3. BATTERY CHARGERS AND CONNECTOR APPLY TO 54ARC VERSION ONLY.

4. THE VALUES SHOWN ON THE SCHEMATIC MAY DIFFER FROM THOSE IN THE INSTRUMENT; IF SO, EITHER VALUE CAN BE USED FOR REPLACEMENT PURPOSES.

2. Place the probe in the sample and stir. Switch to TEMPERATURE. Refer to Calibration Table I for the mg/l value corresponding to the temperature.
3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.
4. Multiply the mg/l value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is 8.9 mg/l X 0.96 = 8.54 mg/l.
5. Switch to an appropriate mg/l range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

Air Calibration — Fresh Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing CALIBRATION CHAMBER) or the small calibration bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.
2. Switch to TEMPERATURE and read. Refer to Table I — Solubility of Oxygen in Fresh Water, and determine calibration value.
3. Determine altitude or atmospheric correction factor using Table II.
4. Multiply the calibration value from Table I by the correction factor from Table II.

   EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. Therefore, the corrected calibration value is 8.9 mg/l X 0.96 = 8.54 mg/l.
5. Switch to the appropriate mg/l range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability.

   Readjust if necessary.

Air Calibration — Sea Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing Calibration Chamber) or the small storage bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is polarizing

2. Switch to TEMPERATURE and read. Refer to Table III — Solubility of Oxygen in Sea Water, and determine calibration value.
3. Switch to the appropriate mg/l range, and adjust the CALIBRATE knob until the meter reads the calibration value determined in Step 2. Wait 2 minutes to verify calibration stability. Readjust if necessary.

The probe is now calibrated and should hold this calibration value for many measurements. Calibration can be disturbed by physical shock, touching the membrane, or drying out of the electrolyte. Check calibration after each series of measurements and in time you will develop a realistic schedule for recalibration. For best results when not in use, follow the storage procedures recommended for the various probes described under OXYGEN PROBES AND EQUIPMENT. This will reduce drying out and the need to change membranes.

Calibration Chamber

The YSI 5075A Calibration Chamber is an accessory that helps obtain optimum calibration in the field and is also a useful tool for measuring at shallow depths (less than 4').

As shown in Figure (A), it consists of a 4-1/2 foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), and two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) as shown in Figure (B). Place the probe in the measuring ring, Figure (C), and immerse the probe in the sample to be measured for five minutes to thermally equilibrate the probe. Quickly transfer the probe to the calibration chamber (5) draining excess water from the chamber and shaking any excess droplets from the probe membrane. For maximum accuracy, wet the inside of the calibration chamber with fresh water. This creates a 100% relative humidity environment for calibration. Place the chamber in the sample for an additional five minutes for final thermal equilibrium. Calibrate the probe as described in the air-calibration procedure. Keep the handle above water at all times.

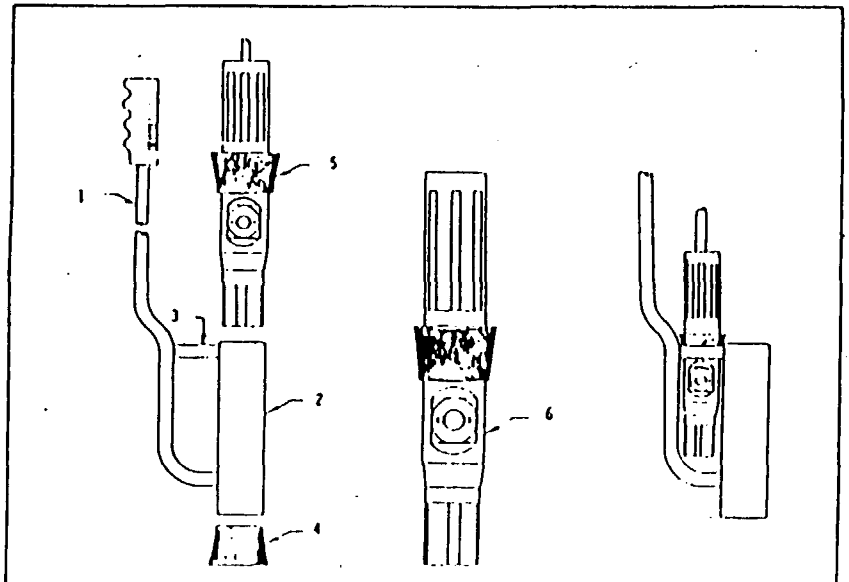


Figure A Figure B Figure C

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5'/second) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.

IV. Dissolved Oxygen Measurement

With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and provide stirring.

1. Stirring for the YSI 5739 Probe can best be accomplished with a YSI submersible stirrer. If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft. per second. If the 5075 Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft. per second.
2. The YSI 5720A has a built-in power driven stirrer.
3. With the YSI 5750 sample stirring must be accomplished by other means, such as with the use of a magnetic stirring bar.
4. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
5. Read dissolved oxygen.

V. High Sensitivity Membrane

Use of high sensitivity .0005" membranes (YSI 5776) in place of standard .001" membrane (YSI 5775) is recommended when measurements are to be made consistently at low temperatures (less than 15°C). Calibration and readings will be made just as if the standard YSI 5775 Membrane was being used.

The YSI 5776 High Sensitivity Membranes can also be used in certain situations to increase sensitivity at temperatures above 15°C. The ranges thus become 0-5 and 0-10 mg/l. When calibration with high sensitivity membranes is attempted at temperatures greater than 15°C the selector switch must be set to 0-20 mg/l. Multiply the calculated calibration value by 2. For example: at 21°C and 1000 ft. altitude the calibration value would be 8.6 x 2 or 17.2. Remember the 0-10 and 0-20 mg/l ranges are now 0-5 and 0-10 mg/l, and all mg/l readings must be divided by 2 for a final reading. When operating in this manner accuracy will be degraded slightly.

VI. Recorder Output

Red and black recorder jacks are provided on the YSI Models 54ARC and 54ABP, if you wish to record data while measuring. The high terminal of the recorder is connected to the red tip jack and the low terminal to the black. Output of the YSI 54A at full scale is between 114 to 136 mV.

Use a 50K or higher input impedance recorder and operate it with the terminals ungrounded. The recorder should be operated with its terminals ungrounded. Calibration of the instrument should be checked after connection of

Many recorders have an adjustable full scale sensitivity feature. When these recorders are used with the Model 54A, use the 100 millivolt range and adjust the full scale chart deflection when there is full scale meter deflection. Refer to the instruction book for the recorder. For recorders without this feature, a simple divider network as shown below can be constructed. This is adequate to adjust the signal for full scale chart and meter deflection on the 100 mV fixed range recorders.

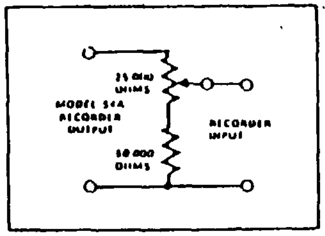


Figure 10

VIII. Calibration Tables

Table I shows the amount of oxygen in mg/l that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

Table I — Solubility of Oxygen in Fresh Water

Temperature °C	mg/l Dissolved Oxygen	Temperature °C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater."

Table II — Correction for Atmospheric Pressure

Table II shows the correction factor that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

Table II

Atmospheric Pressure mmHg	or	Equivalent Altitude Ft.	=	Correction Factor
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

The temperature-solubility relationship of oxygen in sea water is not the same as that in fresh water. For this reason the compensation error when used with sea water is greater than when used with fresh water. For a ±5°C span the error could be +2.2% of reading and over the temperature range of -2° to +30°C the error could be 6.3% of reading.

Table III — Solubility of Oxygen in Sea Water

SOLUBILITY OF OXYGEN IN SEA WATER
(Chloride concentration 20,000 mg/1)

Temp. °C	Solubility mg/l	Temp. °C	Solubility mg/l
0	11.41	16	7.91
1	11.11	17	7.78
2	10.83	18	7.61
3	10.56	19	7.47
4	10.30	20	7.33
5	10.05	21	7.20
6	9.82	22	7.07
7	9.59	23	6.95
8	9.37	24	6.83
9	9.16	25	6.71
10	8.96	26	6.60
11	8.77	27	6.49
12	8.58	28	6.38
13	8.41	29	6.28
14	8.24	30	6.18
15	8.07		

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

Correcting for Salinity

When measuring dissolved oxygen in water samples with a salinity or chlorinity between sea water and fresh water, calibrate the instrument for fresh water and make your measurements. Then correct the data according to the following formula:

FORMULA:

$$A = M\left[1.0 - \left(\frac{(Cs/Co\,[Sf - So])}{Sf}\right)\right]$$

Where: A = Actual DO of sample. (mg/l dissolved O_2)
M = Measured DO with instrument
*Co = Chlorinity of ocean water (20 o/oo Cl ion)
*Cs = Chlorinity of sample (o/oo Cl ion)
Sf = DO of saturated fresh water at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from charts in instruction manual)
So = DO of saturated ocean water (20,000 mg/l Chloride ion) at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from instruction manual)

*NOTE: *If salinity is used instead of chlorinity the ratio Cs/Co is computed using 36.11 o/oo for Co (salinity of ocean water), and the salinity of your sample of Cs.*

EXAMPLE: Measured Data
DO = 4.1
Temp = 22°C
Salinity = 31 o/oo salinity
M = 4.1 mg/l DO from data
Co = 36.11 o/oo salinity from manual
Cs = 31.0 o/oo salinity from data
Sf = 8.8 mg/l DO from Table I in manual
So = 7.1 mg/l DO from Table II in manual

$$\begin{aligned}
A &= 4.1\left[1.0 - \left(\frac{([31.0/36.11]\ [8.8 - 7.1])}{8.8}\right)\right] \\
&= 4.1\left[1.0 - \left(\frac{([.86]\ [1.7])}{8.8}\right)\right] \\
&= (1.46) \\
&= 4.1\left[1.0 - \left(\frac{(1.46)}{8.8}\right)\right] \\
&= 4.1\,[1.0 - 0.166] \\
&= 4.1\,[0.834] \\
&= 3.41 \text{ mg/l}
\end{aligned}$$

DISCUSSION OF MEASUREMENT ERRORS

There are three basic types of errors which can occur. Type I errors are related to limitations of the instrument design and tolerances of the instrument components. These are chiefly the meter linearity and resistor tolerances. Type II errors are due to basic probe accuracy tolerances, chiefly background signal, probe linearity, and variations in membrane temperature coefficient. Type III errors are related to the operator's ability to determine the conditions at the time of calibration. If calibration is performed against more accurately known conditions, Type III errors are appropriately reduced.

Individual Sources of Error

This description of sources of error can be used to attach a confidence to any particular reading of dissolved oxygen. The particular example given is for a near extreme set of conditions. As a generality, overall error is diminished when the probe and instrument are calibrated under conditions of temperature and dissolved oxygen which closely match the sample temperature and dissolved oxygen.

Type I

A — is the error due to meter linearity
Error = +1% full scale of the measurement range.

B — is the error due to tolerances in the instrument when transferring a reading from one range to another. Error = ± 1% of the reading.

Type II

A — errors due to probe background current

$$\text{Error} = 1.0\% \left(1 - \left(\frac{\text{Meter Reading mg/l}}{\text{Calibration Value mg/l}}\right) \times \text{Calib. Value, mg/l}\right.$$

B — errors due to probe non-linearity. Error = ± 0.3% of reading.

C — error caused by variability in the probe membrane temperature coefficient.
Error = zero if readings are taken at the calibration temperature
Error = ± 1% of meter reading if readings are taken within 5°C of the calibration temperature.
Error = ± 3% of meter reading for all other conditions.

Type III

A — errors due to the accuracy of the instrument thermometer when used to measure the exact probe temperature during calibration.
Error = ± 1.5% of reading.

B — errors due to the assumption of mean barometric pressure.
Daily variation is usually less than 1.7%.
Error = ± 1.7% of reading.

C — errors assume an ability to estimate altitude to within ± 500 ft. when computing the altitude correction factor.
Error = ± 1.8% of reading.

D — errors consider the possibility of only 50% relative humidity when calibrating the probe. If the actual relative humidity is 50% instead of 100% the errors will be as follows:

Calibration Temperature ± C	Error in percent of reading
0	(-) 0 3
10	(-) 0.6
20	(-) 1.15
30	(-) 2 11
40	(-) 3.60

Example of a Typical Error Calculation

The example given presumes the air calibration technique. If calibration is done with air saturated water, the relative humidity consideration (III-D) is eliminated. If the Winkler calibration method is used, Type III errors are deleted and replaced by the uncertainty attributable to the overall Winkler determination.

Data: Instrument calibrated at 25°C, elevation estimated at 2000' ± 500', normal barometric pressure assumed, calibrated on 0-10 mg/l range at 7.8 mg/l, readings taken on 0-20 mg/l range at 10.5 mg/l at 8°C.

Type	Description		Calculations		Error mg/l
IA	Linearity	=	.01 X 10.5 mg/l	=	.10
IB	Range Change	=	.01 X 10.5 mg/l	=	.10
IIA	Probe Background	=	$.01 \times \left(1 - \frac{10.5}{7.8}\right) 7.8$ mg/l	=	.03
IIB	Probe Linearity	=	.003 X 10.5 mg/l	=	.03
IIC	Temp. Compensation	=	.03 X 10.5 mg/l	=	.31
IIIA	Temp. Measurement	=	.015 X 10.5 mg/l	=	.16
IIIB	Pressure	=	.017 X 10.5 mg/l	=	.18
IIIC	Altitude	=	.18 X 10.5 mg/l	=	.19
IIID	R.H.	=	.016 X 10.5 mg/l	=	.17
			Maximum Possible Error	=	1.27 mg/l
			Probable Error	=	± .63 mg/l

Considering a statistical treatment of the probable error at any time for any instrument, it is likely that the actual error in any measurement will be about 1/2 of the possible error. In this case the probable error is about ± .5 mg/l out of a reading of 10.5 mg/l, or 4.8% of the reading.

INSTRUMENT BATTERIES

Battery replacement or recharging on the YSI Model 54A is indicated if the "red line" adjustment cannot be made or O_2 calibration cannot be achieved. *(Warning: a faulty probe will also not permit O_2 calibration.)*

To replace batteries remove the four screws holding the rear cover of the instrument. The four batteries will be found on the battery terminal board inside. CAUTION: *disconnect battery charger on YSI Model 54ARC before removing cover.*

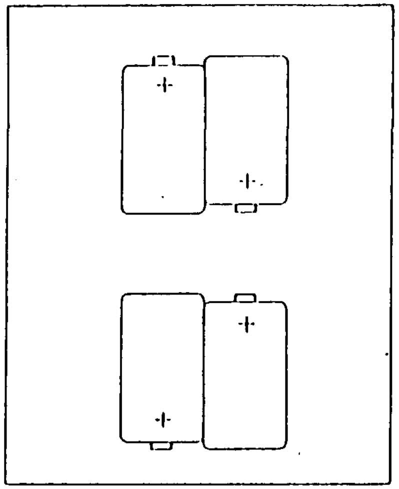


Figure 11

The YSI Model 54ARC contains four 1.25V Ni-Cd batteries (Burgess CD6 or equivalent). (See Figure 11). These batteries should be recharged when the instrument can no longer be red lined. Battery life should be three years or longer. Deeper discharge because of longer intervals between recharge will result in shorter battery life. The batteries should be recharged overnight, about 16 hours with the instrument off or 20 hours with the YSI Model 54ARC turned on.

The YSI Model 54ABP contains four 1.5V carbon-zinc (Panasonic UM-2N or equivalent). The life of these batteries is 1000 hours. Replace batteries every six months to minimize danger of corrosion due to dead or leaky batteries.

Battery holders are color coded. Positive (+ button) end of battery must go to red. (See Figure 11).

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing for all YSI products.

YELLOW SPRINGS INSTRUMENT CO., INC.
SERVICE DEPARTMENT
P.O. BOX 279
YELLOW SPRINGS, OHIO 45387, U.S.A.

PHONE: 513-767-7241

APPENDIX C-5

REDOX POTENTIAL

FIELD MEASUREMENT OF OXIDATION-REDUCTION POTENTIAL

Method: Electrometric

Reference: Beckman Instruments, 1987

Sensitivity: 1 mV

Optimum Range: -999.9 mV to +999.9 mV

Sample Handling: Determine on-site or within 4 hours

Reagents and Apparatus:

1. pH meter in absolute millivolt mode,
2. Platinum combination electrodes,
3. Beakers or plastic cups,
4. Certified pH buffer solutions, pH 4 and 7 saturated with a few crystals of quinhydrone,
5. Deionized water in squirt bottle.
6. All glassware soap and water washed, followed by two hot water rinses and two deionized water rinses.

Calibration:

1. Short the meter glass and reference inputs, and adjust the STANDARDIZE control until zero millivolts is displayed.
2. Place electrode in pH 4 buffer solution saturated with quinhydrone.

3. Record mV reading and compare to chart on Table 1.

4. Rinse electrode with deionized water and place in pH 7 buffer solution saturated with quinhydrone.

5. Record mV reading and compare to chart on Table 1.

6. If mV readings do not agree within ± 10 mV of the Table 1 values at the given temperature, follow electrode maintenance procedures described in the attached manual and recalibrate.

Procedure:

1. Calibrate meter using calibration procedure.

2. Pour the sample into a cleaner beaker or plastic cup.

3. Immerse electrode in solution allowing several minutes for meter to stabilize. Make sure the white AgCl junction on side of electrode is in the solution. The level of electrode solution must be approximately one inch above sample to be measured.

4. Rinse electrode with deionized water between samples. Recheck calibration with pH 4 buffer solution saturated with quinhydrone after every 5 samples.

Notes:

1. Eh is temperature and pH dependent. Therefore, the temperature and pH of samples should be measured at the same time as redox. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

2. Weak organic and inorganic salts and oil and grease are interferences in Eh measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, polish with scouring powder and rinse with distilled water. Then recalibrate meter.

3. Before going into the field:

   a. Report any problems;
   b. Do a quick calibration with quinhydrone saturated pH buffer solution to check electrode;
   c. Prepare fresh quinhydrone saturated pH 4 and pH 7 solutions daily.

4. Following field measurements:

   a. Report any problems;
   b. Compare with previous data;
   c. Clean all dirt off of meter and inside case;
   d. Store electrode as follows;

      1) Slide rubber sleeve into position over the filling hole.
      2) Place cot over tip of electrode by threading platinum wire through opening and sliding cot onto glass body until porous plug is completely covered.

JDD/jkk/CAW
[wpmisc-600-39]
60776.05

Table 1

Redox Potential Calibration Chart

Quinhydrone Saturated pH 4 Solution

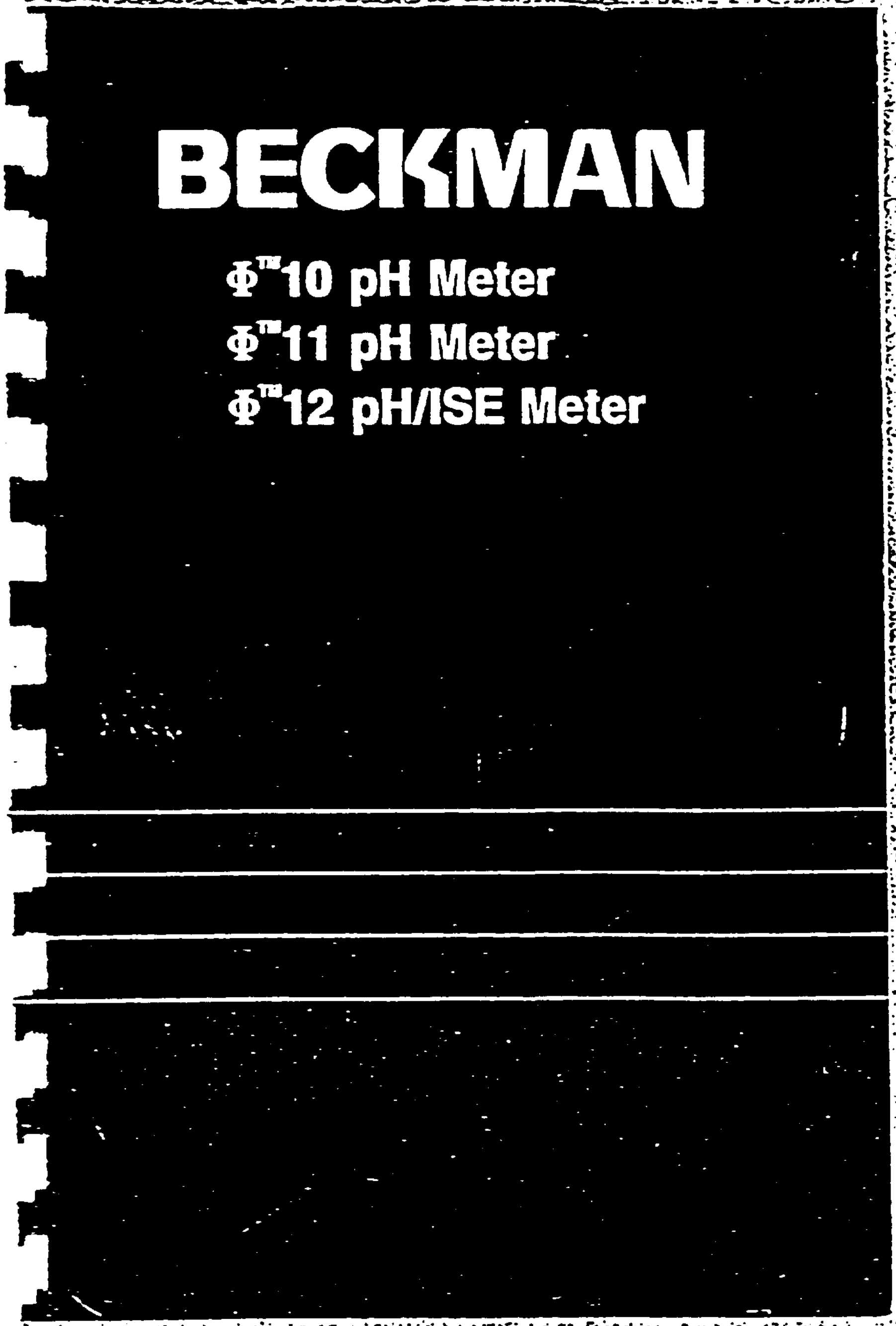
Temperature °C	20°	25°	30°
Theoretical Value (mV)	+268 mV	+263 mV	+258 mV

Quinhydrone Saturated pH 7 Solution

Temperature °C	20°	25°	30°
Theoretical Value (mV)	+92 mV	+86 mV	+79 mV

Instrument reading should be within ± 10 mV of Theoretical

[wpmisc-400-12]



BECKMAN

Φ™10 pH Meter
Φ™11 pH Meter
Φ™12 pH/ISE Meter

ASSC.

WARRANTY

Your Φ™ (pHI™) 10, 11, or 12 pH Meter is warranted to be free of manufacturing defects for one (1) year from the date of purchase. This does not include any defects that are the result of abuse or misuse of the instrument. Beckman Instruments, Inc., will, at Beckman's option, repair or replace your instrument with a comparable unit. This is a limited warranty. You may have additional rights under your state laws. Batteries are not included in this warranty.

WARNING: *This equipment generates, uses, and can radiate radio frequency energy and may cause interference to radio communications. Improper installation or modification of the equipment may increase interference. It has been tested and found to comply with the limits for a Class A computing device pursuant to Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference when operated in a commercial environment.*

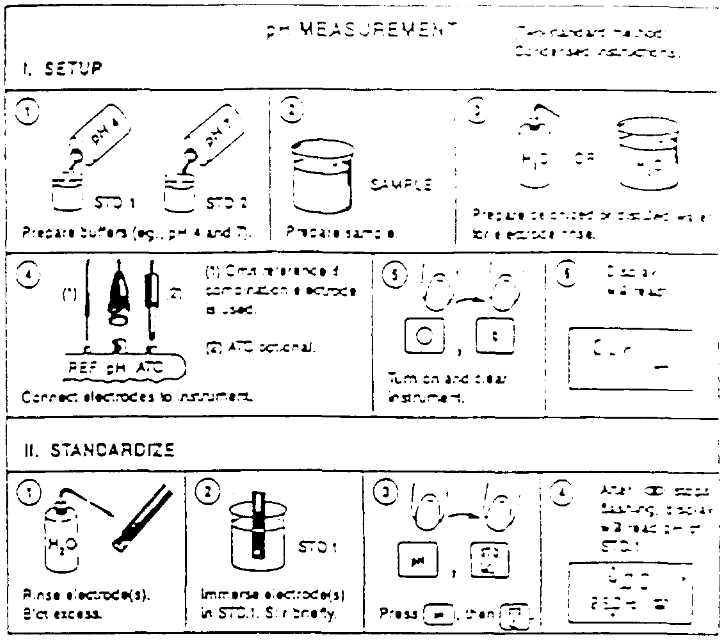
Operation of this equipment in a residential area may cause interference, in which case the user at his own expense will be required to take whatever measures may be required to correct the interference.

Beckman Instructions 015-246200-B

BECKMAN

Φ™10 pH Meter
Φ™11 pH Meter
Φ™12 pH/ISE Meter

Beckman Instruments, Inc. • Scientific Instruments Division • Fullerton, CA 92634-3100



pH MEASUREMENT

Two-standard method
Condensed instructions.

I. SETUP

① Prepare buffers (eg., pH 4 and 7).

② Prepare sample.

③ Prepare deionized or distilled water for electrode rinse.

④ Connect electrodes to instrument.

(1) Omit reference if combination electrode is used.

(2) ATC optional.

⑤ Turn on and clear instrument.

⑥ Display will read:

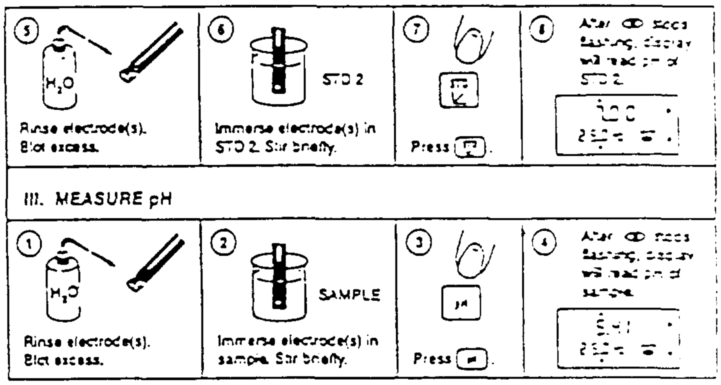
II. STANDARDIZE

① Rinse electrode(s). Blot excess.

② Immerse electrode(s) in STD 1. Stir briefly.

③ Press pH, then STD.

④ After ⊂⊃ stops flashing, display will read pH of STD 1.



⑤ Rinse electrode(s). Blot excess.

⑥ Immerse electrode(s) in STD 2. Stir briefly.

⑦ Press STD.

⑧ After ⊂⊃ stops flashing, display will read pH of STD 2.

III. MEASURE pH

① Rinse electrode(s). Blot excess.

② Immerse electrode(s) in sample. Stir briefly.

③ Press pH.

④ After ⊂⊃ stops flashing, display will read pH of sample.

FOR MORE DETAILED INSTRUCTIONS ON pH MEASUREMENT, SEE NEXT PAGE.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

DETAILED INSTRUCTIONS

METHODS: The pHI 10, 11, and 12 can measure pH from 0 to 15.99. They will perform one or two-point standardization automatically, using any buffer listed below, at any temperature between −5°C and 100°C.

STANDARD pH BUFFERS RECOGNIZED BY THE pHI 10, 11, AND 12:

1.68, 4.00, 7.00, 10.01, 12.45

TWO-POINT STANDARDIZATION METHOD:

Two-point standardization, the preferred and more accurate method of pH measurement, should be used when pH accuracy of beyond ±0.1 pH is required. Use buffers as close to the sample pH as possible; one above, and one below (For example, if sample pH is about 8.5, use 7.00 and 10.01 pH buffers).

ONE-POINT STANDARDIZATION METHOD:

One-point standardization, a somewhat faster procedure, is recommended only if (a), accuracy of ±0.1 pH unit is acceptable, and (b), sample pH is within 1.5 pH of that of the buffer used for standardization.

pH MEASUREMENT PROCEDURE:

1. Connect electrode(s) to appropriate input(s):
   a. If a combination electrode is used, connect it to the input marked "pH".
   b. If an electrode pair is used, connect the indicating electrode to the input marked "pH" and the reference electrode to the input marked "REF".
   c. For better accuracy, or when measuring and/or standardizing at a temperature of other than 25°C, connect a Beckman 558115 Automatic Temperature Compensator probe to input marked "ATC".

2. Press ⓪ to turn on instrument, then press [C] to clear. Display will show [CAL AUTO].
3. Rinse electrode(s) (and ATC if used) with deionized water. Blot excess.
4. Immerse electrode(s) (and ATC if used) in first standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press [pH]. Displayed pH value will have a resolution of 0.01. If 0.1 resolution is desired, press [illegible].
5. Press [illegible]. When [ ] stops flashing, display will show [pH value locked, , ▶ $\overset{1}{m}$].
6. Rinse electrode(s) (and ATC probe if used) with deionized water. Blot excess. Proceed to appropriate step, according to desired type of standardization:
   a. If ONE-POINT standardization is to be used, instrument is ready for sample measurement; proceed to Step 9.
   b. If TWO-POINT standardization is desired, proceed to Step 7.
7. Immerse electrode(s) (and ATC if used) in second standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press [illegible]. When [ ] stops flashing, display will show [pH value locked, , ▶ $\overset{1}{m}$, ▶ $\overset{2}{m}$].
8. Rinse electrode(s), (and ATC probe if used) with deionized water. Blot excess.
9. Immerse electrode(s) (and ATC if used) in sample. Stir briefly with electrodes. Press [pH]. When [ ] stops flashing, display will show [pH value locked]. Measurement is now complete. Repeat Steps 8 and 9, above, for additional samples.
10. If continuous pH monitoring is desired, press [AR] to turn off Auto Read function.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

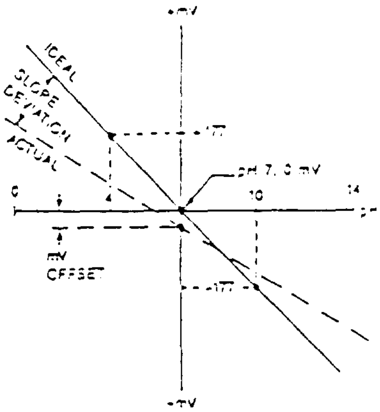
ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

SPECIFICATIONS

The pHI 10/11/12 pH Meter is essentially a high-impedance voltmeter with a microcomputer that translates voltage and temperature data into pH units. At 25°C the ideal pH electrode system develops -59 mV per pH unit increase, with 7.00 pH = 0 mV.

Standardization allows the meter to compensate for non-ideal electrode characteristics. One-point standardization compensates for millivolt offset; two-point standardization compensates for both millivolt offset and slope deviation. See diagram below.



Ideal and Actual Electrode Response Compared

The pHI 10, 11, and 12 may be standardized with any of five standard pH buffers: 1.68, 4.00, 7.00, 10.01, and 12.45. Standardization may be accomplished with any two buffers, used in any order (and at any temperature, if ATC is used). When $\boxed{\text{pH}}$ is pressed, the instrument automatically recognizes the buffer.

The relationship between pH and electrode voltage changes with temperature. For precise pH measurements or temperatures not close to 25°C, a Beckman 558115 ATC (Automatic Temperature Compensator) probe should be used. With this probe, the instrument automatically compensates for the temperature characteristics of the buffer, permitting a sample to be measured at any temperature, even if different from the buffer temperatures. With ATC, the instrument measures and displays temperatures from -5°C to 100°C.

If an ATC probe is not used, the instrument defaults and displays 25°C.

The pH calculation is based on the Nernst equation:

$$E = E_a - \frac{2.3\,RT}{nF} \log a_i$$

E is the total potential, in millivolts, developed between the sensing and reference electrodes; E_a varies with the choice of electrodes, temperature, and pressure; $2.3RT/nF$ is the Nernst factor (*R* and *F* are constants, *n* is the charge on the ion, including sign, *T* is the temperature in degrees Kelvin), and a_i is the activity of the ion to which the electrode is responding.

For further information on principles and theory of pH measurement, refer to The Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

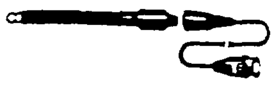
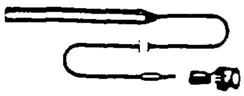
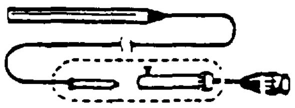
INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

FOR ACCURATE mV MEASUREMENTS WITH THE Φ11 OR Φ12, THE FOLLOWING ITEMS ARE RECOMMENDED:

1.		pH indicating electrode, Futura II. Futura II cable with BNC connector. NOTE: If combination pH electrode is used, omit separate reference electrode (item 2, below).
OR		
1a.		Metallic electrode with 2 mm pin connector + pin-to-BNC adaptor.
OR		
1b.		Ion-Selective electrode with BNC connector (or U.S. standard connector + U.S. standard-to-BNC adaptor).
2.		Reference electrode, Futura II. Cable with 2 mm pin connector, Futura II. NOTE: Omit reference electrode if combination pH electrode is used.
3.		Standard solution(s) appropriate to the application.
4.		Clean beaker(s) or equivalent container(s), 100–250 mL, for containing standard solution(s).
5.	 H_2O OR  H_2O	Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.
6.		Clean towels, "Kimwipes™", etc., for blotting electrodes.
7.	 SAMPLE	The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For mV measurement procedures, see next page.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

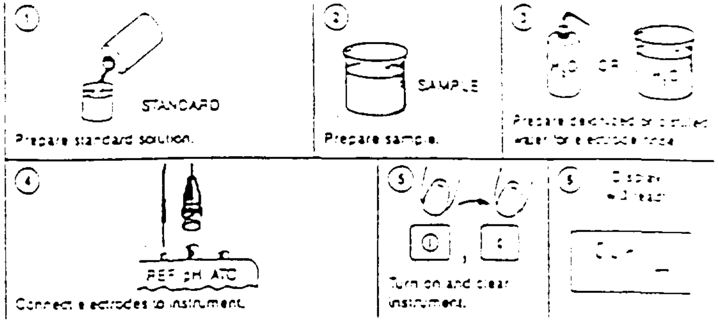
INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

SPECIFICATIONS

I. SETUP



① Prepare standard solution.

② Prepare sample.

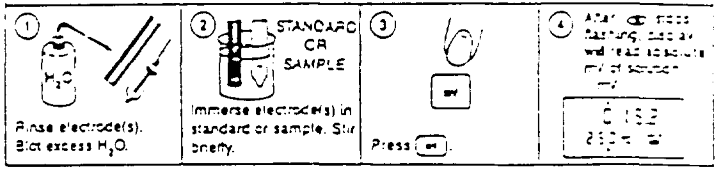
③ Prepare deionized or distilled water for electrode rinse.

④ Connect electrodes to instrument.

⑤ Turn on and clear instrument.

⑥ Display will read

II. mV MEASUREMENT, ABSOLUTE. FOR RELATIVE mV MEASUREMENT, SEE III, BELOW.



① Rinse electrode(s). Blot excess H_2O.

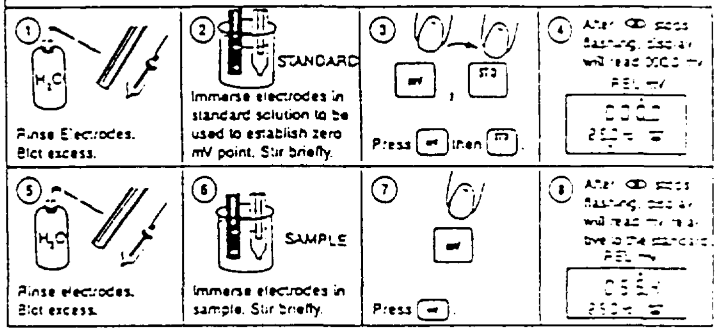
② Immerse electrode(s) in standard or sample. Stir briefly.

③ Press mV.

④ After ◑ stops flashing, display will read absolute mV of solution. mV

For RELATIVE mV Measurement, Proceed with following steps:

III. mV MEASUREMENT, RELATIVE



① Rinse Electrodes. Blot excess.

② Immerse electrodes in standard solution to be used to establish zero mV point. Stir briefly.

③ Press mV then STD.

④ After ◑ stops flashing, display will read 00.0 mV REL mV

⑤ Rinse electrodes. Blot excess.

⑥ Immerse electrodes in sample. Stir briefly.

⑦ Press mV.

⑧ After ◑ stops flashing, display will read mV relative to the standard. REL mV

NOTE
INSTRUMENT WILL REMAIN IN RELATIVE mV MODE UNTIL EITHER mV, Conc, OR C IS PRESSED.

FOR MORE DETAILED INSTRUCTIONS ON mV MEASUREMENT, PROCEED TO NEXT PAGE

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

mV MEASUREMENT: TYPICAL USES

Some uses of the *mV* mode are monitoring chemical reactions, quantifying ions, and determining the oxidizing-reducing potential (ORP) of a given sample. Because such measurements are usually not specific for a particular ion or species, readings must be interpreted carefully to obtain meaningful results. The user should have an understanding of the reaction that is occurring, or is desired, and of any sample components that could potentially interfere. For more detailed information, refer to the Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

The *mV* mode may also be used with ion-selective electrodes. The relative *mV* mode can be used in the standard addition or standard subtraction method of ion analysis.

STANDARD SOLUTION(S)

Make up appropriate standard solution(s) to provide known voltage(s), depending on the reference electrode used and the temperature. For example, common standards used in redox measurements are pH 4 and pH 7 buffers saturated with quinhydrone.

mV MEASUREMENT PROCEDURE

1. Connect electrodes to appropriate inputs:
   a. Connect indicating electrode to input marked "pH". A Pin-to-BNC Adaptor may be required as most metallic electrodes have a pin connector.
   b. Connect reference electrode to input marked "REF".
2. Press [①] to turn on instrument, then press [C] to clear. Display will show [Clr, AUTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in desired solution. Press [mV]. Displayed value is absolute mV, as indicated by display of [$\overset{mV}{\blacktriangle}$]. When [ꝏ] stops flashing, display will show [mV reading locked, ꝏ].

RELATIVE mV MEASUREMENT PROCEDURE

1. Perform Steps 1 through 3 of mV MEASUREMENT PROCEDURE, above.

2. Immerse electrodes in standard solution to be used to establish the zero mV point. Press [mV], then [std]. When [ꝏ] stops flashing, display will read [000.0 mV]. Note that, in mV mode, pressing [std] causes the instrument to establish the zero mV point at the value of the current reading. If desired, this step may be repeated at any time to re-establish the zero mV point.

3. Rinse electrodes with deionized water. Blot excess.

4. Immerse electrodes in sample. Press [mV]. Displayed value is *relative* mV, as indicated by display of [$REL_{\blacktriangle}mV$]. When [ꝏ] stops flashing, display will show [sample relative mV value locked, ꝏ]. Absolute mV reading of the standard solution is *automatically* subtracted from the absolute mV reading of the sample, resulting in a relative mV reading for the sample.

5. If *continuous* readout of relative mV is desired, press [ar] to turn off Auto Read function.

NOTE

VOLTAGE DIFFERENCE BETWEEN STANDARD SOLUTION AND SAMPLE MUST NOT EXCEED 1000 *mV*. MAXIMUM DISPLAY RANGE IN *mV* MODE IS ± 999.9 *mV*.

NOTE

IN *mV* MODE, THE 598115 AUTOMATIC TEMPERATURE COMPENSATOR PROBE MAY BE USED FOR TEMPERATURE *MEASUREMENT* AND *DISPLAY*, BUT DOES *NOT* HAVE ANY *TEMPERATURE-COMPENSATING* EFFECT.

MEASURING CONCENTRATION (Φ12)

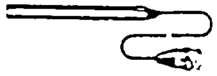
INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

FOR ACCURATE CONCENTRATION MEASUREMENTS WITH THE Φ12, THE FOLLOWING ITEMS ARE RECOMMENDED:

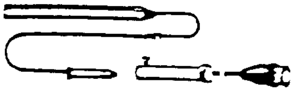
1.



Ion-Selective electrode with BNC connector

OR

1a.



Ion-Selective electrode with U.S. standard connector + U.S. standard to BNC adapter

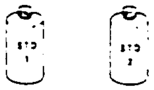
2.

Reference electrode, Futura II

Futura II cable with 2 mm pin connector

NOTE: Depending on the application, a salt bridge or double junction electrode may be required.

3.



Two standard solutions of appropriate concentration, selected from the following values: 1.00, 2.50, 5.00, 10.00, 25.0, 50.0, 100.0, 250.0, 500, and 1000 units. Concentration can be expressed in any desired units such as ppm, mM, mg/L, and ug/gal.

Make up these solutions per procedure or by diluting a stock solution to suit your requirement.

NOTE
CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

4.

Two clean beakers or equivalent containers, approximately 100–250 mL, for containing the two standard solutions.

5.

OR

Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.

6.

Clean towels, "Kimwipes™", etc., for blotting electrodes.

7.

The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For concentration measurement procedure, see next page.

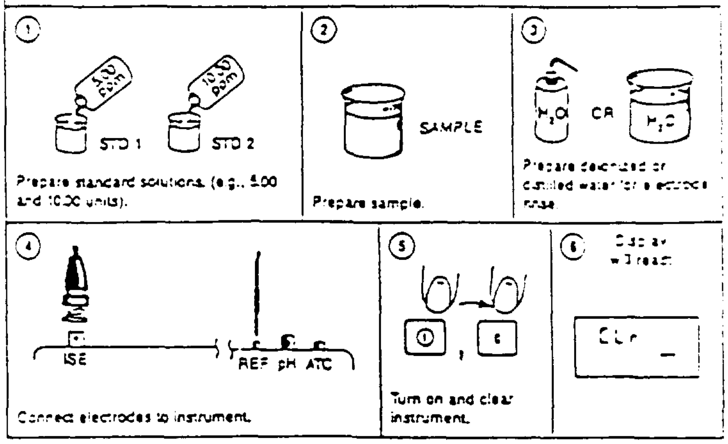
MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

I. SETUP



① Prepare standard solutions. (e.g., 5.00 and 10.00 units).

② Prepare sample.

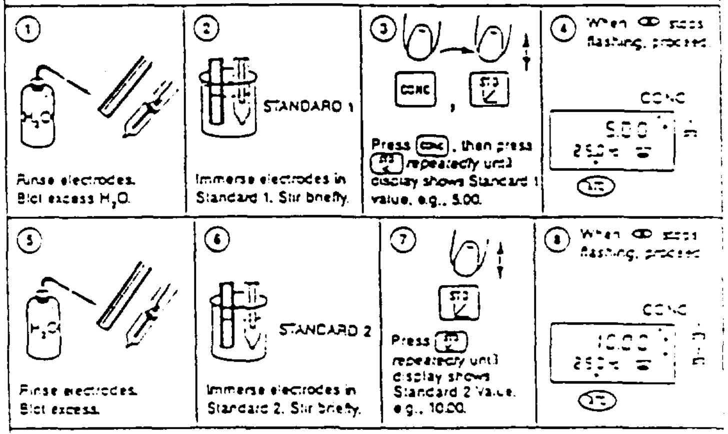
③ Prepare deionized or distilled water for electrode rinse.

④ Connect electrodes to instrument.

⑤ Turn on and clear instrument.

⑥ Display will read:

II. STANDARDIZE



① Rinse electrodes. Blot excess H_2O.

② Immerse electrodes in Standard 1. Stir briefly.

③ Press CONC, then press STD repeatedly until display shows Standard 1 value, e.g., 5.00.

④ When ◯ stops flashing, proceed.

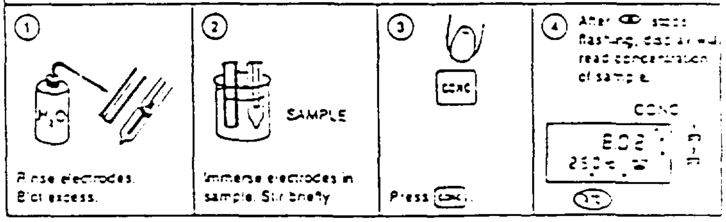
⑤ Rinse electrodes. Blot excess.

⑥ Immerse electrodes in Standard 2. Stir briefly.

⑦ Press STD repeatedly until display shows Standard 2 Value, e.g., 10.00.

⑧ When ◯ stops flashing, proceed.

III. MEASURE CONCENTRATION



① Rinse electrodes. Blot excess.

② Immerse electrodes in sample. Stir briefly.

③ Press CONC.

④ After ◯ stops flashing, display will read concentration of sample.

FOR MORE DETAILED INSTRUCTIONS ON CONCENTRATION MEASUREMENT, PROCEED TO NEXT PAGE.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

ions in almost any desired units.

STANDARD SOLUTIONS.

Standards can be made from any type of solution, with concentrations selected from the following values: 1.00, 2.50, 5.00, 10.00, 25.0, 50.0, 100.0, 250.0, 500, and 1000 units.

Units of concentration may be any that the user finds convenient. CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

Some examples of units are: parts per million, percent, moles per liter, parts per billion, milliequivalents per liter, and ounces per gallon.

Select two standard values as close as possible to the anticipated sample value, preferably with one standard value below and one standard value above the sample. For example, if sample solution is about 150 millimoles per liter (mM), make up standards of 100 mM and 250 mM. If sample concentration varies widely, for example, between 10 molal and 75 molal, make up standards of 10 molal and 100 molal.

Standards and samples should be at the same temperature to avoid temperature-dependent variations in readings.

NOTE

Standard and sample solutions may require ionic strength adjustment or interfering ion removal. Consult electrode instructions for details.

CONCENTRATION MEASUREMENT PROCEDURE:

1. Connect electrodes to appropriate inputs:
   a. Connect ion-selective electrode to input marked "ISE".
   b. Connect *reference electrode* to input marked "REF".

NOTE

If, in addition to the ion-selective electrode, a combination pH electrode is connected to the instrument AND is to be immersed in the same solution, DO NOT use a separate *reference electrode.*

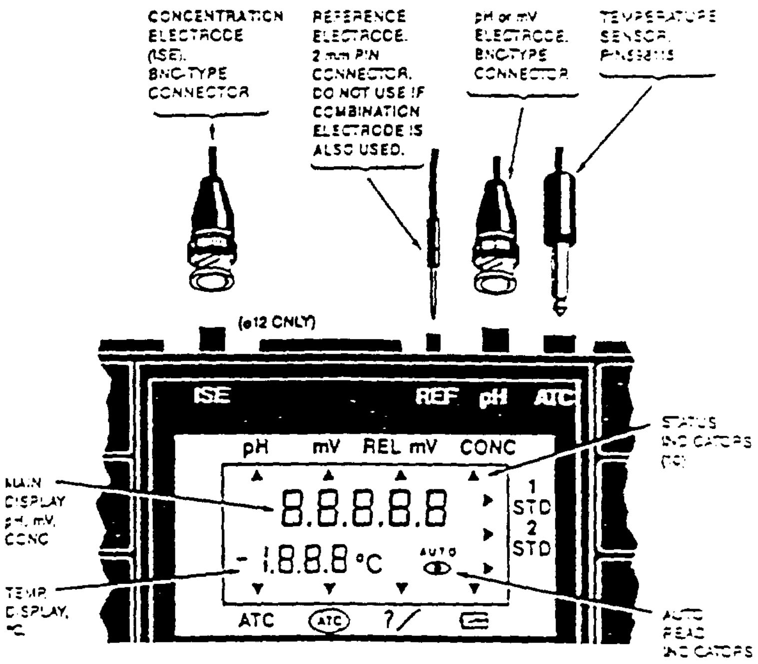
2. Press ⏻ to turn on instrument, then press [c] to clear. Display will show [C, AUTO]
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in first standard solution. Press ▭, then press ▭ as many times as needed *for the concentration value of the first standard to show on the display.* When ▭ is pressed repeatedly the display steps through the following values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units. For example, if the concentration of the standard is 100 units, press ▭ seven times and the display will show [100]. When [◐] stops flashing, display will show [100 locked ◐ , ▶ $\frac{1}{m}$].
5. Rinse electrodes with deionized water. Blot excess.
6. Immerse electrodes in second standard solution. The first and second standards must be different, *but can be measured in any order. Press* ▭ *as many times as needed for the display to show the* concentration value of the second standard, e.g., 250. When [◐] stops flashing, display will show [250 locked, ◐ , ▶ $\frac{1}{m}$].
7. Rinse electrodes with deionized water. Blot excess.
8. *Immerse electrodes in sample. Press* ▭. When [◐] stops flashing, display will show [sample value locked, ◐]. Measurement is now complete. Repeat Steps 7 and 8, above, for additional samples.
9. If *continuous* concentration readout is desired, press ▭ to turn off Auto Read function.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE

ELECTRODE CONNECTIONS



DISPLAY FEATURES AND STATUS INDICATORS

DISPLAY

Main Display The large digits show the following:

1. Readout of the measured variable: pH, mV, or concentration.
2. [Clr] is displayed, indicating that instrument is cleared, when [c] is pressed.
3. Error message:

   [Er] indicates an excessive, potentially damaging, input voltage, typically caused by static electricity when the electrode pair is not in solution. In this case, immerse electrodes in solution, press [c], and proceed with measurement. If [Er] again appears, check connections and electrodes for possible open circuit.

Temperature Display (°C) The small digits display temperature in °C. Will read 25°C if ATC not plugged in.

AUTO AUTO READ ON/OFF Indicator for AUTO READ ON/OFF *Key*, described subsequently.

⊕ AUTO READ Status Indicator (eye symbol). Functions during standardization and when instrument is in AUTO mode. During *standardization*, the eye symbol starts flashing when [F2] is pressed, and locks on when the reading has stabilized. During *sample measurement* in AUTO mode, the eye symbol starts flashing when a mode key is pressed, and locks on when the reading has stabilized. The reading remains locked until a mode key is pressed. If an interval of approximately 30 minutes elapses without a key being pressed, the instrument turns off automatically to conserve the batteries, but retains all standardization data in memory.

STATUS INDICATORS

▼ ATC Indicates that ATC is plugged in. The instrument measures and displays temperature within the range of −5°C and 100°C. Display of [Er] indicates that the temperature sensed is outside the measurement range, or the ATC is nonfunctional.

▼ (ATC) Indicates that ATC is *not* plugged in. The temperature reading defaults to 25°C.

pH ▲ Indicates that instrument is in pH mode.

mV ▲ Indicates that instrument is in mV mode. } Φ11 and Φ12 only

REL mV ▲ Indicates that instrument is in relative mV mode. } Φ11 and Φ12 only

CONC ▲ Indicates that instrument is in concentration mode. Φ12 only

▶ 1 STD Indicates that one standard has been used to standardize for the selected measurement mode (pH or CONC)

▶ 2 STD Indicates that two standards have been used to standardize for the selected measurement mode (pH or CONC).

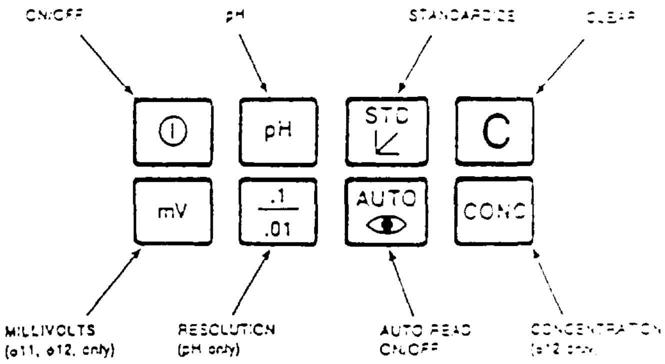
▼ ?/ Indicates a questionable electrode and/or standardization.

▼ Indicates that batteries should be replaced.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

KEYPAD



KEYPAD FUNCTIONS

KEY	
⏻	Instrument ON/OFF Key. When OFF, the instrument retains the standardization data in memory. Instrument shuts off automatically after 30 minutes of inactivity if AUTO READ is ON. (See below.)
C	Clear Key. Clears instrument, resetting all standardization data to default values, and returning instrument to AUTO Mode.
AUTO 👁	Auto Read Key. Turns Auto Read function ON and OFF: 1. When Auto Read is ON: a. The word [AUTO] appears on the display. b. The instrument tests the electrode signal for stability. During this test, [👁] flashes ON and OFF. When the signal has met the stability requirement (see SPECIFICATIONS), [👁] remains on continuously, and the digital display locks onto the reading. No further measurements are made until a key is pressed. c. After 30 minutes without keypad input, the instrument turns off automatically but retains all standardization data. 2. When Auto Read is OFF: a. [AUTO] disappears from display. b. The instrument continuously measures and displays in the selected mode (pH, mV, or CONC. c. After 1 to 2 hours without keypad input, the instrument turns off automatically but retains all standardization data.
.1/.01	pH Resolution Selection Key. Changes resolution of the displayed pH reading from 0.01 to 0.1 pH unit, or vice versa. At the lower resolution (0.1), time required for the Auto Read to lock is shorter. (See SPECIFICATIONS).
pH	Selects the pH measurement mode.
mV	Selects the mV mode (pHI 11, pHI 12), for measurement of either absolute or relative millivolts. See MEASURING mV AND RELATIVE mV.
CONC	Selects the concentration measurement mode (pHI 12). Used with specific ion electrodes.
STD	Standardize Key. Standardizes instrument. Depends upon mode. a. pH Mode: [STD] Key causes the instrument to automatically identify the pH value of the buffer from any one of the following: 1.68, 4.00, 7.00, 10.01, and 12.45. b. mV Mode (pHI 11, pHI 12): [STD] Key causes the instrument to establish the zero-millivolt level at the value of the current reading. Instrument is now in Relative mV mode. c. CONC Mode (pHI 12): Repeated pressing of [STD] Key causes the instrument to step through the following sequence of values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units.

COMBINATION ELECTRODES:

	Standard 5" x 1/2"	Probe 8-10" x 3/8"	Test-Tube 8-3" x 5-6mm
Glass Body Ag/AgCl, Refillable	39520	39521	39522
Glass Body Calomel, Refillable	39527	39528	39525, 39525 [illegible]
Epoxy Body Calomel, Refillable	39838	—	39829
Epoxy Body Ag/AgCl, Refillable	39831	39832	39835
Epoxy Body Ag/AgCl, Gel Filled	39826	39822	39824
Epoxy Body, Star Ag/AgCl Refillable	39537		
Glass Body, Star Ag/AgCl Refillable	39524		
Flat Bulb, Epoxy Body	39523		

ELECTRODE PAIRS:

pH INDICATING ELECTRODES:

0–14 pH, Spherical Bulb	39314
0–11 pH, Dome Bulb (Durable)	39316

METALLIC ELECTRODES

Silver Billet	[illegible]
Platinum Inlay	[illegible]

REFERENCE ELECTRODES:

Calomel Half Cell, Quartz Fiber Junction	39416
Calomel Half Cell, Ceramic Frit Junction	39417
Ag/AgCl Half Cell, Quartz Fiber Junction	39418
Calomel Half Cell, Sleeve Double Junction	39419
Calomel Half Cell, Inverted Sleeve Junction	39420
Ag/AgCl Half Cell, Inverted Sleeve Junction	39421

2. FUTURA II KEEPER CABLES

COMBINATION AND INDICATING ELECTRODE CABLES

1m,	BNC Connector	597578
2m,	BNC Connector	597579
6m,	BNC Connector	597580

REFERENCE ELECTRODE KEEPER CABLES

1m,	2mm Pin Connector	598582
2m,	2mm Pin Connector	598583
6m,	2mm Pin Connector	598584

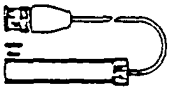
3. SALT BRIDGE: 563853

4. SUBMERSIBLE COMBINATION pH ELECTRODE WITH ATC: 39530

5. AUTOMATIC TEMPERATURE COMPENSATOR, 598115:

Permits temperature measurement and display, and temperature compensation of pH and ion-selective electrodes, within range of −5°C to 100°C. Epoxy body. For use with standard 5-inch (13-cm) electrodes. Includes 39" (1 meter) cable with miniature phone jack.

6. ELECTRODE ADAPTORS:



592362 Standard to BNC Adaptor

Adapts Glass Electrode (GE) BNC terminal on pHI Series pH Meters to accommodate electrodes with U.S. Standard Connectors.

592367 PIN to BNC Adaptor

Adapts Glass Electrode (GE) BNC terminal on pHI Series pH Meters to accommodate electrodes with PIN Connectors.

Typically used to connect metallic electrodes.

7. BUFFERS

	6 Pack of Pints	1 Gallon	5 Gallons	Powder (Colorless)
pH 4 Buffer (red)	532517	556001	532522	3005
pH 7 Buffer (green)	582521	556003	532523	3007
ph 10 Buffer (blue)	532525	556005	532524	3015
pH 12.45				3012

8. FILLING SOLUTIONS

Description	Quantity	Part No.
Combination Electrode Filling Solution or Ag/AgCl Reference Electrode Filling Solution (4M KCl/AgCl saturated: to be used with Ag/AgCl internals)	4-pack of 100 mL bottles	556457
Reference Electrode Filling Solution (saturated KCl to be used with Calomel internals)	4-pack of 100 mL bottles	556453
Electrode Soaking Solution	4-pack of 100 mL bottles	556275
Salt Bridge Solution, Contains Sodium Nitrate and Sodium Acetate	4-pack of 100 mL bottles	556459
Filling Solution, 1M, KCl Saturated with AgCl (Star-Series electrodes only)	4-pack of 100 mL	598942

	Part No.
9. pHI START-UP KIT: 39831 Electrode, Cable, Thermocompensator, Sample Buffers, Filling solution	120125
10. pHI STAND LAB ORGANIZER	120135
11. pHI DELUXE FIELD CASE	120123
12. pHI SOFT CASE	120127
13. pHI MOUNT, WALL/SHELF BRACKET	599192

FOR PRICE AND DELIVERY INFORMATION, CALL 1-800-742-2345.

of continuous operation. Replacement batteries can be obtained by ordering Part No. 945754 from your local Beckman office. (In U.S. call 1-800-742-2345).

9-55

Acceptable replacement batteries are also available on a world-wide basis.

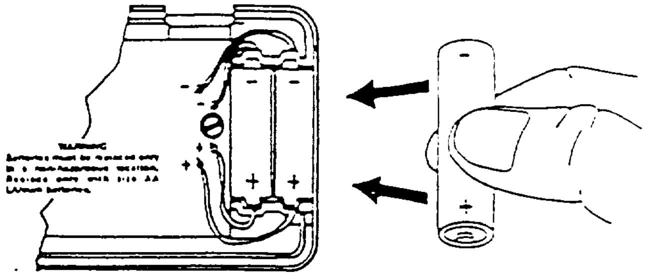
Mfr	Part No.
Electrochem Industries	[illegible]
Power Conversion Inc.	TC-[illegible]
Saft Advanced Battery Div.	LS[illegible]
Tadiran	TL-2100 AA/S

Local suppliers may be found in your telephone directory.

> Note that these batteries are 3.6 volt lithium cells. Do not attempt to replace them with 1.5 volt alkaline or carbon-zinc cells.

If instrument display indicates low battery voltage ⟨▼▭⟩ or if display is blank when instrument is turned on, batteries should be replaced:

1. Remove 2 Phillips screws and bottom cover from instrument.
2. Lift out old batteries.
3. Note (+) and (–) markings in battery compartment.
4. Check (+) and (–) markings on batteries and insert as shown:



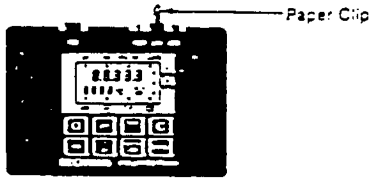
5. Replace back cover and screws.

> IMPORTANT: A "BREAK-IN" PERIOD OF UP TO 30 MINUTES IS REQUIRED WHEN SOME NEW LITHIUM BATTERIES ARE FIRST PLACED INTO SERVICE. DURING THIS PERIOD, THE LOW BATTERY SYMBOL AND SOME "GHOSTING" MAY APPEAR ON THE DISPLAY. IF SO, LEAVE INSTRUMENT ON FOR 20–30 MINUTES AND THEN PRESS ▭. THE LOW BATTERY SYMBOL AND "GHOSTING" SHOULD DISAPPEAR.

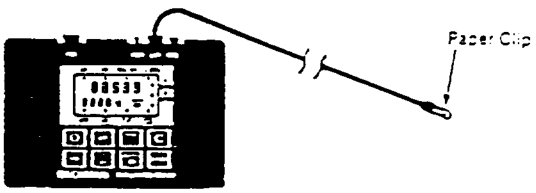
TROUBLESHOOTING PROCEDURE

1. Disconnect electrode cable(s) from instrument. Press ⏻ to turn on instrument, then press ▭ to clear. Display should show [Clr, AUTO]. If not, replace batteries per BATTERY REPLACEMENT above. If instrument is still inoperative, call Service Hot Line: 1-800-662-6217.

2. Insert one end of a paper clip into the small hole in the center of the "pH" input connector. Hold the other end of the clip to the inside barrel of the same connector as shown.



3. Press ▭, then ▭. The display should lock at pH 7.00, indicating a one-point standardization.
   a. If instrument passes test, go to Step 4.
   b. If instrument fails test, call Service Hot Line: 1-800-662-6217.
4. Reconnect pH electrode cable to "pH" input connector. Short the input connector of the cable



Press ▭ and then ▭. Display should lock at pH 7.00. Press ▭, then remove paper clip. Reading should drift.
   a. If instrument passes test, go to Step 5.
   b. If instrument fails test, call Beckman Electrochemistry Applications:
      1-800-854-8067 Outside California
      714-871-4848 Within California
5. Reconnect pH electrode(s). Immerse electrode(s) in pH 4 buffer and perform one-point standardization. Then immerse electrode(s) in pH 10 buffer and take pH reading. At 25°C, the reading should be between 9.7 and 10.1 pH.
   a. If the test is passed, the pH meter, cable, and electrode(s) are functioning properly.
   b. If test is failed, the pH electrode(s) must be rejuvenated or replaced. The electrode rejuvenation

INSTRUMENT BECKMAN PART NO.	pHI 10 123132	pHI 11 123133	pHI 12 123134
pH MEASUREMENT			
Range	0 to 15.99 pH	0 to 15.99 pH	0 to 15.99 pH
Resolution (Selectable)	0.01, 0.1 pH unit	0.01, 0.1 pH unit	0.01, 0.1 pH unit
Relative Accuracy	± 0.01 pH	± 0.01 pH	± 0.01 pH
Auto Read Mode	0.1 pH Resolution: Display locks after reading is stable within 1.0 mV for 4 seconds. 0.01 pH Resolution: Display locks after reading is stable within 0.5 mV for 8 seconds.		
Buffers Recognized by Instrument: 1.68, 4.00, 7.00, 10.01, 12.45 pH.			
MILLIVOLT MEASUREMENT			
Range	—	-999.9 to + 999.9 mV	-999.9 to + 999.9 mV
Resolution	—	0.1 mV	0.1 mV
Accuracy	—	± 0.2 mV ± 0.02% of reading, relative mV	± 0.2 mV ± 0.02% of reading, relative mV
Auto Read Mode	—	Display locks after reading is stable within 0.5 mV for 8 seconds.	
CONCENTRATION MEASUREMENT			
mV Accuracy	—	—	± 0.1 mV
Auto Read Mode	—	—	Display locks after reading is stable within 0.25 mV for 8 seconds.
Useable Standard Values	—	—	Two values, in any desired units, selected from the following: 1.0, 2.5, 5.0, 10, 25, 50, 100, 250, 500, and 1000.

TEMPERATURE MEASUREMENT (ALL MODELS)

Range: -5°C to 100°C
Resolution: 0.1°C
Accuracy (with Beckman 598115 Probe): ± 0.5°C

MISCELLANEOUS (ALL MODELS)

Input Connections:
1. BNC input for pH, mV, and concentration.
2. 2-mm pin connector for reference electrode.
3. Miniature phone jack for Beckman 598115 Automatic Temperature Compensator.

Operating Temperature: 15°C to 40°C, ambient, non-condensing.
Power Source: Two lithium cells, 3.5 volts each, AA Size.

Error Indications:
1. Input overvoltage (all modes)
2. Temperature compensation non-functional
3. Low batteries
4. Questionable electrode/standardization.

Size: 5.2 inches x 3.9 inches x 1.3 inches.

Bekman Inc
National
West Poplar Ave
CA 93257

Instructions

E-11

Platinum Combination Electrode

Catalog No. 13-639-82

The Fisher platinum combination electrode combines a silver/silver chloride reference element and a platinum-wire indicating element in a single probe. This "dual element" configuration eliminates the need for two separate electrodes and is especially applicable to measurements in narrow-neck flasks and other restricted-entry receptacles.

The platinum combination electrode is recommended for use with automatic titrators and similar electroanalytical equipment. The close physical proximity of the porous-plug liquid junction to the platinum-wire indicator section results in reduced resistance between these elements and produces a rapid dynamic response for both redox measurements and potentiometric titrations. Additionally, the electrode is ideally suited for any application that involves the measurement of oxidation-reduction potentials or requires the use of a "noble metal" sensor.

The electrode measures 5 inches with a 30-inch lead, and functions over a −5° to 100°C temperature range. The filling solution is 4M KCl saturated with AgCl (Fisher No. So-P-135), and flow rate at the junction is less than 8 µl per hour at an 8 cm head. Reference output is 44 ± 1mV vs. S.C.E., while junction resistance is less than 10K ohms.

INSTALLATION

Place the platinum combination electrode into service as follows:

1. Remove cap from supplied filling-solution bottle, and screw on dispenser spout.
2. Lower rubber sleeve on electrode body until filling hole is exposed, and fill reference cavity with electrolyte until meniscus reaches a level approximately ¼-inch below filling hole.

   NOTE: Always use 4M KCl solution saturated with AgCl (Fisher No. So-P-135) as the electrolyte. NEVER USE SATURATED KCl FILLING SOLUTION.
3. Place electrode upright in empty beaker to permit filling solution to wet and flow through porous plug, as evidenced by formation of KCl crystals on outer surface of plug.

   NOTE: If no flow is observed within 30 minutes, or if response is unsatisfactory during an analysis, soak electrode in dilute KCl (0.1M) for several hours, and then perform the following procedure:
   a. Hold electrode (cap up) at a 45° angle between thumb and forefinger on left hand, so that filling hole faces out and is directly opposite base of thumb.
   b. Insert dispensing spout into filling hole.
   c. Make sure that electrode is supported by base of thumb, then firmly press spout into filling hole to make an airtight seal.

      NOTE: Normally, spout tip will not touch internal element; while applying pressure, however, care should be exercised to prevent contact. If necessary, cut off a portion of the tip.
   d. While maintaining seal, squeeze filling bottle firmly so that electrode becomes pressurized.

      NOTE. A bead of liquid should form at liquid junction in about 30 seconds; in some cases, however, it may be necessary to maintain pressure for several minutes. If flow cannot be established, refer to REJUVENATION section.
4. Mount electrode on suitable holder and connect jacks to pH meter.

OPERATION

For optimum operation with the platinum combination electrode, observe the following general procedures:

1. Rubber sleeve should always be lowered on electrode body to expose filling hole and permit proper electrolyte leakage.
2. Level of electrolyte must always be maintained above surface of sample solution to avoid backflow of sample into electrolyte. Refill reference cavity as required.
3. After removing electrode from one solution and before immersing in another, the outer surface should be rinsed with distilled water.

STORAGE

When not in use, store the platinum combination electrode as follows:

1. Slide rubber sleeve into position over the filling hole.
2. Place supplied cot over tip of electrode by threading platinum wire through opening and sliding cot onto glass body until porous plug is completely covered.

REJUVENATION

Rejuvenation of the platinum combination electrode may only require a simple cleaning. Occasionally, a more thorough cleaning is required, or the porous-plug junction may have to be unblocked. Each is covered separately below.

Simple Cleaning

A simple cleaning of the electrode is done as follows:

1. Wash electrode surface with a good detergent.

   NOTE: RBS-25 detergent (Fisher No. So-C-181) is recommended.
2. Polish platinum wire with scouring powder.
3. Rinse electrode thoroughly with distilled water.

Thorough Cleaning

For a more thorough cleaning, perform the following:

1. Connect tip of large cable plug to negative terminal of a 22V dry cell, then immerse tip of electrode in a 1N solution of hydrochloric acid.
2. Similarly connect a platinum or graphite electrode to positive terminal of dry cell and immerse tip of electrode in same solution.

   NOTE: Hydrogen will evolve rapidly, and the metallic electrode will be cleaned by electrolysis in 5 to 10 seconds.
3. After cleaning, disconnect both electrodes and rinse

Instrument Manufacturing Division Fisher Scientific Company

each with distilled water

Unblocking the Junction

If the liquid junction should become partially blocked, perform the following:

1. Inspect reference cavity for crystallization.
2. If crystals are evident, proceed as follows:
   - **a.** Remove filling solution by shaking it out through filling hole.
   - **b.** Rinse cavity repeatedly with distilled water until all crystals are dissolved.
   - **c.** Refill cavity with fresh 4M KCl solution saturated with AgCl (Fisher No. So-P-135).

     CAUTION: Never use saturated KCl as the electrolyte.
   - **d.** Repeat all of step 3 under INSTALLATION.
3. If difficulty persists, perform the following in sequence depending upon the severity of the blockage:
   - **a.** Soak electrode overnight in dilute KCl (0.1M).
   - **b.** Boil junction in dilute KCl for 5 to 10 minutes.
   - **c.** Carefully sand or file the porous plug junction.

Electrode Instructions E-11
Revised and Published 4-78
Fifth Issue (8-0316-08)

Litho in U.S.A.

APPENDIX C-6

PERCENT METHANE AND OXYGEN

INSTRUMENT OPERATION PROCEDURE

GASTECHTOR Model 1939 Two-Gas Meter

For the Detection of Oxygen and
Explosive/Combustible Gases

January, 1991

SECTION 1
SCOPE AND APPLICATION

The GASTECHTOR (GASTECH) Model 1939 OX Gas Surveyor is a battery-powered portable instrument that can detect and indicate concentrations of combustible gases and oxygen in air. Combustible gas is measured in two ranges: 0 to 100% Lower Explosive Limit (LEL) and 0 to 100% by volume. Oxygen is measured over a range of 0 to 25% by volume. The instrument is equipped with a meter and audible alarms that sound whenever gases or oxygen concentrations reach preset levels.

REFERENCE

GASTECH Gas Surveyor Model 1939 OX Instruction Manual

APPARATUS

- GASTECH Model 1939 OX
- Battery charger
- Probe, 10 inch long, 1/4 inch OD plastic tube and dust filter
- Hose, 6 foot flexible nylon-jacketed polyethylene tube
- Calibration kit consisting of cylinders of methane, 2 to 5% LEL and 50 to 100% by volume concentrations

SECTION 2
DESCRIPTION

2.1 METER

Instrument readings are displayed on a meter, visible through a window on the top face of the instrument case. A mark on the scale, BATT CK, represents the minimum permissible battery voltage, as an indication of the state of the charge of the battery. The meter face has three scales.

2.1.1 The top scale is graduated from 0 to 25% O_2 for indicating concentration of oxygen. A calibration CAL mark indicates 21% O_2.

2.1.2 On the middle scale the meter reads gas concentration as 0 to 100% LEL. 100% LEL represents the minimum concentration of vapor in air that will ignite or explode when an ignition source is present.

2.1.3 The bottom non-linear scale reads 0 to 100% gas (%GAS) for indicating high concentrations of combustible gas.

2.2 CONTROLS

The six controls that are used in normal operation of the instrument are arranged on the left side of the instrument as viewed from the rear, and on the top. These controls are recessed to minimize possibility of accidental operation.

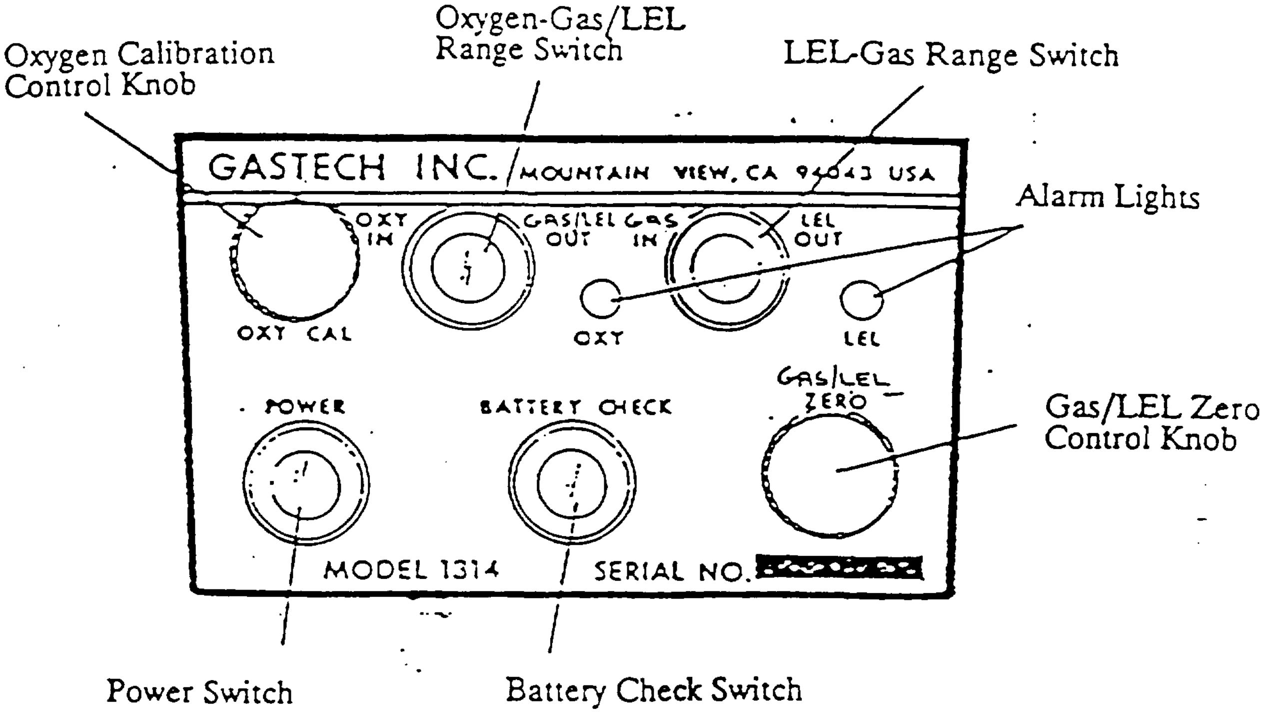


FIGURE 1

2.2.1 Power switch

An alternate-action push button switch which energizes the circuit when pressed. An orange indicator dot is exposed when the switch is in the ON position, serving as a mechanical pilot light.

2.2.2 Battery Check

A momentary push button switch, when pressed connects the meter as a voltmeter for battery condition check.

2.2.3 Gas/LEL Zero

A potentiometer which is used to adjust circuits to read zero in the absence of natural gas. The potentiometer shaft is fitted with a control knob for convenience in adjustment.

2.2.4 OXY/Gas-LEL Switch

An alternate-action push button switch used to select the combustible gas or oxygen range. In the OUT position, the instrument is in the GAS or LEL range. When IN, as indicated by the colored indicator dot, the circuit is in the OXY range.

2.2.5 Gas/LEL Switch

An alternate-action push button switch which selects the operating range for detection of combustible gas. In the OUT position, the instrument measures in the 0 to 100% LEL range. In the IN position, the instrument measures the 0 to 100% GAS range. A colored indicator dot shows when the switch is IN.

2.2.6 OXY Cal

A potentiometer which is used to adjust the circuit to read 21% when the detector is surrounded by normal air. The potentiometer shaft is fitted with a control knob for convenient adjustment.

2.3 INDICATORS

Alarm lights, red and amber, illuminate when the corresponding section of the instrument is in an alarm condition. The red light blinks in an on-off pattern of equal length pulses, to show that the instrument is in the combustible gas (LEL or GAS) alarm condition. Amber light blinks in a short-long pattern to show an abnormal oxygen condition.

2.4 BUZZER

A solid-state electronic buzzer is mounted at the rear interior of the instrument. The buzzer gives a pulsed tone on detection of gas, and a continuous tone in case of a malfunction, either low battery voltage or a down-scale drift of the meter. The audible tone pulses correspond to the visual pulses of the corresponding alarm light except in the case of excess O_2 which gives a steady tone.

TABLE 1
ALARM SETTINGS

% Oxygen:	<19.5% and >25%
% LEL:	>20% LEL
% Gas:	>20% Gas

SECTION 3
PRINCIPLE OF OPERATION

3.1 LEL RANGE

This range gives readings in terms of percent of the lower explosive limit (LEL), with 100% corresponding to the LEL of the specified gas.

Detection in this range is by catalytic oxidation on a platinum element, with the heat of oxidation producing a temperature rise and hence a resistance rise of the element. This change is approximately linear up to the LEL provided there is sufficient oxygen in the sample to support combustion.

3.2 OXYGEN SENSOR

The oxygen-sensing detector is an electrochemical cell in which electrodes are immersed in an alkaline electrolyte, and covered by a permeable fluorocarbon membrane. Oxygen from the surrounding atmosphere diffuses through the membrane and enters the electrolyte, causing an electrochemical reaction. The current generated by this reaction is amplified and used to drive the meter and the alarm circuit.

3.3 100 % GAS RANGE

This range gives readings in terms of gross concentration of the specified combustible gas. It uses a thermal conductivity filament, initially heated by the battery current to a point where it assumes a definite temperature and resistance. The temperature and resistance corresponds to the atmosphere, if the instrument is initially set-up in fresh air. Then when gas is sampled, the greater cooling ability of the gas (relative to air) causes the filament to become cooler and assume a lower electrical resistance. The resistance change produces a meter reading which can be calibrated in units of percent gas by volume.

The thermal conductivity phenomenon is continuous from 0 to 100%, so any concentration can be read without limitation. It is a relatively insensitive method, so is used only for the higher concentrations above the LEL. It is not dependent on combustion, hence is applicable regardless of the oxygen content of the sample.

SECTION 4
OPERATION

4.1 Attach the hose to the instrument by means of the threaded swivel fitting.

4.2 Put the OXY/GAS-LEL switch in the GAS-LEL (out) position, and GAS/LEL switch in the GAS (in) position, with its colored indicator showing.

4.3 Press the POWER switch to turn the instrument on, the orange indicator dot should be showing. The meter will normally rise up scale and a pulsing or steady alarm signal may sound. The alarm will stop when the instrument is warmed-up. The audible hum of the pump will be noticed.

4.4 Press the BATTERY CHECK button and note the meter reading. If the reading is close to or below the BATT CHECK mark on the meter, charge the batteries.

4.5 Allow the unit to warm up until the meter stabilizes (about a minute). Then, with the hose inlet in a contaminant-free location, turn GAS ZERO control knob to bring the meter to "0" indication.

4.6 The instrument is now ready to use in detecting high concentrations of the specified gas up to 100% by volume. The meter will indicate the concentration on the lower non-linear scale.

If the concentration indicated on the meter rises above the alarm point (set at 20%) the red ALARM light will blink and the buzzer will sound, both in a pulsing mode.

4.7 For readings in the LEL range, change the GAS/LEL switch to the LEL (out) position. The alarm will sound and the meter needle will move up-scale briefly. Wait about five minutes for the LEL sensor to stabilize. Move the probe about and observe meter indicators on the middle (% LEL) scale. The alarm point for this scale is set for 20% LEL.

4.8 Put the OXY/GAS-LEL switch in the OXY (in) position with the colored indicator showing. Turn the OXY CAL potentiometer to bring the meter to the oxygen CAL mark (21%). As a quick check gently breathe into the hose inlet and allow the instrument to sample air. The reading should come down to about 16% and the alarm should sound at 19.5%. Allow the meter to return to 21%, then put the switch back in the GAS-LEL (out) position.

4.9 Atmospheres containing more than the normal 21% oxygen will produce an increased oxygen reading. If the instrument is provided with a high oxygen alarm, then it will sound in a steady tone and the amber light will blink when readings reach or exceed 25%.

4.10 The instrument continuously tests for oxygen, and a pulsed audible alarm and blinking amber light will occur when oxygen drops to 19.5% or lower. It is not necessary to use the instrument with the switch in the OXY position unless oxygen meter readings are desired. If both abnormal conditions exist simultaneously (combustible gas and oxygen alarm), both lights will blink in their normal pattern, but the buzzer will sound continuously. A steady tone sounds when the oxygen reading exceeds 25%. This characteristic is provided to warn against the increased fire hazard due to excess oxygen. It also serves as a warning in case of oxygen cell failure in the high-output mode, which can occur occasionally. It further precludes accidental or intentional incorrect adjustment of the oxygen calibration control to an abnormally high level above 25%.

SECTION 5
CALIBRATION AND ADJUSTMENT

5.1 LEL CALIBRATION

5.1.1 Turn the instrument on and allow it to warm up and stabilize, preferably for five minutes. Be sure the batteries are charged sufficiently to read above the check mark.

5.1.2 Place the GAS/LEL switch in the LEL (out) position and the OXY/GAS-LEL switch in the GAS-LEL (out) position.

5.1.3 Open the instrument case by loosening the captive screw at the front of the unit. Lift the upper half of the case slightly, moving it 1/4 inch to the to rear to disengage the rear clamp; then separate the two halves. Locate the COARSE ZERO potentiometer on the underside of the circuit board (see Figure 2).

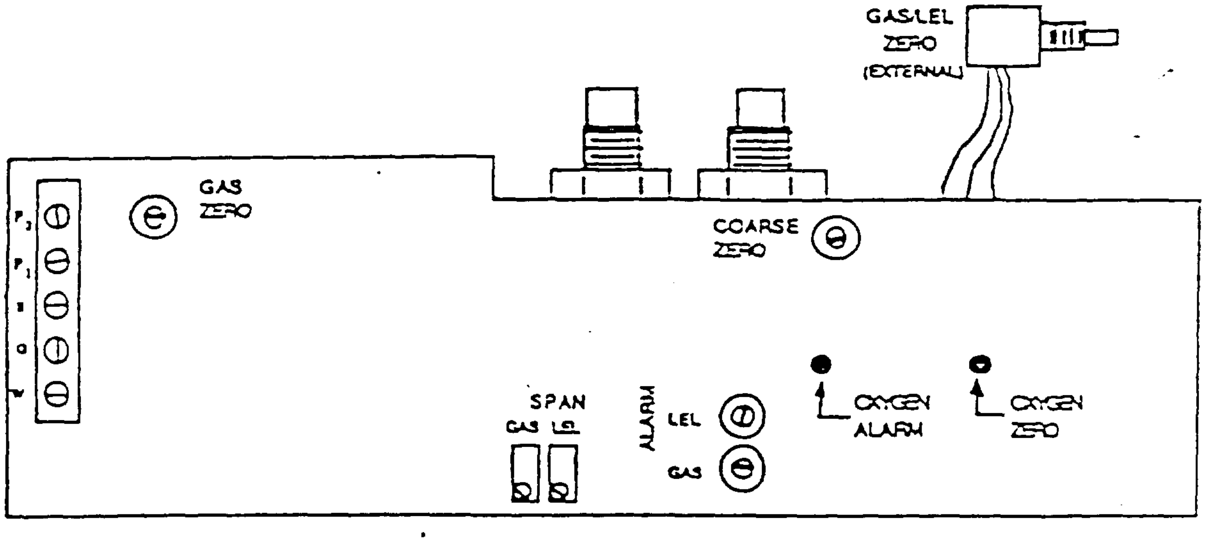


FIGURE 2

5.1.4 Turn the external GAS/LEL ZERO control to the center of its range.

5.1.5 Turn the COARSE ZERO potentiometer to bring the meter to the zero reading.

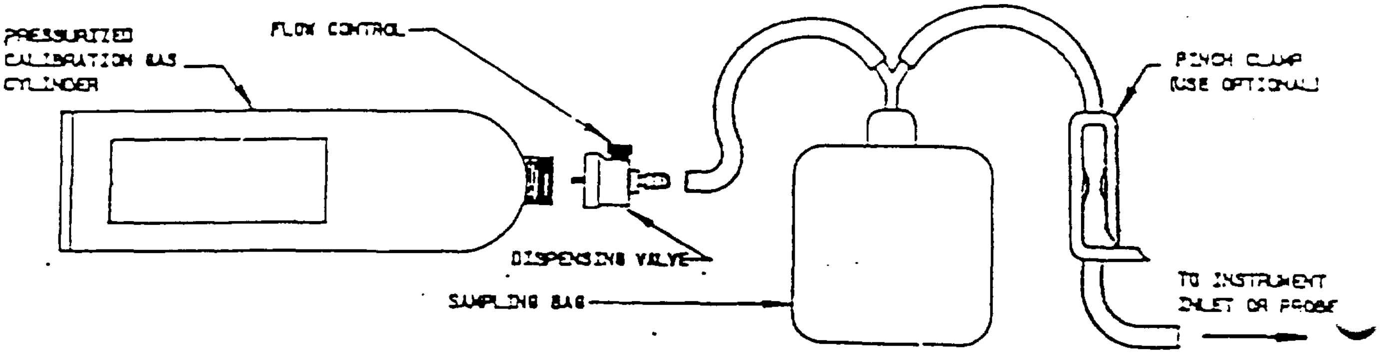


FIGURE 3

5.1.6 Remove the protective red plastic cap from the cylinder outlet of a tank of methane (2 to 5% LEL). See Figure 3.

5.1.7 Check the DISPENSING VALVE to see that the FLOW CONTROL is shut off (clockwise), but not so tightly as to damage the valve seat.

5.1.8 Screw the DISPENSING VALVE onto the cylinder outlet, being careful not to cross-thread. Tighten the valve firmly.

5.1.9 Push the short piece of flexible plastic tubing from the Y-fitting onto the barbed nipple of the DISPENSING VALVE.

5.1.10 Locate the adjustment potentiometer(s).

5.1.11 Connect the longer plastic tube with the PINCH CLAMP to the instrument. The PINCH CLAMP should be open (push latch forward to open).

5.1.12 Allow the instrument to evacuate the SAMPLING BAG until flat then close the PINCH CLAMP (squeeze paralleled faces together until tight and latched).

5.1.13 Open the FLOW CONTROL slightly so that the SAMPLING BAG begins to expand, then quickly open the PINCH CLAMP. As the instrument processes the sample, the bag will either start to expand further or to deflate. If the bag is expanding, close down on the FLOW CONTROL (clockwise), or if it is deflating, open the FLOW CONTROL slightly until the bag stays about half inflated.

5.1.14 Observe the meter on the instrument. The reading will increase, then stabilize. As soon as the reading is stable, compare with the value on the pressurized calibrating gas cylinder and record or change with the LEL SPAN potentiometer as appropriate.

NOTE Care must be taken to ensure that the LEL SPAN potentiometer and not the LEL alarm is adjusted.

5.1.15 Quickly close the FLOW CONTROL on the DISPENSING VALVE (clockwise) and disconnect the LEL calibration cylinder.

5.2 % GAS CALIBRATION

The 0 to 100% GAS range is calibrated in the same manner as the LEL range above, except adjustment is made using the GAS ZERO and GAS SPAN potentiometers and a cylinder of 100% methane (natural) gas. Place the GAS LEL switch in the 100% GAS (in) position for this calibration. **Do not change other zero adjustments.**

When the calibration is complete, remove the DISPENSING VALVE from cylinder before storage, to prevent loss of cylinder contents, then cap the cylinder. SAMPLING BAG, DISPENSING VALVE and PINCH CLAMP may be left connected to the tubing for storage.

5.3 OXYGEN CALIBRATION

5.3.1 Calibrate in fresh air.

5.3.2 Put the OXY/GAS-LEL switch in the OXY (in) position with the colored indicator showing.

5.3.3 Turn the OXY CAL potentiometer to bring the meter to the oxygen CAL mark (21%).

NOTE If problems are encountered during the calibration procedures, the unit must be replaced.

NOTE Instrument alarms are factory-set and must not be changed.

SECTION 6
MAINTENANCE/TROUBLE SHOOTING

6.1 BATTERIES

6.1.1 Check the battery voltage periodically by pressing the BATTERY CHECK switch. Charge the batteries before the voltage reaches minimum.

When connecting the charger, always follow these steps:

a. Confirm that the plug is inserted in the correct way, with the THIS SIDE UP label upwards. The plug is keyed with a non-current carrying third socket to aid in proper insertion.

b. Verify that the amber light on the charger is on. Leave connected until the green light comes on, indicating that the 16 hour charge is completed.

6.1.2 If sufficient voltage cannot be obtained after charging, the battery needs to be changed.

6.2 CATALYTIC SENSOR (LEL RANGE)

The sensor assembly may require replacement if:

a. The meter cannot be set to zero within the range of the GAS/LEL ZERO potentiometer.

b. The meter cannot be set to the desired level within the range of its SPAN potentiometer.

6.3 THERMAL CONDUCTIVITY SENSOR (GAS RANGE)

The sensor assembly may require replacement if:

a. The meter cannot be set to ZERO within the range of the GAS ZERO potentiometer.

b. The meter cannot be set to the desired level within the range of its GAS SPAN potentiometer.

6.4 OXYGEN DETECTOR

The oxygen sensor assembly may require repair if the meter cannot be set to the desired level within the range of the OXY CAL adjustment.

SECTION 7
PRECAUTIONS AND NOTES ON OPERATION

7.1 HEATED SAMPLES

When sampling spaces that are warmer than the instrument, such as hot manholes, remember that condensation can occur as the sample passes through the cooler sample line. Water vapor condensed in this way can block the flame arrestors and interfere with sensors and pump operation.

7.2 FILAMENT POISONING

Certain substances have the property of desensitizing the catalytic surface of the platinum filament. These substances are termed catalyst poisons and can result in reduced sensitivity or in failure to give a reading on samples containing combustible gas. The most commonly encountered catalyst poisons are silicone vapors. Samples containing such vapors, even in small proportions, should be avoided.

Frequent calibration checks on known-gas samples are desirable, especially if the possibility exists of exposure to silicones.

7.3 OTHER GASES AND VAPORS

The instrument is designed and calibrated specifically for detection of methane. It can be calibrated for use on other gases and vapors, by proper adjustment of the calibration control while sampling a known gas-air mixture.

7.4 RICH MIXTURES

When sampling rich mixtures, on the 100% LEL range, the following instrument action may be expected:

1. Mixtures up to 100% LEL - Reading on scale

2. Mixtures between LEL and Upper Explosive Limit (UEL) - Readings at top of meter
3. Mixtures above UEL - As sampling continues, the meter first goes to the top of the scale, then comes back down on scale. Very rich mixtures will give close to a zero reading on the catalytic sensor.

Rich mixtures should always be tested first on the 100% GAS range, to avoid any uncertainty as to actual concentration.

7.5 OXYGEN DEFICIENT MIXTURES

Samples which do not have the normal proportion of oxygen may tend to read low in the LEL range, as there is not enough oxygen to react with all the combustible gas present in the sample. As a general rule, samples containing 10% oxygen or more have enough oxygen to give a full reading on the catalytic sensor for any combustible gas sample up to the LEL. This limitation does not apply to the thermal conductivity sensor used for the 100% GAS range.

NOTE For very rich or oxygen-lean mixtures the instrument should be used in the 0 to 100% GAS range.

Erik A. Goplin

Erik A. Goplin
Loss Control Specialist

Mary Ann Latko

Mary Ann Latko, CSP
Corporate Health and Safety Manager

EAG/dlk/MAL/H&SPC
SAFETY:IOPS

APPENDIX C-7

CARBON DIOXIDE

BLUE BOOK

SENSIDYNE®

Gastec Precision Gas Detector System Manual

SENSIDYNE®

CONTENTS

I. GENERAL

1. Gas Detector Tube System

In order to insure environmental improvement it is most important to measure simply and rapidly very minute gas or vapor concentration in the work place.

Modern technology provides a variety of instruments for the measurement of hazardous gases and vapors. Such relatively sophisticated instruments must:

1. be capable of measuring concentrations in the range of threshold limit values.
2. must be easy to use, and available for taking quick samples in the work environment.
3. must produce results for evaluation and analysis within a few minutes, making it unnecessary to take samples back to the laboratory.
4. must provide dependable accuracy and reproducibility.
5. make it unnecessary to use expensive and delicate instrumentation.

The following three steps are essential for the chemical analysis of toxic gas or vapor:

1. collecting of samples
2. preparation of measuring apparatus and reagents
3. chemical treatment and analysis of the samples

The introduction of detector tube technology eliminates steps two and three above and makes it possible to achieve immediate results with the accuracy and reproducibility necessary for environmental assessment.

2. Principle of Gastec Detector Tube

Each detector tube contains a precise amount of detecting reagents in a constant inner diameter glass tube and is hermetically sealed at both ends.

To operate, break tips off a fresh tube and connect the tube to the Gastec sampling pump and pull the handle to take the required sample (sample gas). The chemical reagent in the detector tube will then react with the sample gas immediately and a color stain will develop starting at the inlet of the detector tube. The gas concentration is measured at the interface of stained-to-unstained reagent when staining stops.

Gastec detector tubes contain colorimetric reagents adsorbed on fine grain silica gel, activated alumina or other adsorbing media. The reagents are sensitive to particular gases or vapors and react quantitatively to provide a length-of-stain indication.

3. Characteristics of Gastec Gas Detector System

Detector Tube

1. Precision direct reading calibration scale printed on each tube.
2. Every tube and tube box displays the quality control number and chemical symbol. The expiration date is stamped on each box.
3. Controlled small internal diameter of tubes provide longer stain length with one *pump stroke (100 ml), resulting in more accurate readings and shorter sampling* times.
4. Sensitive reagents with high reaction rates provide a clear line of demarcation of color stain, for improved accuracy in tube reading.

Sampling Pump

1. Light weight, compact (approx. 10 oz.).
2. Pulls the highest vacuum (8.1" of Hg).
3. Gastec design does not require flow-rate orifices.
   Malfunction of the pump by clogging or leaking orifices is eliminated.
4. Handle locks at precise intake volume of 50 or 100 cc.
5. Friction proof lubricant seal packing provides complete leakproof sampling at all times.

4. Accuracy of Gastec Detector Tube

Calibration Scale

The calibration curve on most Gastec detector tubes is a straight line, and points on the scale are at equal intervals (Fig. 1). Therefore, it is possible to measure the concentration below the lowest calibration scale by repeating pump strokes. It is also possible to measure the concentration above the highest calibration scale by using a half-pump stroke. This means that the measuring range of Gastec detector tubes is easily and accurately expanded by varying the number of pump strokes.

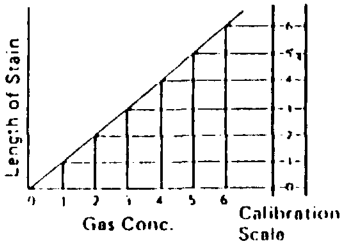


Fig. 1

Since all of the Gastec tubes are direct reading, the calibration scales are printed on the basis of individual production lots. Therefore, the variation of inner tube diameters, precision of tube packing, and the quality and reactivity of each reagent are eliminated. There is no need for extra charts or graphs whose accuracy is questionable for anything other than a single production run.

Certification of Gas Detector Tube Units

The National Institute for Occupational Safety and Health (NIOSH) has established a testing and certification program to insure that devices and instruments used in assessing and protecting against health hazards meet acceptable performance requirements. The Occupational Safety and Health Act (1970) requires the use of certified equipment unless no such equipment exists. At this point, detector tubes and sound level meters are the only instruments being tested under this program.

Following are the details of the test standard as published in the Federal Register dated May 8, 1973:

1. Every manufacturer must submit the quality control documentation including the quality control system of the company, the quality control program and the quality control plan of the measuring apparatus to the Quality Control Section of TCL prior to the certification test. The applicant will be granted its basic qualification by the Section after the documentation check.
2. In accordance with the Federal Register, the applicant must submit the quality control documents for the detector tube to be tested, with test results certifying that the tubes satisfy the standard, and an appropriate number of tubes. After passing this check, the applicant may proceed to the next step.
3. The applicant submits the detector tubes for inspection. The inspection procedure is divided into two parts:
   1) physical inspection tests such as the longitudinal axis of glass tubes, detecting layer and calibration scale and
   2) chemical reaction tests for indicating accuracy with standard gas mixture.

   Physical inspection tests are performed during a sampling inspection in accordance with Inspection Level II as described in MIL-STD-105D. The tubes which pass the above inspection proceed to the Air Sampling Section and are tested for accuracy.
4. The accuracy test is conducted on four concentrations; one-half, one, two and five times the TLV in accordance with the Inspection Level IV as described in MIL-STD-104.

   The standard gas generation method and the standard method of analysis for the test are verified in the Microanalysis Laboratory of the National Bureau of Standards; then the concentration of the gas is confirmed by the Gas Detector Tube Standard Committee. The detector tube stained by the standard gas concentration is read by the three (3) independent tube readers. (They will read 4 end points of circumference on the longitudinal axis.) The results of the tube reading are put into the computer, and the standard deviation percentages are then calculated.
5. To be certified by NIOSH, the detector tube must satisfy the following test standards:
   1) For the accuracy test, gas detector tube units shall produce measurements of contaminant concentration with ±35% of the actual value at one-half the test standard and within ±25% of the actual value at 1, 2 and 5 times the test standard.
   2) The standard deviation of the tube readings obtained from three independent tube readers shall be within 10%.
   3) On channelling of airflow through the detector tube, the maximum variation of stain length around the circumference of the tube at the interface between stained and unstained reagents shall be within 20%.
6. Detector tubes certified under this program will be published in the Federal Register at that time, the certification number will be issued, and the government will place the item on the approved list. Thereafter, NIOSH may purchase the tubes from the market at random and test them to determine if the tubes continue to meet these exacting standards. Tubes which fail to meet the test standard will have the certification withdrawn until the defect is corrected. The following Gastec Detector Tubes have been certified by NIOSH and issued the certification number:

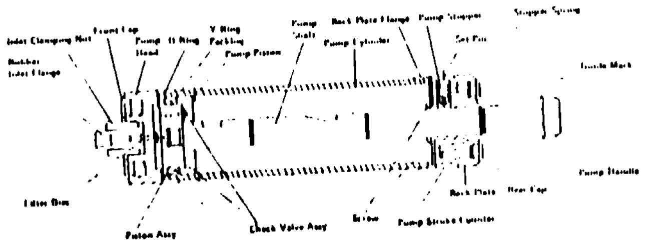
NOTE: NIOSH Certification Program has been discontinued.

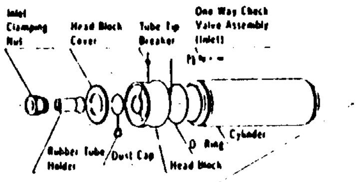
Tube No. 1La	Carbon Monixide	TC-84-014
Tube No. 5La	Sulfur Dioxide	TC-84-017
Tube No. 9L	Nitrogen Dioxide	TC-84-018
Tube No. 4LL	Hydrogen Sulfide	TC-84-020
Tube No. 2L	Carbon Dioxide	TC-84-021
Tube No. 3M	Ammonia	TC-84-023
Tube No. 134	Carbon Tetrachloride	TC-84-036
Tube No. 132H	Trichloroethylene	TC-84-038
Tube No. 8La	Chlorine	TC-84-041
Tube No. 121	Benzene	TC-84-043
Tube No. 10	Nitric Oxide	TC-84-049
Tube No. 122	Toluene	TC-84-053
Tube No. 131La	Vinyl Chloride	TC-84-060
Tube No. 12L	Hydrogen Cyanide	TC-84-068

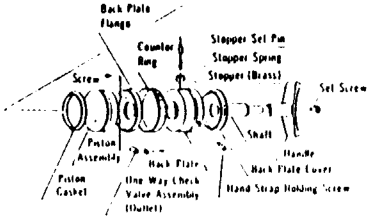
5. Gastec Pump Performance

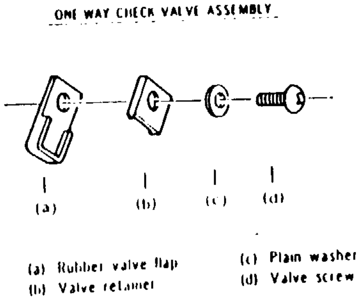
Description of Pump

Construction of the Gastec pump is illustrated below. This pump pulls the highest vacuum for its type (8.1" of Hg). There are no flow-rate orifices to cause malfunction of the pump by clogging or leaking. A friction-proof piston gasket (lubricant seal packing) provides complete leakproof sampling at all times.









(a) Rubber valve flap
(b) Valve retainer
(c) Plain washer
(d) Valve screw

Checking Pump Performance

A. Visually check rubber inlet flange for cracks or tears. Replace if damaged. Tighten inlet clamping out.

B. Valve Leak Check

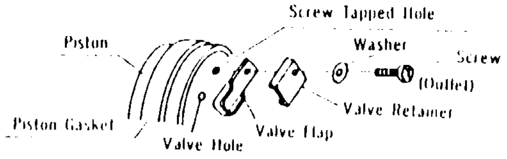
1. Insert a fresh sealed detector tube into pump. Misalign red dots on pump and handle. Pull several fairly rapid continuous full pump strokes.
2. Pull handle out 6 mm (1/4 inch) and hold in this position for 1 or 2 seconds.
3. Release handle.
4. If handle return to within 1.5 mm (1/16 inch) or less of fully closed position, continue to step C.
5. If handle does not return to within 1.5 mm (1/16 inch) of fully closed position (or less), perform the following Valve Lubrication instruction outlined below.

C. Field Volume Check

1. Insert a fresh sealed detector tube into pump.
2. Align Guide Marks on pump body and handle.
3. Pull handle firmly and at a moderate speed until handle locks into position. Wait 1 minute.
4. Unlock handle by turning it and guide it back.
   TO PROTECT PUMP STOPPER from breakage, do not release the handle and allow it to spring back when conducting a leak test. Make sure you hold your hand onto the handle and guide it back.
5. Pump handle should return to within 6 mm (1/4 inch) or less of the fully closed position.
6. If pump handle does not close to within 6 mm (1/4 inch) or less, follow lubrication instructions and retest.

D. Lubrication Instructions (Perform Laboratory Volume Check "E" after each lubrication)

1. Valve Lubrication
   a. Unscrew back plate and withdraw piston from pump cylinder.
   b. Remove check valve from piston.
   c. Clean valve and piston with lint-free cloth. Proper valve cleaning is as follows:
      Place cloth flat on desk.
      Wipe rubber valve flap in a flat position across cloth.
      Do not bend the rubber flap valve.
   d. Apply a small amount of grease evenly around the valve opening to form a thin film. A thin film is nearly invisible.
   e. Replace valve assembly loosely in the same manner as removed.

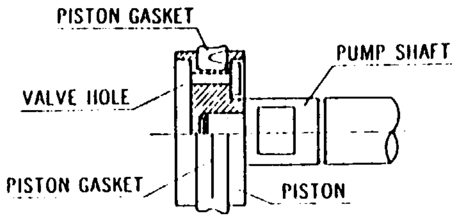


f. Before tightening the screw, align [illegible] so th[illegible] [illegible]e hol[illegible] cent[illegible] [illegible]lve flap.

g. Then push the rectangular valve retainer all the way toward loose end of valve falp.

h. Now tighten screw. If a torque driver is available, tighten to 0.8 Kg-cm. Otherwise, be careful not to overtighten screw. When tightened, screw must not deform rectangular valve retainer.

2. Piston Gasket Lubrication
   a. Wipe off piston and cylinder with a clean lint-free cloth.
   b. Remove piston gasket with a small bladed screwdriver. Take care not to cut gasket.
   c. Clean slot in piston with lint-free cloth. Wipe off rubber gasket.
   d. Wipe an ample supply of grease into gasket slot on piston and inside gasket.
   e. Replace gasket making sure that open side of gasket is toward pump handle.



   f. With the excess grease from piston slot, wipe around outside of gasket and piston.
   g. Wipe an ample amount of grease into cylinder at the area of piston entrance.
   h. Insert piston slowly into the cylinder. Work the piston back and forth slowly in the cylinder several times.
   i. Now screw back plate firmly onto cylinder.
   j. Repeat leak tests.
   k. If any leak remains, replace piston gasket.
   l. Only if a leak persists, go to procedure below.

3. Pump Head Lubrication
   a. This is only necessary where all previous procedures have failed to correct a leak.
   b. Visually check pump head "O" ring for cracks.
   c. Replace "O" ring if cracked.
   d. Place a light coat of grease on pump cylinder head screw threads and the "O" ring.
   e. Insert new "O" ring.
   f. Screw pump head firmly on to "O" ring and make sure "O" ring is seated uniformly. Overtightening pump head may push "O" ring out of place. Do not overtighten.
   g. Wipe off excess grease.

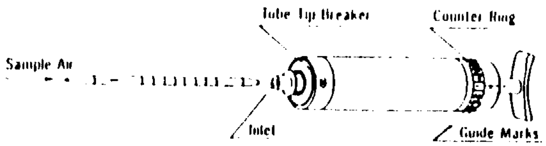
[illegible] Vol [illegible]

The Gastec pump can be checked periodically to assure that 100 ±5 ml are being sampled.

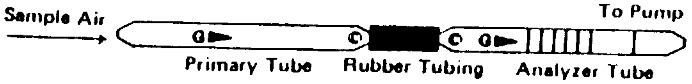
1. Arrange a graduated 100 ml soap film flow meter in a volume test mode.
2. Insert a fresh Gastec tube into the pump. The tube must be broken at both ends (ready for use).
3. Attach the Gastec tube to top of soap film flow meter with rubber hose. Make sure there are no leaks.
4. Pull pump handle out full to lock at one stroke in normal sampling manner.
5. Wait until the bubble stops moving and read the volume evacuated.
6. If the volume evacuated is other than 100 ± 5 ml, proceed to lubrication instruction and retest.

6. Operating Procedure for Gastec Precision Gas Detector System

Sampling & Measurement Procedure



1. Break tips off a fresh detector tube by bending each tube end in the tube tip breaker of the pump.
2. Insert tube securely into pump inlet with arrow on tube pointing toward pump.
3. For twin tubes, connect (c) marked ends with rubber tubing after breaking each end. Insert analyzer tube into pump with arrows on tubes pointing toward pump. See figure below.



4. Make certain pump handle is all the way in. Align guide marks on pump body and handle.
5. Pull handle out to desired stroke volume. Handle can be locked on either 1/2 pump stroke (50 ml) or 1 pump stroke (100 ml).
6. Read concentration at the interface of stained-to-unstained reagent when staining stops. Unlock handle by making 1/4 turn and return it to starting position.
7. In case more pump strokes are indicated in the instruction sheet included in each box of tubes, take additional sample by repeating pump strokes without removing tube.

Correction for Temperature, Humidity or Pressure:

Calibration of the Gastec detector tubes is based on a tube temperature of 20°C

(68° F), approximately 50% relative humidity, and normal atmospheric pressure.

1. No correction is normally required for tube temperature of 0° – 40°C (32° – 104° F) and for relative humidity range of 20 – 90%.
2. Where the detecting reagent is abnormally sensitive to temperature or humidity, a correction table or chart is provided in each box of tubes. In this case, the tube reading must be corrected using correction table or chart.
3. *Tube reading is proportional to absolute pressure. To correct for pressure,* multiply by

$$\frac{760}{\text{Atmospheric Pressure (mmHg)}}$$

How to Use Counter Ring:
In case numerous pump strokes are required, use the counter ring of the pump.
(1) Turn the counter ring manually counterclockwise after each pump stroke.
(2) *The operator may thus keep count of the number of pump strokes.*

For more details see the instruction sheet included in each box of detector tubes.
Detector tube should be stored in accordance with instructions on each box.

7. Different Methods of Sampling

1. Effect of sampling time and related data

Concentration of contaminants and chemical substances in the air is always fluctuating. On the other hand, sampling is usually conducted within certain time and frequency limits. *Therefore, a deviation naturally exists between the average environmental concentration and* results obtained on the basis of "grab" samples.

For example, Fig. 2 below shows time-lapse variation of dust concentration which was observed by using Tyndallometer readings plotted in 30 second intervals. This test was conducted at a raw material mixing plant in the glass mill.

The variation of concentration during the hour was up to 60 times the average concentration of dust. This idea can be adapted to the gas concentration in air of toxic *substances as follows:*

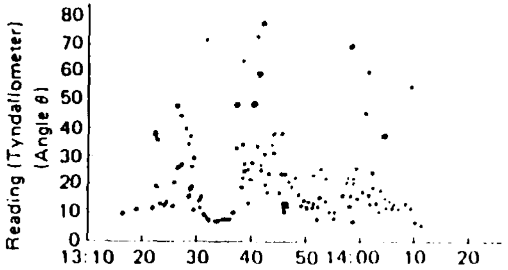


Fig. 2 Time-lapse Variation of Dust Concentration
(Place: Raw material mixing plant in glass mill)
by Tyndallometer

The variation of the measured concentration is largely dependent on the sampling time and position. Large differences occur when the operation is intermittent. If the sampling time is short and the quantity of sample gas or vapor is relatively small, large variations will result between instantaneous concentration and average concentration. Under extremely adverse conditions, there can be as much as ten fold difference in detector tube readings taken at different times in the same location.

2. Tanks, Manholes, or Closed Places

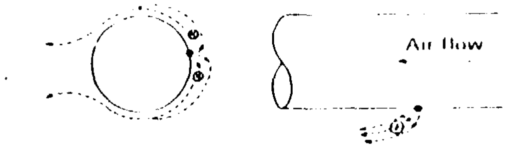
It is dangerous to enter a manhole directly without a pre-check. Before entering any such closed place, the workman must first check the gas level by using an extension hose in at least three levels, that is the higher, middle, and lower level of the closed area. The reason for this is as follows:

$$\text{Specific Gravity of Gas} = \frac{\text{Molecular Weight of Gas}}{\text{29 (Average MW of Air)}}$$

If the total fraction is greater than 1.0, it means the gas will rise to the top. However, if the total fraction equals more than 1.0, it means the gas concentration is increasing at the lower level.

3. Leak-check from Pipeline

If an air flow or a breeze is observed prior to taking the measurement, measuring points must be moved away from the direction of the air flow.



◄- - - - - : Gas flow
⊗ : Measuring points

Fig. 3

Caution: If the measurement is conducted at a point some distance from the pipeline, the gas emitted will be diluted very rapidly, and the true concentration of the gas cannot accurately be detected. Therefore, measurement must be *conducted at a point as close as possible to the suspected leak.*

4. Measurement in a Chemical Plant, Gas Concentration in Pipelines

It is sometimes necessary to collect the gas sample temporarily in a sampling bag or a bottle as shown in the two examples below. Measurement of the concentration is made by passing a sample through an appropriate Gastec tube.

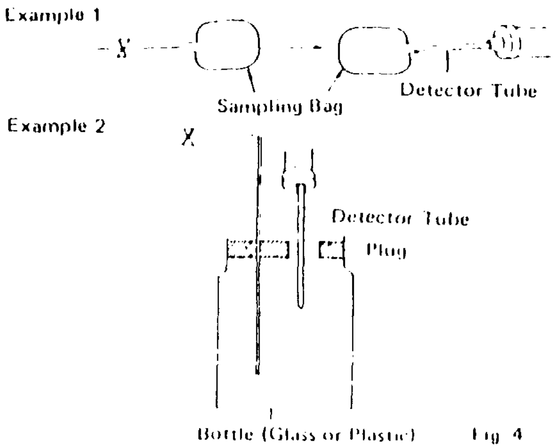


Fig. 4

5. Measurement and Threshold Limit Value of Liquid Mixture

A rapid reliable method for measuring concentration of industrial solvents in air is available using the Gastec Precision Gas Detector System.

5-1 Elements Evaporating from Solvent

Industrial solvents are liquids but have an appreciable vapor pressure at room temperature. The composition of the vapor arising from the solvent can be quite different from the composition of the solvent itself.

Take, as an example, a lacquer thinner mixture of 66% of toluene, 14% each of ethyl acetate and butyl acetate, 4% of butyl alcohol and 2% of cellsolve. At the early stage of its evaporation, the lower-boiling point metarial vaporize.

If a coating plant uses the above thinner, the thinner will vaporize according to the chart below.

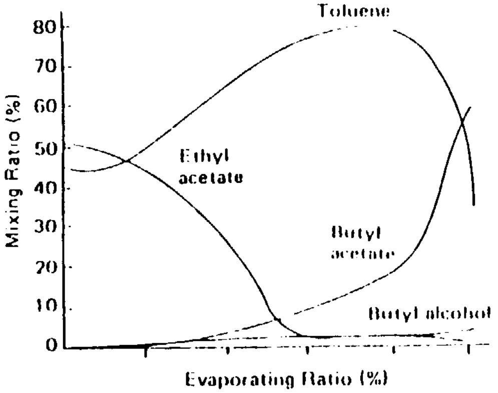


Fig. 5 Evaporation from Mixing Solvent

The ratio of vaporized components is greatly dependent upon the working environment even if the same solvents are used. Therefore we must carefully consider the environmental assessment of exposure to complex mixtures. Many lacquer thinner manufacturers keep the ratio of materials secret. However, OSHA has legislated that each producer must publish the principal components and hazardous properties of all proprietary solvents in Material Safety Data Sheets.

5-2 Selecting Measuring Apparatus

One of the best methods used in measuring each component of a solvent mixture is by means of gas chromatography. However, a simpler analysis using detector tubes is usually preferred for daily environmental measurement. Use of detector tubes and selection of tubes is greatly dependent on the individual case. It is recommended that a specialist be consulted for correct interpretation of the results. Items such as interferences and reaction rates have a major effect on the accuracy and precision of the measurement.

5-3 Threshold Limit Value for Mixtures

When two or more hazardous substances are present, their combined effect, rather than that of either individually, would be given primary consideration. In the absence of information to the contrary, the effects of the different hazards should be considered as additive. That is, if the sum of the following fractions,

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_n}{T_n} = 1$$

exceeds one, then the threshold limit of the mixture should be considered as being exceeded. C_1 indicates the observed atmospheric concentration, and T_1 the corresponding threshold limit (see example below).

It is essential that the atmosphere be analyzed both qualitatively and quantitatively for each component present, in order to evaluate compliance or noncompliance with this calculated TLV.

$$\frac{C_1}{T_1} + \frac{C_2}{T_2} + \cdots\cdots \frac{C_3}{T_3} + \cdots\cdots = 1$$

Example: Air contains 5 ppm of carbontetrachloride (TLV = 10 ppm)
20 ppm of 1, 2-dichloroethane (TLV = 50 ppm) and 10 ppm of
1, 2-dibromoethane (TLV = 20 ppm)

Atmospheric concentration of mixture = 5 + 20 + 10 = 35 ppm of mixture

$$\frac{5}{10} + \frac{20}{50} + \frac{10}{20} = \frac{25+20+25}{50} = 1.4$$

Threshold limit is exceeded. Furthermore, the TLV of this mixture may be calculated by reducing the fraction to 1.0; i.e.

$$\text{TLV of mixture} = \frac{35}{1.4} = 25 \text{ ppm}$$

When a given operation or process characteristically emits a number of harmful dusts, fumes, vapors, or gases, it will frequently be only feasible to attempt to evaluate the hazard by measurement of a single substance. In such cases, the threshold limit used for this substance should be reduced by a suitable factor, the magnitude of which will depend on the number, toxicity and relative quantity of the other contaminants ordinarily present.

Examples of processes which are typically associated with two or more harmful atmospheric contaminants are welding, automobile repair, blasting, painting, lacquering, certain foundry operations, diesel exhausts, etc.

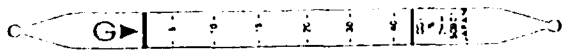
II Gastec Detector Tubes

These definitions apply to each of the following pages.

1. Performance of the Tube (n: Number of Pump Strokes)
2. Detecting Principle of the Gas
3. Interferences of the Tube
4. Method of Standard Gas and Analysis
    - 4.1 Standard Gas and Analysis
    - 4.2 Method of Analysis
5. Dangerous and Hazardous Properties
    - 5.1 T.L.V.-T.W.A.: The timeweighted average concentration for a normal 8-hour workday or 40-hour workweek, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect.
      Threshold Limit Value-Time Weighted Average for 1985 adopted by American Conference of Governmental Industrial Hygienists.
    - 5-2 T.L.V.-STEL: The maximum concentration to which workers can be exposed for a period up to 15 minutes continuously without suffering from 1) *introlerable irritation,* 2) chronic or irreversible tissue change, or 3) narcosis of sufficient degree to increase accident proneness, impair self-rescue, or materially reduce work efficiency, provided that no more than four excursions per day are permitted, with at least 60 minutes between exposure periods, and provided that the daily TLV-TWA also is not exceeded.
      Threshold Limit Value-Short Term Exposure Limit for 1985 adopted by American Conference of Governmental Industrial Hygienists.
    - 5-3 F.L.: Flammable Limits in Air (lower and upper limits)
6. Physical Constants
    - 6-1 C.F.: Chemical Formula
    - 6-2 M.W.: Molecular Weight
    - 6-3 S.G.: Specific Gravity, Gas @20°C (68°F), NTP 1 atm unless otherwise stated
    - 6-4 M.P.: Melting Point
    - 6-5 B.P.: Boiling Point
    - 6-6 AIT: Autoignition Temperature
    - 6-7 V.P.: Vapor Pressure (mmHg)
7. Application for other gases — Other gases for with the some detector tube may be used to detect.

CARBON DIOXIDE EXTRA HIGH RANGE TUBE | No. 2HH

1. Performance



Calibration Scale	5 - 40%	(n=1/2)	Color Change	White - Purple
Measuring Range	2.5 - 5% 5 - 40%	(n=1) (n=1/2)	Sampling Time	1 min./pump stroke
Detecting Limit	0.5%	(n=1)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazid, which discolors redox indicator (crystal violet).

$CO_2 + N_2H_4 — NH_2{\cdot}NH{\cdot}COOH$

3. INTERFERENCES:

Other gases and vapors do not affect the tube reading

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

T.L.V.: 5000 ppm
STEL: 15000 ppm
F.L.:

Concentration	Phisical Effects
2.5%	No effect for a few hours
10%	Can endure for a few minutes
12 – 15%	May cause death in exposures for several hours

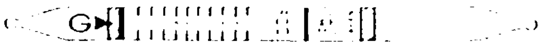
6. Physical Constants

C.F.: CO_2
M.W.: 44.01
S.G.: 1.53
M.P.: −56.6°C
B.P.:
AIT:
V.P.:

7. Application for other gases

CARBON DIOXIDE HIGH RANGE TUBE | No. 2H

1. Performance



Calibration Scale	1.0 - 10%	(n=1)	Color Change	White - Purple
Measuring Range	0.5 - 1.0% 1.0 - 10% 10 - 20%	(n=2) (n=1) (n=1/2)	Sampling Time	1 min./pump stroke
Detecting Limit	0.1%	(n=2)	Shelf Life	3 years

2. Detection Principle

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$CO_2 + N_2H_4 \rightarrow NH_2\,NH\,COOH$

3.

Interferences:
Other gases and Vapors do not affect the tube reading.

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system

4.2 Method of Analysis: Gas chromatography (TCD)

5. Dangerous & Hazardous Properties

T.L.V. = 5,000 ppm
STEL = 15,000 ppm
F.L. =

Concentration	Physical Effects
2.5%	No effect for a few hours
10%	Can endure for a few minutes
12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2
M.W.: 44.01
S.G.: 1.53
M.P.: −56.6°C
B.P.: −78.5°C
AIT:
V.P.:

7. Application for other gases

CARBON DIOXIDE LOW RANGE TUBE

No. 2L

1. **Performance**

Calibration Scale	0.25 – 3.0% (n=1)	Color Change	White · Purple
Measuring Range	0.13 – 0.25% (n=2) 0.25 – 3.0% (n=1) 3.0 – 6.0% (n=½)	Sampling Time	2 minutes/pump stroke
Detecting Limit	0.025% (n=2)	Shelf Life	3 years

2. ***Detection Principle***

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \longrightarrow NH_2 \cdot NH \cdot COOH$$

3.

Interferences:
Other gases and vapors do not affect the tube reading.

4. 4.1 Standard Gas Generation Method: Cylinder bottle gas & dynamic gas flow system
4.2 Method of Analysis: Gas chromatography (TCD)

5.

Dangerous & Hazardous Properties	Concentration	Physical Effects
T.L.V. = 5000 ppm	2.5%	No effect for a few hours
STEL = 15000 ppm	10%	Can endure for a few minutes
F.L. =	12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: -56.6°C B.P.:
AIT: V.P.:

7. Application for other gases

CARBON DIOXIDE EXTRA LOW RANGE TUBE

No. 2LL

1. **Performance**

Calibration Scale	300-5000 ppm (n=1)	Color Change	White Purple
Measuring Range	100 - 300 ppm (n=3) 300-5000 ppm (n=1) 4600-11500ppm(n=1/2)	Sampling Time	2 min./pump stroke
Detecting Limit	30 ppm (n=1)	Shelf Life	3 years

2. **Detection Principle**

Carbon dioxide reacts with hydrazine to form carbonic acid monohydrazide, which discolors redox indicator (crystal violet).

$$CO_2 + N_2H_4 \rightarrow NH_2 \cdot NH \cdot COOH$$

3.

Interferences	Concentration	Result	Comment
Ammonia	Up to 1,000 ppm	No effect	At more than 1,000 ppm gives minus error
Carbon monoxide	Up to 500 ppm	"	
Sulfur dioxide	Up to 30 ppm	"	
Nitrogen dioxide	Up to 30 ppm	"	
Chlorine	Up to 20 ppm	"	

4. 4.1 *Standard Gas Generation Methods: Cylinder bottle gas & dynamic gas flow system*
4.2 Method of Analysis: Gas chromatography (TCD)

5.

Dangerous & Hazardous Properties	*Concentration*	*Physical Effects*
T.L.V. = 5000 ppm	2.5%	No effect for a few hours
STEL = 15000 ppm	10%	Can endure for a few minutes
F.L. =	12 – 15%	May cause death in exposures for several hours

6. Physical Constants

C.F.: CO_2 M.W.: 44.01 S.G.: 1.53 M.P.: -56.6°C B.P.:
AIT: V.P.:

7. Application for other gases

APPENDIX C-8

TOTAL VOCS
(HNU)

INSTRUMENT OPERATION PROCEDURE

HNu Model PI101 Photoionization Detector

For the Detection of Organic Vapors
Through Photoionization

January, 1991

INSTRUMENT OPERATING PROCEDURE
HNU TRACE GAS ANALYZER MODEL PI 101

SCOPE AND APPLICATION

The Trace Gas Analyzer (See Figure 1), is a portable instrument used to detect, measure, and provide a direct reading of the concentration of a variety of toxic gases in the air. The analyzer employs the principle of photoionization. The sensor consists of a sealed ultraviolet light (UV) source that emits photons with an energy level high enough to ionize many trace species, particularly organics.

The analyzer consists of a probe, a readout assembly, and a battery charger. The analyzer has a concentration range from 0 PPM to 2000 PPM.

Reference:
Instruction Manual Portable Photoionization Analyzer Model PI 101.

Apparatus:
HNu Model PI 101

Battery Charger

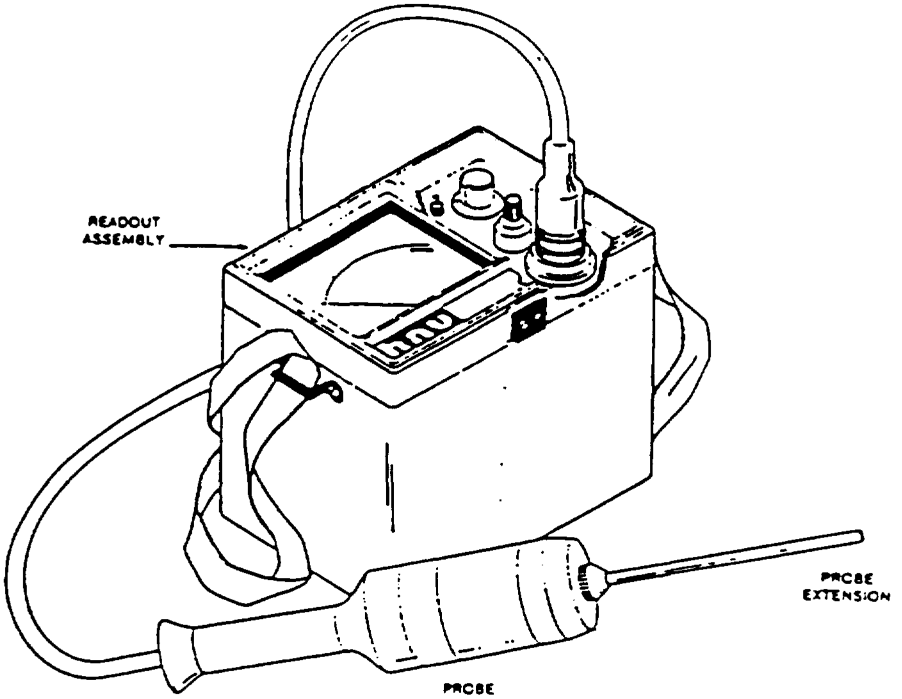
Probe options: 9.5 eV, 10.2 eV or a 11.7 eV UV lamps.

Calibration Kit - Kit containing a gas cylinder of isobutylene at concentrations of 52.1 PPM and 59.7 PPM (check the concentration marked in the cylinder provided) for the 10.2 and 11.7 eV probe respectively, and a gas regulator.

1.0 DETAILED DESCRIPTION

1.1 Meter

Indications of the instrument are displayed on a meter, visible through a window on the top face of the instrument case (See Figure 2). A mark on the scale, "BATT CK", represents the minimum permissible battery voltage, as an indication of the state-of-charge of the battery. The meter face has a scale from 0 to 20.



TRACE GAS ANALYZER

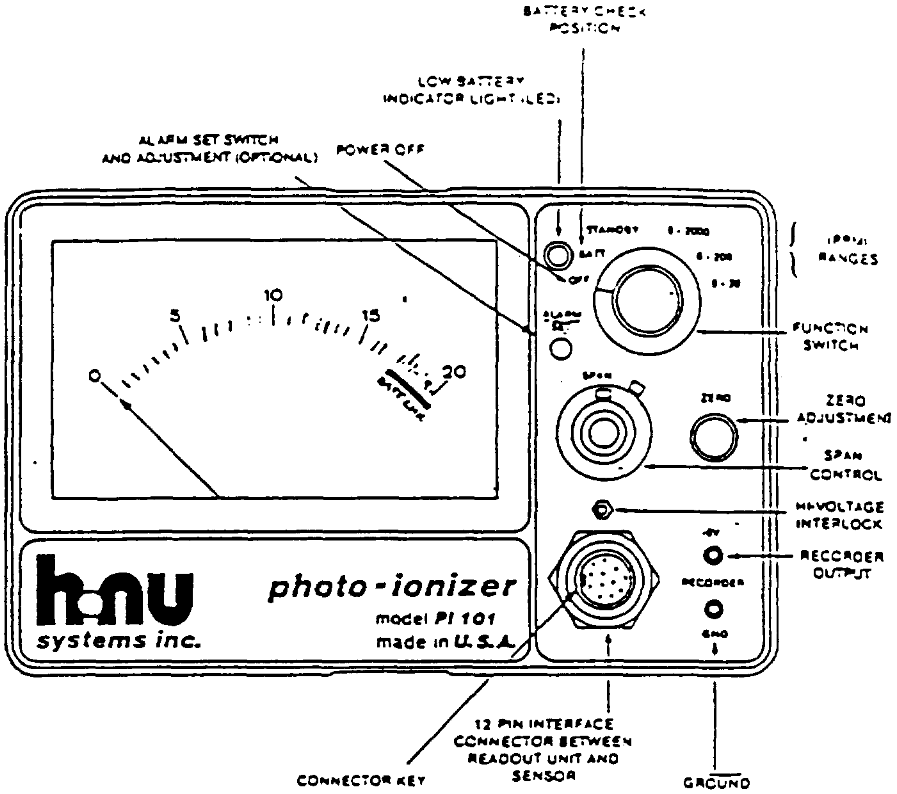
FIGURE 1

1.2 Controls and Indicators

The control and indicators are located on the front panel of the readout assembly (See Figure 2) and are as follows:

1.2.1 FUNCTION switch, a switch with six functions: OFF, BATT, STANDBY, and ranges of 0-2000, 0-200, and 0-20.

In the OFF position all operations are OFF; in the BATT position, checks the condition of the battery; in the STANDBY, all electronics are ON but the UV light source is OFF.



CONTROLS AND INDICATORS

FIGURE 2

1.2.2 ZERO adjustment, with the Function Switch in the STANDBY position, this potentiometer is used to adjust the reading to zero.

1.2.3 SPAN, this vernier potentiometer is used to set the gain of the amplifier to give direct readings of the trace gas concentrations in ppm. The whole number of the setting appears in the window of the control, decimal appears on the dial. A lock secures it at a specific setting.

1.2.4 HI-VOLTAGE, this is a normally open microswitch. Switch is open when cable not connected. Switch is automatically closed when the cable is attached. This switch may also be closed manually during maintenance checks of the readout assembly without the probe cable attached.

1.2.5 ALARM SET (optional), a potentiometer that turns the audible alarm ON and OFF and sets the ppm level at which the alarm sounds.

1.2.6 LOW BATTERY INDICATOR LIGHT, illuminates when the battery is discharged, indicates need for recharge. Readings may be taken while the unit is being recharged.

1.2.7 RECORDER (optional), provides a record of readings while analyzer operates unattended.

2.0 OPERATING PROCEDURES

The following procedures are to be used in operating the analyzer:

2.1 Unclamp the cover from the main readout assembly.

2.2 Attach the handle to the front part of the probe.

2.3 Connect the probe cable plug to the 12 pin keyed socket on the readout assembly panel (Figure 1). Carefully match the alignment slot in the plug to the key in the connector. Screw down the probe connector until a distinct snap and lock is felt.

2.4 Screw the probe extension into the probe end cap. The probe may be used without the extension if desired.

2.5 The SPAN control is set when the instrument is calibrated, refer to Section 3 below. Do not move this knob unless you are calibrating the instrument.

2.6 Turn the function switch to the BATT (battery check) position. The needle on the meter will go to the green zone if the battery is fully charged. If the needle is below the green arc or the Low Battery Indicator comes on, the battery must be recharged before the analyzer is used.

2.7 Turn the function switch to the STANDBY position. Turn the zero adjustment until the meter needle is at zero.

2.8 Turn the function switch to the appropriate operating position. Start with the 0-2000 ppm position and then switch to the more sensitive ranges. The UV light should be ON, confirmed by briefly looking into the probe to observe the purple glow from the lamp.

WARNING

Do not look at the light source closer than 6 inches with unprotected eyes.

Check that the fan is working properly by using a "Sharpie" marking pen; put the probe extension close to the tip of the pen and observe the needle deflect to the right. If the fan is not working return analyzer for repair.

2.9 The analyzer is now operational.

2.10 Hold the probe so that the extension is at the point where the measurement is to be made

---

WARNING

The instrument measures gases in the vicinity of the operator working and breathing zone and a high reading when measuring toxic or explosive gases should be cause for action for operator safety.

2.11 Take the readings as desired taking into account that air currents, or drafts or power lines in the vicinity of the probe tip may cause fluctuations in readings. Change the ranges as required.

2.12 After completion of use, check battery condition as described in paragraph 2.6.

2.13 Turn function switch to OFF position.

2.14 When not operating, leave analyzer in assembled condition, and connected to battery charger.

2.15 When transporting, disassemble probe and extension from readout assembly and return equipment to its stored condition.

2.16 Calibrate daily as indicated in Section 3.

3.0 CALIBRATION

The PI 101 Analyzer is calibrated at HNU Systems with certified standards of benzene, vinyl chloride and isobutylene. The analyzer is calibrated in the field with a certified standard of isobutylene. The analyzer is designed for operation at ambient conditions and therefore the gas standards used for calibration should be delivered to the analyzer at ambient temperatures and pressure and at the proper flow rate.

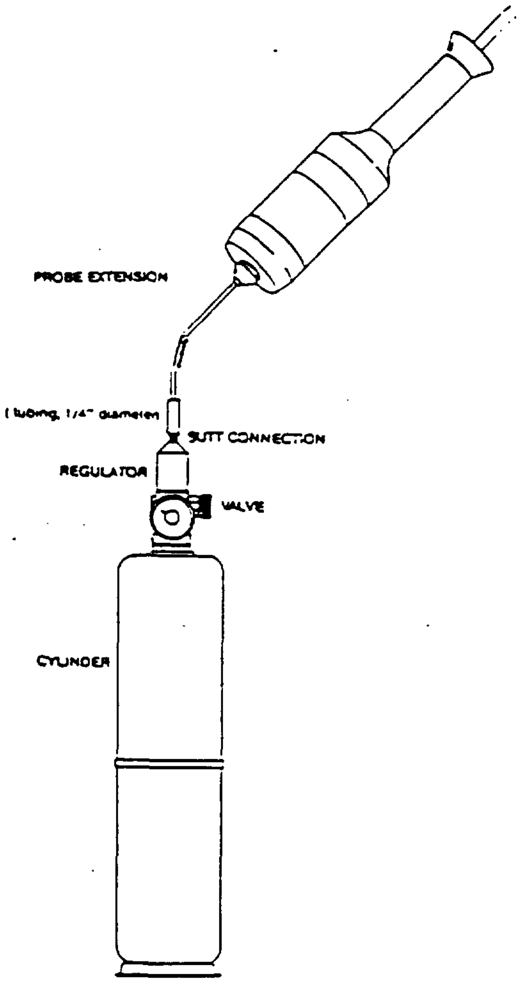
3.1 Calibration Procedure

3.1.1 Turn the function switch to BAT. The needle should be in the green region. If not, recharge the battery.

3.1.2 Turn the function switch to STANDBY. In this position the the lamp is OFF and no signal is generated. Set the zero point with the ZERO set control.

3.1.3 Turn the function switch to the 0 -20 or 0-200 range. Adjust the SPAN control setting to 9.8 when using the 10.2 eV lamp and to 5.0 when using the 11.7 eV lamp. For calibration on this range only one gas standard is required.

3.1.4 Connect the calibration gas as shown in Figure 3. The small cylinder included with the HNU unit contains Isobutylene gas standard. With the 10.2 eV lamp, the concentration should be 52.1 PPM and with the 11.7 eV lamp the concentration should be 59.7 PPM. NOTE; Normally the cylinders indicate what concentrations to expect; make sure to check this before calibration.



CALIBRATION TEST SET UP

FIGURE 3

Turn the function switch to the range position and note the meter reading. Adjust the SPAN control setting as required to read the ppm concentration of the standard. Recheck the zero setting (step 3.1.2). If readjustment is needed, repeat step 3.1.4.

3.1.5 For calibration on the 0-2000 range, use of two standards is recommended. First calibrate with the higher standard using the SPAN control for setting. Then calibrate with the lower standard using the ZERO adjustment. Repeat these several times to ensure that a good calibration is obtained.

3.1.6 If the span setting resulting from calibration is 0.0 or if calibration cannot be achieved, the lamp then must be cleaned (see section 4).

3.1.7 Shut off the cylinder as soon as the reading is established.

3.1.8 The analyzer is ready to take measurements.

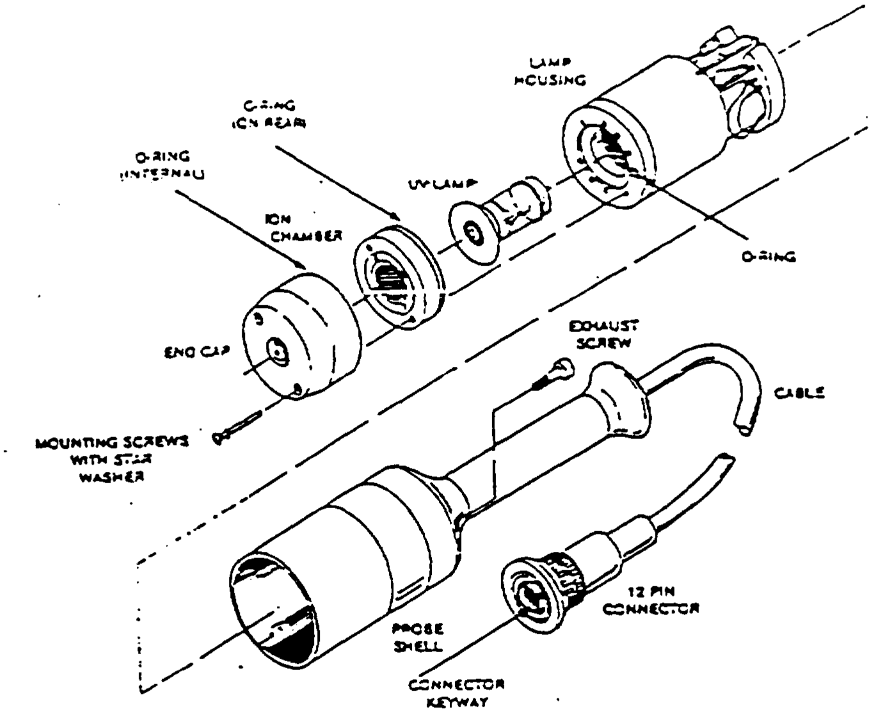
4.0 MAINTENANCE

Maintenance of the analyzer consists of cleaning the lamp, the ion chamber and the fan. During operation of the analyzer, dust, moist or other foreign matter can be drawn into the probe forming deposits on the surface of the UV lamp or in the ion chamber. This condition is indicated by meter readings that are low, erratic, unstable, non-repeatable, or drifting, or show apparent moisture sensitivity.

4.1. UV Lamp and Ion Chamber Cleaning.

Disassemble the probe by first removing the exhaust screw at the base of the probe adjacent to the handle (See Figure 4). Grasp the end cap in one hand and the probe shell in the other, gently pull to separate the end cap and the lamp housing from the shell.

Hold the lamp housing with the black end cap upright. Loosen the screws on the top of the end cap, separate the end cap and ion chamber from the lamp and lamp housing.



PROBE ASSEMBLY

FIGURE 4

CAUTION

Care must be taken so that the ion chamber does not fall out of the end cap or the light source does not fall out of the lamp housing.

Turn the end cap over in the hand. Tap lightly on the top. The ion chamber should fall out the end cap into the hand. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.

Exercise great care in doing so to prevent inadvertent damage to these components.

4.1.1 For the 9.5 and 10.2 eV lamps:

a. First clean by rubbing gently with lens tissue dipped in a detergent solution. If this does not remove deposit, apply a small amount of HNU cleaning compound (PA101534) directly onto the lens of the lamp and spread evenly over surface with a non-abrasive tissue.

b. Wipe off compound with a new tissue.

c. Rinse with warm water or damp tissue to remove all traces of grit or oils and any static charge that may have built up on the lens. Dry with a new tissue.

d. Reinstall lamp in the detector and check analyzer operation. If performance is still not satisfactory replace the lamp.

4.1.2 For the 11.7 eV lamp:

a. Clean by putting a freon or chlorinated organic solvent on a tissue and rubbing gently.

b. **DO NOT CLEAN THIS LAMP WITH WATER OR ANY WATER MISCIBLE SOLVENTS (such as methanol or acetone). IT WILL DAMAGE THE LAMP.**

c. **DO NOT USE THE CLEANING COMPOUND USED FOR THE 9.5 OR 10.2 eV LAMPS.**

4.2 Ion chamber Cleaning.

Inspect the ion chamber for dust or particulate deposits. If such a matter is present, the chamber can be cleaned by removing the outer Teflon ring, and the four screws holding the retaining ring. Carefully remove the retaining ring aside (Note: this ring is soldered) and remove the screen. A tissue or cotton swab, dry or wetted with methanol, can be used to clean any deposits. Let the liquid dry before reassembly.
Reassemble the probe and check analyzer operation. If performance is still not satisfactory replace the lamp.

4.3 Fan Cleaning

Remove the pogo contacts and the retaining screw shown in Figure 5.

a. Lift up top supporting fan and slowly pull fan out. CAUTION: the fan does not come out all the way; it is attached to the wires.

b. Blow into the fan to remove any dust particles. If the fan does not move freely, check for any larger particles that can be removed without damaging the impellor rotor or the blades.

c. Check for wiring connections at fan motor and at probe cable connector (J3 pins A and C).

Reassemble the probe by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the lamp housing, making sure that the contacts are properly aligned. The ion chamber fits only one way.

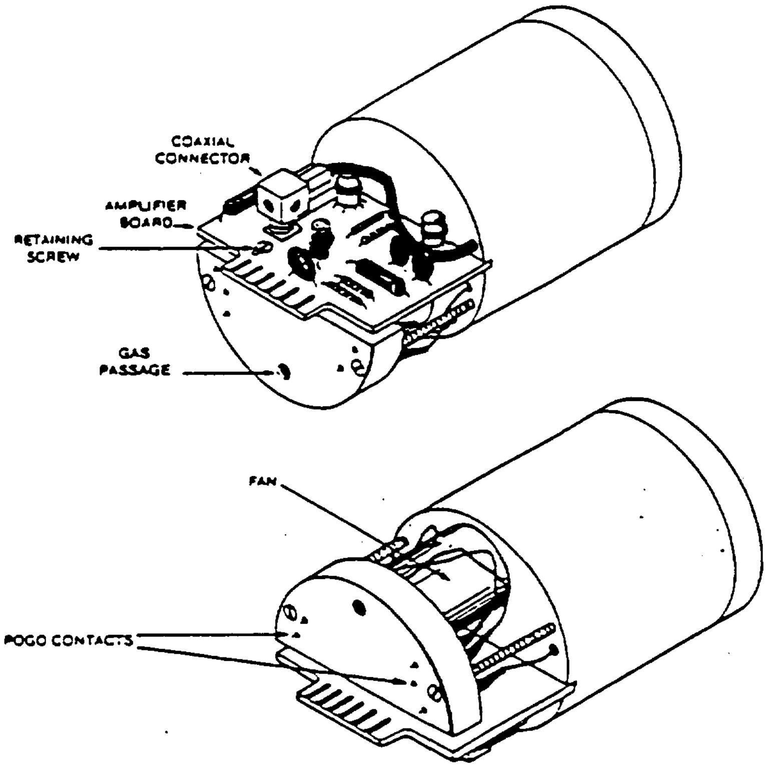
Reassemble the probe by first sliding the lamp back into the lamp housing. Place the ion chamber on top of the lamp housing, making sure that the contacts are properly aligned. The ion chamber fits only one way.

Place the end cap on top of the ion chamber and replace the two screws. Tighten the screws only enough to seal the O-ring.

CAUTION

Do not over-tighten these screws.

Line up the pins (pogo contacts) on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. The end cap should meet the probe shell evenly after final assembly. If not, the ion chamber may be installed wrong.



FAN/LIGHT SOURCE ASSEMBLY

FIGURE 5

CAUTION

DO NOT FORCE the assembly into the shell. It fits only one way.
If it does not reassemble readily, remove the check pin alignment, Check to ensure pogo contacts are not bent. Refasten the exhaust screw at the base of the probe.

APPENDIX D

LABORATORY CUSTODY SOPs

APPENDIX D-1

ETC

ETC Corporation Network Document

TITLE	ETC-Edison Log-In Procedure	Doc.#ED210003 Revision #3
Approval:	Lab Manager 11/2/90 QA Manager 11/2/90	Effective Date 11/1/90

1.0 SCOPE AND APPLICATION

1.1 Procedure is intended to assure proper receipt of samples and entry of sample receipt data into lab management system. This process is referred to as "logging in/log-in of samples".

1.2 Samples are received daily (Monday - Saturday) from both commercial carriers and clients. Samples may be received in either ETC Shuttles or in client provided containers. Special arrangements should be made for ETC Saturday receipt situations.

1.3 Upon receipt ETC must maintain custody and must maintain samples at a temperature of 4° +/- 2°C.

1.4 All receipts must be documented in a "loglink" log book. At a minimum the following information is required:

Carrier or courier identity
Client identity
Number of Shuttles (or client provided containers)
ETC sample number(s) received
Date of receipt
If a problem note(s) was required (Yes or No)

1.5 All deficiencies are documented on an "ETC Problem Note" or equivalent (Appendix 9).

1.6 Samples are logged in on an ETC "log-in" form which documents the number and type of bottles received, all order information, comments regarding the samples and any other information requested by the individual work group managers. Log-in forms may be generated through the ETC data base or manually.

1.7 An additional "Log-in form" is used for USEPA-CLP samples, which documents additional pieces of specific USEPA-CLP information for the case file. This form is used with and replaces in certain instances the standard problem note form.

2.0 SAFETY

2.1 Read ETC Safety Manual before proceeding.

2.2 Wear the following while working with any sample

2.2.1 Lab coat

2.2.2 Safety-shield glasses

2.2.3 Gloves, Solvex (Baxter G7197 or equivalent)

2.3 Wear ETC provided OSHA approved respirators as required.

2.4 Use ventilation hoods as required.

3.0 APPARATUS AND MATERIAL

3.1 Pliers

3.2 pH indicator strips, wide range (S/P #P1119-1A, or equivalent)

3.3 Capillary pipette (Kimble #71900, or equivalent)

3.4 Temperature monitoring device, calibrated

3.5 Clean work surface

3.6 Terminal and printer

3.7 Black ball point pen

4.0 PROCEDURE

CAUTION: DO NOT write on any custody document except as indicated on attachments. Use black ball point pens ONLY. These are legal documents which become part of the final report provided to the clients. Do not write on ETC "CC2" custody form.

4.1 Opening ETC Shuttles

4.1.1 Segregate Shuttles and client provided containers by workgroup. Determine work group priority. Notify supervisor if shipping containers cannot be assigned to a work group.

4.1.2 Reconcile number of Shuttles received with the corresponding airbill. If discrepancies exist, alert the Technical Project Manager (TPM) so that proper actions may be taken.

4.1.3 Note status of seal integrity. Open Shuttle by breaking seal with pliers. Remove all custody documents (CC1, CC2, NJDEP and any others provided by clients, Appendix 1, 2, 3). Document time and date of Shuttle opening, Shuttle identification number and custody seal number on custody form(s). If custody seal was not present write "NONE". Notify TPM and document on Problem Note.

4.1.4 Open unlabeled Shuttles first.

4.1.5 Insert temperature monitor into the Shuttle between the sleeve and the wall. Close the Shuttle. Allow reading to equilibrate.

4.1.6 Review custody form(s) for completeness (signature, date, seal number, etc.). Notify the TPM of all the comments made by the samplers.

4.1.7 Document integrity status of seal on custody form(s).

4.1.8 Reopen Shuttle if necessary to read temperature. Record measurement on custody form(s). Notify TPM if temperature measurement is NOT between 2-10°C and document on Problem Note.

4.1.9 Carefully remove bottles from Shuttle. Match ETC job numbers against information on custody form. Line up bottles by job number. Document nonconformances on Problem Note.

4.1.10 Check and record the condition of each bottle on the custody form(s). Document if headspace is present in TOX and VOA bottles; if bottles are empty or broken, etc. Notify TPM and document nonconformances on Problem Note.

4.1.11 Check pH of all samples which were to be preserved. DO NOT CHECK preserved VOA samples. For each sample remove a small aliquot using a new capillary pipet. Dispense aliquot on pH paper. Acid preserved samples should have a pH <2 and basic preserved samples should have a pH >12. Preservatives that should have been added at the time of sampling are on the CC1 custody form. Note observations on the custody form(s) by placing a check mark (" ") next to the preservative which was to be added. Notify TPM and document nonconformances on Problem Note.

4.1.12 Sign and date custody form(s) and air bills. Bring custody form(s), air bill, any other documentation and Problem Notes to TPM in log link jacket.

4.4.1 Log onto SYSA and access FT CSLIST.

4.4.2 Enter ETC job numbers as requested.

4.4.3 Check A-types on resultant CSLIST against the custody form(s) and bottles received (Appendix 7).

4.4.4 If a loglink number is present, add the new receipt to that link. (NOTE: presence indicates that the sample had previously been received or may be indicative of sampling problems.) Document on Problem Note and describe cause if known.

4.5 Generation of a log-in form (Appendix 8).

4.5.1 Log onto SYSA and access TR ELOGN.

4.5.2 Generate a separate log-in for each individual client, facility and turnaround time. Log-ins are referenced by the assigned loglink number (Step 1.4). Problem Notes must be initiated for all discrepancies.

4.5.3 Record air bill number. If air bill was not received enter "NONE".

4.5.4 Record matrix of each sample.

4.5.5 Record the analyses requested.

4.5.6 Assure that client name matches log-in.

4.5.7 Verify that actual sample matrices match those documented on the log-in. Enter the actual matrix in the appropriate area on the log-in. Use best judgement. Record discrepancies on Problem Note.

4.5.8 Record number of bottles received and analyses required.

4.5.9 Record presence or absence of client custody form(s), EPA or state traffic reports and/or SAS packing lists in comment section.

4.5.10 Document if samples are aliquotted. Aliquotting is necessary if sample bottles are received broken or additional analyses are requested after Shuttle

4.1.13 If shuttles are received empty.

1. Remove the contents for cleaning.
2. Complete custody form(s) as in Steps 4.1.3, 4.1.7, and 4.1.12.

3. Bring forms to TPM for follow-up action (drywell, cancellation, etc.).

4.2 Opening non-ETC Sample Shipping Containers

4.2.1 For each client identified sample, have TPM assign an ETC job number. Verify that client ID number(s) match custody information. Document discrepancies on a Problem Note.

4.2.2 Label client bottles with ETC Job number, analysis required, bottle number (for example, if 3 aliquots of one sample are received, label 1 of 3, 2 of 3, 3 of 3).

4.2.3 Initiate an ETC "Chain of Custody" form and any others required by clients (Appendix 5).

4.2.4 Proceed with Step 4.1.5.

4.2.5 Refer to Section 4.6 for receipt of USEPA CLP samples.

4.3 Receipt of Client Deliveries

4.3.1 Document receipt in "ETC Client Delivery" logbook. At a minimum record client identity, address and sample description. Logbook is shared by all work groups.

4.3.2 Request that client sign log book as the relinquisher.

4.3.3 Sign as receptor. Record date and time in the appropriate column.

4.3.4 Complete an ETC "Client Chain of Custody" and any forms required by the client (Appendix 6, 4). Provide a copy to the client.

4.3.5 Proceed with Step 4.1.5 or 4.2 as appropriate.

4.4 Generate a CSLIST (optional if automated log-in is unavailable). If automated log-in is available, go to Step 4.5.

shipment. Notify TPM before proceeding.

4.5.11 Summarize all problems on Problem Notes in the login comment section.

4.5.12 Secure ETC "Spec" Sheet(s) for sample(s). Record number on log-in.

4.5.13 Review log-in form upon completion. Sign log-in.

4.5.14 Color code (dot) bottle caps with the assigned loglink number.

4.5.15 Samples to be sent to subcontract labs.

1. Identify samples
2. Initiate a subcontract "Request for Analysis" form. This form serves as a custody document. Refer to Appendix 10.
3. Bring a copy of the "Request for Analysis" form to the TPM or designate.
4. Pack samples in an ETC Shuttle or prepare for pickup as appropriate. Include the original "Request for Analysis" form.

4.5.16 Place all samples in designated cold storage area(s).

4.5.17 Link samples into the system by logging on SYSA and accessing TR LOGIN. Enter the following when requested:

Operator ID: [Type first and last name initial and last four digits of social security number]

Edit All N/Y: [N] <R>
R-Date N/Y: [Y] <R>
Due-Date N/Y: [Y] <R>
F-Code N/Y: [N] <R>
Next Loglink: [Type in loglink number] <R>
Next Jobnum: [type in job number] <R>
Red'd Date: [Type in accurate sample receipt (YYMMDD)] <R>
Due-Date: [type in due date from the top left hand corner of the screen] <R>
Up-Date: [Y]

4.5.18 If samples are from NJDEP, initiate "DEP-077" form (Internal Chain of Custody). Refer to SOP SMS01600.ED.

4.5.19 Attach ETC "Spec" sheets and NJDEP "DEP-077" form (s) to log-in. Circulate original log-in and copies as directed by each work group. Hand deliver all EMERG turnaround log-ins directly to the responsible individuals for each analytical cost center. Copies of all log-ins must go to Planning and Project Services in addition to designated work group personnel.

4.6 Receipt of USEPA CLP Samples

4.6.1 A USEPA case is initiated by a telephone call from the Sample Management Office (SMO) to the designated ETC/Edison contact.

4.6.2 The information given by the SMO follows: contract #, bid lot (if applicable), case #, region, ship date(s), number of samples, matrix, level , and parameters.

4.6.3 The work group designated to analyze USEPA CLP samples will be notified; the original documentation of the SMO telephone contact will be retained for inclusion in the case file.

4.6.4 USEPA samples are shipped by overnight courier. At the time of courier delivery, the sample custodian verifies that the number of containers recieved matches the information on the airbill. The sample custodian signs the airbill with the date and time of delivery. The airbill is retained for inclusion in the case file.

4.6.5 Proceed with Sections 4.1.5 through 4.1.13, 4.2.1 through 4.2.3, Sections 4.4 and 4.5, and the following Sections 4.6.6 through 4.6.12.

4.6.6 The unopened ice chests are inspected for the presence or absence of intact custody seal(s), intact strapping tape, or evidence of damage. This information is recorded on the USEPA Traffic Report (TR) and othe documentation.

4.6.7 All samples should be listed on the TR present in the cooler(s). The TR is completed.

4.6.8 The "USEPA Sample Log-In" form is utilized to document additional pieces of specific information for the case.

4.6.9 If there are any questions or discrepancies noted on the receipt of the samples, the SMO is notified immediately by telephone by the designated ETC/EDison contact or technical project manager. A record of the clarification (which may be attached to the USEPA SAmple Log-In Form) is retained in the case file.

4.6.10 The receipt time and date stated on the airbill is the Verified Time of Sample Receipt (VTSR); all holding times start from this date.

4.6.11 The completed TR is returned to the SMO within 3 calendar days following receipt of the last sample in the sample delivery group. A copy of the TR is retained for the case file.

4.6.12 The empty ice chest is shipped back to the return address indicated within 14 days of case reciept at the facility.

5.0 SUPPLEMENTAL DOCUMENTS

5.1 SMS01600.ED - SOP for NJDEP Sample/Extract Internal Custody

5.2 GES00600.ED - SOP for Assignment of Log-Link Numbers at Edison.

6.0 REFERENCES

- NJDEP Contract X-408.
- USEPA CLP SOW for Organics, 2/88.

7.0 SUBSTANTIVE REVISIONS

- Revision 3 includes detail regarding receipt of USEPA CLP samples.

APPENDICES

Appendix 1 - ETC "CC1" form
Appendix 2 - ETC "CC2" form
Appendix 3 - NJDEP "DEP-060" form
Appendix 4 - NJDEP "DEP-009" form
Appendix 5 - ETC "Chain of Custody" form for non-ETC sample containers
Appendix 6 - ETC "Client Chain of Custody" for receipt of client deliveries
Appendix 7 - CSLIST report
Appendix 8 - ETC "Log-In" form
Appendix 9 - ETC "Problem Note"
Appendix 10 - ETC subcontract "Request for Analysis"
Appendix 11 - USEPA Traffic Report
Appendix 12 - ETC "USEPA Sample Log-In" Form.

APPENDICES

APPENDIX 1

[redacted]

ETC Environmental Testing and Certification Corp.

CHAIN OF CUSTODY FORM (CC1)

Seal No. 175719 ETC Job # -A3064

ORIGINAL Date Sealed 90/09/18 By: AB

Company: ×
Attn.: ×
Facility/Site: ×
Phone: ×
Address: ×

SAMPLE IDENTIFICATION

Facility: [redacted] (Facility/Site Code) (Optional Sample Point Descriptions)

Sample Point: ~~W~~-DUPLICATE | 900926 | 1415 |

Source Code (from below) X | Your Sample Point ID (left justify) | Start Date (YY/MM/DD) | Start Time (2400 hr clock) | Elapsed hours (composite)

WW 10/4/90

Source Codes:
Well (W) Outfall (O) Bottom Sediment (B) Surface Impoundment (I) Leachate Collection Sys. (C) Other (X)
Soil (S) River/Stream (R) Generation Point (G) Treatment Facility (T) Lake/Ocean (L) Specify

SHUTTLE CONTENTS

BOTTLE				ANALYSIS	SAMPLER		LAB
No	Type	Size	Preserv.		Filt. (Y/N)	Observations	Observations
2	UVOA	40	NONE	HPT/VOLATILES	N		✓
2	VOA	40	HCL	PT/VOLATILES	N		✓
1	VS	40	HCL	VOA SCREEN	N		✓ (4.1.10)
1	UTB	40	GC/MS	UNPRES TB	N		✓ (4.1.11)
1	TB	40	GC/MS	TRIP BLANK	N		✓
4	EXT	1000	NONE	Extractables	N		✓
1	MET	1000 ✓	HNO3	METALS	N		✓
2	SULF	125	✓ Z/NAOH	SULFIDES	N		✓
4.1.11							

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By: (print) SAMPLER Date: 9/19/90 Time: 1500
Signature: Sampler Seal #: 175719 Intact: yes

2. I have received these materials in good condition from the above person.
Name: Signature: SEAL # MUST MATCH
Date: Time: Remarks:

3. I have received these materials in good condition from the above person.
Name: Signature:
Date: Time: Remarks:

4. Shuttle Sealed By: (print) SAMPLER Date: 9/26/90 Time: 1435
Signature: Sampler Seal #: 0175720 Intact: yes

LAB USE ONLY Opened By: (4.1.12) Date: (4.1.3) Time: (4.1.3)
SHUTTLE # (4.1.3) TEMP. °C (4.1.8) SEAL # 175720 COND. (4.1.7)

APPENDIX 2

ETC

FIELD PARAMETER FORM (CC2)

Form 0002
Sample Management
10 89

ETC JOB # HA2064

Sample Point W DUPLICATE
Source Code   Sample Point ID

FIELD PROCEDURES

900926	0905	5	4.94	15.0
PURGE DATE (YY MM DD)	START PURGE (2400 Hr Clock)	ELAPSED HRS	WATER VOL IN CASING (Gallons)	VOLUME PURGED (Gallons)

SAMPLING METHOD: Stainless Steel Bailer

Sampler Type E — A-Submersible Pump, B-ISCO, C-Bladder Pump, D-Dipper/Bottle, E-Bailer, F-Scoop/Shovel, X-Other ____ (SPECIFY OTHER)

Sampler Material B — A-Teflon, B-Metal, C-PVC, D-Plastic, X-Other ____ (SPECIFY OTHER)

Tubing Material ☐ — A-Teflon, B-Tygon, C-Polyethylene, D-Silicon, X-Other ____ (SPECIFY OTHER)

Sample Composited Y/N NO
Procedure/Proportions

FIELD MEASUREMENTS

Well Elevation (ft/msl) ____

Depth to Ground water (ft) ____

Groundwater Elevation (ft msl) ____

Well Depth (ft) ____

Sample Depth (non-well) (ft) ____

1st	ph	(STD)	1st	spec. cond.	um/cm at 25°C	(other parameter)	value	units
2nd	ph	(STD)	2nd	spec. cond.	um/cm at 25°C	(other parameter)	value	units
3rd	ph	(STD)	3rd	spec. cond.	um/cm at 25°C	(other parameter)	value	units
4th	ph	(STD)	4th	spec. cond.	um/cm at 25°C	(other parameter)	value	units

Sample Temp (°C) ____

Turbidity NTU ____

FIELD COMMENTS

Sample Appearance: cloudy

Weather Conditions:

Other:

FILTERING: Use Chain of Custody (CC1) to indicate which bottles were filtered

Sampler: [redacted] (Print)

Employer: [redacted]

I certify that sampling procedures were in accordance with applicable EPA state and corporate protocols.

9/26/90 [redacted]
(Date) (Signature)

ORIGINAL

APPENDIX 3

Form DEP 060
10/86

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTECTION

CHAIN OF CUSTODY RECORD WITH SHUTTLE

Use One Form for Each Sample

Shaded Areas for DEP Field Use Only

NAME OF UNIT AND ADDRESS NJDEP

Name of Person(s) Preparing Container(s) for Sample Shuttle

Name [redacted] Title Sample Manager Agency ETC

Name Title Agency

Date above-mentioned Person(s) Placed Container(s) in Sample Shuttle 90/10/16 | Military Time 1520 | Laboratory Affixed Sample Shuttle Seal No. 0185429

Name of Person(s) Breaking Laboratory Seal on Sample Shuttle and Taking Field Sample

Name [redacted] Title Sr Env Specialist

Name [redacted] Title Env. Specialist

Name Title

Date Laboratory Sample Shuttle Seal No. 0185429 Broken 10/17/90 | Time (Mil) Seal Broken and Sampling Begun 0855

FIELD SAMPLE NUMBER	CONTAINER NUMBER(S)	VOLUME OF CONTAINERS	DESCRIPTION OF SAMPLE
BJR-042	2	40-ml VOA	
	2	500 ml EXT	
	2	125 ml	
	1	250 ml	
	1	40 ml TRIP BLANK	

LAB CONTROL NO.
HA3299 - Prior. Pll.
HA3302 - EPTox

Date Sampling Completed & Field Seal Affixed to Sample Shuttle 10/17/90 | Military Time 1100 | Field Affixed Sample Shuttle Seal No. 0185428

Laboratory Person Breaking Field Seal on Sample Shuttle & Accepting Responsibility for Sample Name (4.1.12) Title (4.1.12)

(4.1.3) Field Sample seal Seal No. 185428 | (4.1.3) Date Broken 10/17/90 | (4.1.3) Military Time Seal Broken 1430

SAMPLE RELINQUISHED BY	SAMPLE RECEIVED BY	DATE	TIME	REASON FOR CHANGE OF CUSTODY
[illegible] T. Seekinger	To ETC Shuttle	10/17/90	1100	ag 10/25/90
	[redacted]	10/17/90	~~230~~ 1430	
	(4.1.12)	(4.1.3)	(4.1.3)	

COPIES: *Gold - Container/Shuttle Preparation Receipt*
Yellow - Analyst Chain of Custody
Pink - Field Sampler Receipt
White - Sample Custodians Chain of Custody

APPENDIX 4

Form DEP 009
10/86

STATE OF NEW JERSEY
DEPARTMENT OF ENVIRONMENTAL PROTECTION
TRENTON, NEW JERSEY 08625

CHAIN OF CUSTODY WITHOUT SHUTTLE

I.D. # ____________________

NAME OF UNIT AND ADDRESS:

CLIENT NAME AND ADDRESS

SAMPLE NUMBER	Number of Containers	DESCRIPTION OF SAMPLES
CLIENT SAMPLE POINT ID	AS REQUIRED	MATRIX, DESCRIPTION OF CONTAINERS ETR JOB #

PERSON ASSUMING RESPONSIBILITY FOR SAMPLE:	TIME	DATE

SAMPLE NUMBER	RELINQUISHED BY:	RECEIVED BY:	TIME	DATE	REASON FOR CHANGE OF CUSTODY
CLIENT	CLIENT	ETR	AR	AR	DELIVERY TO LAB
SAMPLE	SIGNATURE	SIGNATURE			
POINT ID					

APPENDIX 5

ETC

1006

CHAIN OF CUSTODY

Company: NJDEP Job #

Address: TRENTON, NJ

Attention:

Sample Description:

CUSTOMER ID	DESCRIPTION	ETC #
Field Blank	Field Blank	#A3304
	(1) 1L Metal (1) 125 CY (1) 125 Phenol	
	(2) 1L EXTBL (2) 40ml VOA	
Trip Blank	(1) 40ml. TB	#A3301

Sample(s) Relinquished by: (CLIENT SIGNATURE / PRINTED NAME)

Time: (AS REQUIRED) Date: (AS REQUIRED)

Sample(s) Received by: (SAMPLE MANAGER)

Time: 230 Date: 10/17/96

721

i 78928

APPENDIX 6

ETC

CLIENT CHAIN OF CUSTODY

COMPANY: ETC Corporation
284 Raritan Center Parkway
Edison, NJ 08818

Samples Relinquished by: ____________________

Samples Received by: ____________________

Time: ____________________

Number of Shuttles: ____________________

APPENDIX 7

(4.4.4)

```
.............................................................................
 JOB#: BD4445  XRND: NORM  COMPANY:
 LOG#:                     FACILITY:

/PROGRAM ELEMENT 2a/ /SPECVOA/DM/RP--REPORT ONLY--BENZENE, METHYLENE CHLORIDE,/
/TETRACHLOROETHYLENE, TRICHLOROETHYLENE, TOLUENE,/ /1,1,1 TRICHLOROETHANE, MEK A
ND XYLENES ON GR01 AND/ /DM-1HC, KEN BAKER TO REVIEW PRIOR TO MAILING/ /FIELD/MI
SC.-STICKUP/

              ABRP                 ETCL/
              DM-1HC              -ETCL/
              DMPKG                ETCL/
              SPECDM               ETCL/
              SPECRP               ETCL/
              FIELD/TEMP           ETCL/
              FIELD/DTGW           ETCL/
              FIELD/GWE            ETCL/
              FIELD/MISC.          ETCL/
              FIELD/ELEV.          ETCL/
              FIELD/WELD           ETCL/
              1PP/VOA              ETCL/
              SPECVOA              ETCL/
              1SU/XVOA             ETCL/
              XVOA2                ETCL/
              XVOA17               ETCL/
              SUBOTTL              ETCL/GCLL
              1GWP3/Q/FIELD        ETCL/
              1GWP2                ETCL/
              FIELD/PH             ETCL/
              FIELD/SCOND          ETCL/
              1TOC4                ETCL/
              1TOX4                ETCL/
              FIELD/DENTRY         ETCL/
              1CHLOR               ETCL/GCLL
              1GW/MET/P2           ETCL/GCLL
              1PHENOLS             ETCL/GCLL
              1SO4                 ETCL/GCLL
```

APPENDIX 8

E LO(8H

Pa[illegible] 1 [illegible]

(4.5.11)

Company Name
(4.5.6)

LogLink
(4.5.2)

Facility
(4.5.2)

T/A **Date received**
(4.5.2)

Shipper **# of shuttles**
(4.5.3)

Airbill #
(4.5.3)

Revision

10/9, - REVISED A-TYPE, AND COMMENT FOR HA3062, 64

Order Entry Comments

HA3062-SPECVOA: 7 DAY HOLDING TIME FOR HPT/VOLATILES.
HA3062-SPECVOA/ACID/BN/GC/AA/CV: TIER I DELIVERABLES REQ'D.
HA3062-SPECSUB: RLEASE PROVIDE ALL RAW DATA AND QC INFORMATION
HA3062-WITH DATA PACKAGE.
HA3064-SPECVOA: 7 DAY HOLDING TIME FOR HPT/VOLATILES.
HA3064-SPECVOA/ACID/BN/GC/AA/CV: TIER I DELIVERABLES REQ'D.
HA3064-SPECSUB: PLEASE PROVIDE ALL RAW DATA AND QC INFORMATION
HA3064-WITH DATA PACKAGE.
HA3062, 3064, - SPECCV.: PLEASE ALIQUOT FROM EXTRACTABLE BOTTLE FOR PH/SCOND.

(4.5.4)

Sample Mgmt Comments

(4.5.9)
(4.5.10)
(4.5.11)

Cr^{+6} transferred to sub-lab from field.

Problem note #: Yes

Shuttle #	Temp	Sample Id	Sample Date	Job#	(4.5.5)				Sample Type	← (4.5.8)		→
999 103	7 6	BF99	09/26/90	HA3062	1AP9/HPTVOA 1AP9/PEST(FPD) 1AP9/MET 1AP9/ACID 1AP9/CY 1AP9/PTVOA 1CR(+6) SPECVOA SPECBN SPECAA SPECSUB (4.5.15)	APD5/ APD5/ APD5/ APD5/ APD5/ APD5/ APD5/CHYN APD5/ APD5/ APD5/ APD5/CHYN	1AP9/PEST(EC) 1AP9/PCB(EC) 1AP9/SULFIDE 1AP9/BN 1AP9/HCP 1AP9/HERB(EC) SPECVOA SPECACID SPECGC SPECCV 1 PH 1 SCOND SPECCV	APD5/ APD5/ APD5/CHYN APD5/ APD5/ APD5/ APD5/ APD5/ APD5/ APD5/ *	H20	~~CR+6(1) 500~~ NA HPT/VOLATI(2) 40 SULFIDES(2) 125 VOA SCREEN(1) 40	CY/T(2) 500 METALS(1) 1000 TRIP BLANK(1) 40	Extractabl(8) 1000 PT/VOLATIL(2) 40 UNPRES TB(1) 40
773 348	9 8	DUPLICATE	09/26/90	HA3064	See Job HA3062 *				H20	~~CR+6(1) 500~~ NA HPT/VOLATI(2) 40 SULFIDES(2) 125 VOA SCREEN(1) 40	CY/T(2) 500 METALS(1) 1000 TRIP BLANK(1) 40	Extractabl(8) 1000 PT/VOLATIL(2) 40 UNPRES TB(1) 40

APPENDIX 9

100G-6

PROBLEM LOG

TODAY'S DATE 9/27/90

COMPANY NAME

FACILITY CODE

PS REP

DATE SUBMITTED 9/27

PROBLEM DESCRIPTION	CHECK	JOB NUMBER/A-TYPES
[✓ 1 SMPLE PT - UNAPPROVED/OMITTED]	✓	LINKED NOT UPDATED
2 SOURCE CODE - OMITTED		fac. code/sample pts. approved.
3 START DATE - OMITTED		Need Chain CoC
4 SMPL BOTLS REC'D EMPTY A-TYPES		[illegible] 9/27
5 INCOMPLETE SPECS		
6 MISMATCHING JOB#'S (CC1VSC2)		
7 METALS COMMENTS		
8 MISMATCHING SMPLE ID'S (CC1VSCC2)		
9 MISMATCHING FAC-CODES (CC1VSORDER)		
10 REC'D EMPTY (ALL) DRYWELL/OTHER		
11 FIELD DATA-OMITTED/INCOMPLETE		
12 LOCATION CODES		
13 OTHER		

MISSING:

A FIELD/PH	F FIELD/ELEV
B FIELD/SCOND	G FIELD/WELD
C FIELD/TEMP	H FIELD/SAMPD
D FIELD/DTGW	I FIELD/MISC.
E FIELD/GWE	J OTHER

REVIEWED BY: DATE: 9/27/90

RESOLVED BY: DATE: 10/4/90

JACKET COMPLETED BY: DATE:

APPENDIX 10

OHM Corporation
Environmental Testing and Certification Corp.

Request for Subcontract Analysis and Sample Chain-of-Custody

Name of Subcontract Laboratory:

Loglink(s): 100646 **Workgroup:** APD5

Matrix: H2O **Sampling date(s):** 9/26/90

ETC Sample ID Numbers:
HA3062 (2)
HA3064 (2)

Turnaround is 14 days unless otherwise indicated.
(If deadline can not be met, contact **ETC Subcontract Department** - (201)225-6700
Send report and invoice to ETC Corp. P.O.Box 7808, Edison, NJ 08818-7808
Attention: Subcontract Department

The following analyses are requested:

ACIDITY
ALKA
NH3
NH3/D
ASBESTOS/EM
ASBESTOS/LM
BICARB
BOD
BROM
BTU
CARB
CA/CACO3
CHLOR
CL2/T
CL2/D
CL2/R
COD
COLI/F
COLI/T
COLOR/S
COLOR/A
CR(+6)
CY(CHLOR)

CY/T
FLUORIDE
FORM/UV
HARD
NO3
NO2
N2/ORG
N2/TK
ODOR
O+G/GRAV
O+G/IR
PETHY/GRAV
PETHY/IR
PHENOLS
PHENOLS/DL
PO4/T
PO4/ORG
PO4/ORT
PHOS
CY/REACT
SULFIDE/REACT
SOLIDS/T
SOLIDS/TD

SOLIDS/SETL
SOLIDS/TS
SOLIDS/TV
SO4
✓ SULFIDE
SO3
SURFAC
TOX
TOX2
TOX4
TOC2
TOC4
TURB
DW1/ABO
DW1/RA226
DW2/RA228
AP9/SULFIDE
AP9/CY
COMPCV
FILT
HOMO
ECRA/PETHYIR

Others: spec sub please provide all Raw DATA + QC information with data package

Chain-of-custody (complete appropriate section)

Option A: Courier pickup at ETC
Sample(s) **relinquished** by ETC:
Time: 300 **Date:** 9/27/90
Sample(s) **received by:**
Time: 1430 **Date:** 9/27/90

Option B: Sending sample from ETC
Shuttle sealed at ETC by:
Date: Time: **Seal Number:**
Shuttle opened by:
Date: Time: **Seal Number:**
Seal intact? yes / no Shuttle contents in good condition? yes / no

Comments:

Sub form 0067.01

(4.5.15)

APPENDIX 11

Figure E8 - 1

EPA Contract Laboratory Program
PO Box 818 Alexandria, VA 22313
703 557-2490 FTS 557-2490

& ... :sto ... rec
(For Organic CLP Analysis)

Get code from RSCC →

1. Project Code	Account Code	2. Region No: V	Sampling Co.: Company Name
Regional Information: TFA102		Sampler (Name): Sampler Name	
Non-Superfund Program		Sampler Signature: Sampler Signature	
Site Name: Site Name		3. Type of Activity	
City, State: City, State	Site Spill ID: Code		

3. Type of Activity: Lead: SF ☒ PRP ☐ ST ☐ FED ☐ — Remedial: Pre-Remedial PA ☐ SSI ☐ LSI ☐; RIFS ☒ RD ☐ RA ☐ O&M ☐ NPLD ☐ — Removal: CLEM ☐ REMA ☐ REM ☐ OIL ☐ UST ☐

4. Date Shipped: Date
Carrier: Carrier Name
Airbill Number: Airbill Number
5. Ship To:
Laboratory Name
Address
Attn: Name

6. Preservative (Enter in Column D)
1. HCl
2. HNO3
3. NaHSO4
4. H2SO4
5. Other (SAS) (Specify)
6. Ice only
N. Not preserved

7. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type: Comp./Grab	D Preservative from Box 6	E RAS Analysis VOA	BNA	Pest/PCB	High ARO/TOX	F Regional Specific Tracking Number or (Tag Numbers)	G Station Location Number	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Inorg. Samp. No.	K Enter Appropriate Qualifier for Designated Field QC (B = Blank S = Spike D = Duplicate PE = Perform. Eval — = Not a QC Sample)
EPA01	2	L	G	1	X				5-12357→12362	MW01	Date/Military Time		MEPA01	
EPA01	↓	↓	↓			X	X		5-12363→12366	MW01	Date/Military Time			
EPA02				1	X				5-12367→12368	MW02	Date/Military Time		MEPA02	
EPA02						X	X		5-12369→12370	MW02	Date/Military Time			
EPA03				1	X				5-12371→12372	MW03	Date/Military Time		MEPA03	D-EPA04
EPA03						X	X		5-12373→12374	MW03	Date/Military Time			
EPA04				1	X				5-12375→12376	MW03	Date/Military Time		MEPA04	
EPA04	↓					X	X		5-12377→12378	MW03	Date/Military Time			
EPA05	3			1	X				5-12379→12380	FB01	Date/Military Time		MEPA05	B
EPA05	3	↓	↓			X	X		5-12381→12382	FB01	Date/Military Time			

Field ONLY in column

Do not MS/MSD sample

Shipment for Case complete? (Y/N) Circle One	Page 1 of 3	Sample used for a spike and/or duplicate: EPA01	Additional Sampler Signatures	Chain of Custody Seal Number: COC Seal #'s

Lab QC. Sample must be designated here at a rate of 1/20 samples per matrix (org + inorg, waters + soils) - extra volume required for waters.

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature) Signature	Date / Time: Date Military Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)
☐ Declined

EPA Form 9110-2 (Rev. 6-91) Replaces EPA Form (2075-7), previous edition which may be used
DISTRIBUTION:
Blue - Region Copy Pink - SMO Copy White - Lab Copy Yellow - Lab Copy for Return to SMO

APPENDIX 12

ETC

USEPA SAMPLE LOG-IN

Case #: ______________________________

Received Date: ______________________________

Sample Manager/Date: ____________________/____________________

DIRECTIONS: Circle the appropriate answer and complete all blanks.

1.	EPA Chain of Custody Form is present for all samples.	YES	NO
2.	Airbills present for all containers received.	YES	NO
3.	Traffic reports/SAS packing list present.	YES	NO
4.	Custody seals present on shipping containers.	YES	NO
5.	Custody seals present on sample containers.	YES	NO
6.	Sample tags present for all samples.	YES	NO

7. List any sample ID tags not included on chain of custody records:

__________ __________ __________ __________

__________ __________ __________ __________

8.	Condition of shipping containers:	INTACT	NOT INTACT
9.	Condition of sample containers:	INTACT	NOT INTACT
10.	Receiving documents are in agreement.	YES	NO

11. Problem numbers discussed with Sample Management Officer:

____

____

____

____

SMO Contacted By: ______________________________

Name of SMO Contact: ______________________________

Date: ______________________________

Rev. 10/90

ETC Corporation Network Document

TITLE Internal Sample Tracking	Doc.#ED222000 Revision # 0 Effective Date 11/1/90
Approval: Lab Manager QA Manager 11/2/90	

1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP is to describe the mechanisms for internal sample tracking at ETC/EDISON from receipt to final analysis and to include examples of laboratory documents.

2.0 SUMMARY

2.1 Sample tracking begins at time of receipt, when a hardcopy login form is generated and the sample is linked into ETC's computer system for scheduling and tracking. Sample are placed in a secured storage area by the sample manager as part of the log-in process. The samples are accessed by authorized laboratory personnel, utilizing laboratory chronicles to document changes in custody.

2.2 Designated schedulers utilize log-in Forms and computer-generated reports to prepare analytical batches, which are processed by appropriate laboratory personnel. As work progresses on each sample, changes in status are routed through schedulers, who update the computer system. Information from the computer system is available in both hardcopy and screen versions, and is used by ETC personnel to track the status of samples in the laboratory.

2.3 When all analytical work has been completed on a sample, the data batch is routed to the scheduler or other designated person within each work area and the computer system is updated.

2.4 Any remaining sample and/or extract is held in a secured storage area until at least 30 days after the issuance of the technical report. At the end of 30 days, the sample is disposed of. ETC retains samples and/or extracts samples for longer periods of time in those cases where it is requested by the client or is contractually required.

3.0 Safety

3.1 All appropriate safety precautions as described in the ETC Safety Manual should be used when handling samples.

4.0 APPARATUS AND MATERIALS

4.1 Computer terminal with access to

4.1.1 AMSTAT

4.1.2 CSO5AM

4.2 Laboratory Chronicles

5.0 PROCEDURES

5.1 Samples are received at ETC in the sample management area. The sample manager logs the sample in utilizing the procedures described in SOP# ED21003 ETC/EDISON Log-in Procedure. At the time of login, a hardcopy log-in form is generated and circulated to appropriate personnel, including group schedulers, and the samples are linked into ETC's computer system. The samples are placed in a secured storage area.

5.2 The schedulers utilize the log-in forms and other computer generated reports to prepare and schedule analytical batches. The batch sheets are routed to appropriate personnel for processing. The samples on the batch sheets are retrieved by analysts and/or technicians from the secured storage areas. Changes in custody are documented by the use of laboratory chronicles, as described below:

5.2.1 Sample Preparation Chronicle

The analyst who signs under "set-up" claims custody of the sample from sample storage to the laboratory. The verification of the supervisor's signature represents the completion of analysis and subsequent transfer of the remaining sample back to sample storage, and of the extract to the extract storage refrigerator.

5.2.2 GC/MS Laboratory Chronicle

- Volatiles - The analyst who signs the analysis chronicle claims custody of the sample from sample storage to the laboratory. The supervisor's signature represents the completion of analysis and transfer of the sample from the laboratory back to the volatile refrigerator.
- Extractables - The analyst who signs the analysis chronicle claims custody of the extract from the extract refrigerator to the laboratory. The supervisor's signature represents the completion of analysis and the transfer of the remaining extract back to extract storage.

5.2.3 GC and HPLC Analysis Chronicle

The signature of the analyst represents custody of the extract in the laboratory. The supervisor's signature represents the completion of analysis and transfer of remaining extract back to extract storage.

5.2.4 Metals Laboratory Chronicle

The chemist who signs for preparation is responsible for the transfer of the sample from initial storage to the laboratory. The supervisor's signature represents the completion of sample analysis and transfer of remaining sample back to initial sample storage.

5.2.5 Conventionals Laboratory Chronicle

The analyst who signs on the analysis log form claims custody of the sample from initial storage to the laboratory. The signature of the supervisor represents completion of analysis and transfer of remaining sample back to storage.

5.2.6 Percent Moisture Worksheet

The Form may be used independent of the Sample Preparation Chronicle . The analyst who signs on the form claims custody of the sample from initial storage to the laboratory. The signature of the supervisor represents completion of analysis and transfer of remaining sample back to storage.

5.3 As the sample progresses through the laboratory, changes in status are routed through group schedulers, who update the computer system as needed.

5.3.1 When sample preparation re-work is required for semi-volatile organic analysis, a "Rework Required Form for Sample Preparation" is initiated and the sample is rescheduled for the re-extraction or other rework specified.

5.4 When all analytical work has been completed on a sample, the data batch is submitted to the group scheduler or designate. This person updates the computer system, and transfers the batch on to the Report Production area for processing into technical reports. Issued technical reports are stored in a secured area on-site for a period of up to 1 year then transferred to an off-site archive.

5.4.1 USEPA CLP Samples technical reports are purged with the case file as contractually required.

5.5 Any remaining samples and/or extracts are stored in secured areas for at least 30 days after the issuance of technical reports, or for as long as requested by the client or contractually required. After this period, the sample is disposed of as described in SOP# SMS00501.ED.

6.0 QUALITY CONTROL

7.0 APPENDICES

7.1 References

7.2 Supplemental Document

7.3 Quality Control Criteria

APPENDIX

7.0 APPENDICES

7.1 References

7.1.1 ED21003 - SOP for ETC/EDISON Log-In Procedure

7.1.2 SMS00501.ED - SOP for Sample Disposal at EDISON.

7.2 Supplemental Documents

7.2.1 ETC login form

7.2.2 Copy of page from CS05 showing various status' of samples

7.2.3 Copies of laboratory chronicles

7.2.4 Copy of status change request form from the laboratory

7.2.5 Copy of Sample Preparation Rework Form

7.2.6 Copy of computer generated GC/MS schedule

7.3 Quality Control Limits

7.3.1 Not Applicable

SUPPLEMENTAL DOCUMENTS

ETC LOGIN FORM

ETC LOGIN SHEET

Page 1 of 0

Company Name

Loglink

Facility

T/A Date received

Shipper # of shuttles

Airbill #

Revision

Order Entry Comments

Sample Mgmt Comments

Problem note #: ______________

Shuttle #	Temp	Sample Id	Sample Date	Job#		Sample Type				

COPY OF PAGE FROM CS05 SHOWING VARIOUS STATUS' OF SAMPLES

```
ALL atypes, ALL costcenter(s)
Sorted by: DUEDATE, ACCOUNT, JOBNUMBER      Lab Sample Control Report (CSO5A )  Data last updated 90/11/10 at 05:41

 Job No.  LogLnk  Xrnd  DueDate    Hold      Src/Sent   Atype         QaBatch
 <Bkjob>  JobLnk  Team  RecDate    Date      Location                              SP    MS    GC    AA    CV    PR    DM
 Facility SmpPnt        Price
-------------------------------------------------------------------------------------------------------------------------------
Acctid: [redacted]
-------------------------------------------------------------------------------------------------------------------------------
 CAS030-02 100704 CA14   90/11/09   00/00/00  CLP1/      22FB/PST/PCB                ****  ****                    ****
             35507 WS/LA (90/10/25) 90/11/04  CLP1/      22FB/PST       QG70315      D1031       H1031             NEED
 [redacted]  SP-23(1-2)  [redacted] 00/00/00  CLP1/      1FB/PH/M       QG70315      D1031
                                    90/11/04  CLP1/      22FB/PCB       QG70315      D1031       H1031             NEED
                                    00/00/00  CLP1/      SPECGC         QG70315                  H1031             NEED
                                    00/00/00  CLP1/      SPECRP                                                    NEED

/SPECGC/RP: REPORT ONLY PCB'S./ /** 14-DAY VERBAL, 21-DAY HARDCOPY./**/[redacted]
-------------------------------------------------------------------------------------------------------------------------------
 CAS038-03 100704 CA14   90/11/09   00/00/00  CLP1/      11FB/PST/PCB                ****        ****              ****
             35507 WS/LA (90/10/25) 90/10/29  CLP1/      11FB/PCB       QG70316      D1031       H1031             NEED
 [redacted]  FIELDBLANK  [redacted] 90/10/29  CLP1/      11FB/PST       QG70316      D1031       H1031             NEED
                                    00/00/00  CLP1/      SPECGC         QG70316                  H1031             NEED
                                    00/00/00  CLP1/      SPECRP                                                    NEED

/SPECGC/RP: REPORT ONLY PCB'S./ /** 14-DAY VERBAL, 21-DAY HARDCOPY./**/[redacted]
-------------------------------------------------------------------------------------------------------------------------------
 CAS037-03 100704 CA14   90/11/09   00/00/00  CLP1/      11FB/PST/PCB                ****        ****              ****
             35507 WS/LA (90/10/25) 90/10/30  CLP1/      11FB/PCB       QG70316      D1031       H1031             NEED
 [redacted]  FIELDBLANK  [redacted] 90/10/30  CLP1/      11FB/PST       QG70316      D1031       H1031             NEED
                                    00/00/00  CLP1/      SPECGC         QG70316                  H1031             NEED
                                    00/00/00  CLP1/      SPECRP                                                    NEED

/SPECGC/RP: REPORT ONLY PCB'S./ /** 14-DAY VERBAL, 21-DAY HARDCOPY./**/[redacted]
------------ CUMRESOLVE Facility Total:  3          [redacted] ---------------------------------------------------------------
-------------------------------------------------------------------------------------------------------------------------------
                   Account Daily Total:  3          [redacted]
===============================================================================================================================
Daily Totals for 90/11/09       Jobcount:  3        [redacted]                            0     0     9     0     0    12     0
===============================================================================================================================
```

COPIES OF LABORATORY CHRONICLES

ETC CORP. LAB CHRONICLE Sample Preparation

QC BATCH NO:

JOB NO.	LOGLINK	[illegible]	SAMPLE AMT ml gm	MOIST %	DRY WGHT gm	EXTRACT VOL (ml)			[illegible]	REPEAT		ATYPES
										ORIGINAL JOB NO	ORIGINAL QS. NO.	
QS	VERIFY SPIKE											
S1												
S2												
MS1												
MSD1												
MS2												
MSD2												

COMMENTS:

PROCEDURE	SIGNATURE	DATE
☐ CONTINUOUS		
☐ SEP. FUNNEL		
☐ SOXHLET		
☐ SONIFICATION		
☐ DILUTION		
☐ DERIVATION		
☐ CLEANUP		
☐ SETUP		
☐ CONCENTRATION		
☐ SUPERVISOR		

SPIKE	VOL ml	CONC ug/ml	LOT NUMBER
SURROGATES	VOL ml	CONC ug/ml	LOT NUMBER

MATRIX
- ☐ 1 WATER
- ☐ 2 SOIL
- ☐ 3 COMPLEX
- ☐ 4 ORGANIC LIQ.

UPDATES
- ☐ OS
- ☐ WIP
- ☐ DONE

EHD: ____________

EIC CORP.

$$\% = \frac{\text{WET SOIL WEIGHT} - \text{DRY SOIL WEIGHT}}{\text{WET SOIL WEIGHT}} \times 100$$

$$\text{DRY WEIGHT} = 1 - \frac{\%M}{100} \times \text{WET SOIL WEIGHT}$$

$$\%\text{SOLIDS} = (1 - \%M) \times 100$$

PERCENT MOISTURE WORKSHEET

QC BATCH: ____________________

SAMPLE NUMBER	LOG LINK	CRUCIBLE WEIGHT (g)	CRUCIBLE & WET SOIL WEIGHT (g)	WET SOIL WEIGHT (g)	CRUCIBLE & DRY SOIL WEIGHT (g)	DRY SOIL WEIGHT (g)	PERCENT MOISTURE (%)	PERCENT SOLIDS (%)
-D								

COMMENTS: ____________________

PERCENT MOISTURE SET-UP: ____________________ DATE: ____________

PERCENT MOISTURE COMPLETION: ____________________ DATE: ____________

SUPERVISOR SIGNATURE: ____________________ DATE: ____________

ETC Corp. LABORATORY CHRONICLE : GC-MS Department

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Page___of___

Date__________ Instr.__________

A-Type__________

Tune file__________

Seq. file__________ , __________

Method file__________

ID file__________

CB file__________

Analyst__________

Reviewed by/Date__________

Batch #'s __________

__ Standards Updated__________

Date__________By__________

Tape # __________Inj.___ul

Standard	Conc ppm	Lot No.	Lot Vol.

R	NAME	DATA File	init. ml/g	ALS #	Dil	Inj. Time	Comments

R : redo , P : Aroclor search , + : Plus search

ETC Corp. LABORATORY CHRONICLE : GC Department 57 Page___of___

Date__________Instr.______

A-Type__________
Column__________
Col. Lot__________
Calib.File__________
Seq. File______ ,______
Method file__________
ID file__________
Configu. file__________
Analyst__________
Reviewed by/Date__________
Batch #'s

______Standard Updated______
Date________By________

Tape #________ Inj.____ul

Standard	Conc. ppm	Lot No.	Standard	Conc. ppm	Lot No.

R	NAME	Data File Detec.1	Data File Detec.2	init. ml/g	ALS #	Dil	Comments

R : redo

ETC Corp. LABORATORY CHRONICLE : LC Department 3 Page___of___

Date__________ Instr.________

a-Type__________

Seq. file________ ,______

Method file________

ID file________

DB file________

Analyst________

Reviewed by/Date________

Batch #'s________

__ Standards Updated________

Date________By________

Tape #________Inj.___ul

Standard	Conc ppm	Lot No.

R	NAME	DATA File	init. ml/g	ALS #	Dil	Comments

ETC

ETC LOGLINK ______________

ETC CORP.

METALS DEPARTMENT

LABORATORY CHRONICLE

SAMPLES

____

____

____

____

	CHEMIST	DATE
Hg Prep		
AA Prep		
ICAP Prep		
Boron Prep		

SUPERVISOR SIGNATURE ______________________ DATE ______________

ETC

TOTAL PHENOLICS

QW-Batch: ____________

QC-Batch: ____________

Verified: ____________

Instrument: ____________

Method Ref: EPA 420.2, SW-846 9066

Colorimetric, Auto-4AAP, w/Distillation

MDL: 0.050 mg/l

Matrix: Aqueous

Page ______ of ____

Date: ____________

Time: ____________

Analyst: ____________

Seq #	ETC Job #	pH Int.	Analyzed mg/l	Dilution Factor	Reported mg/l	Comments Calculations

CV-10 5/90 Rev. 0

ETC CORPORATION

CLP1 UPDATE FORM

BATCH #: ____________________ DATE: ____________

SAMPLE #'S OR ATTACHED CHRONICLE: ____________________

COST CENTER:
SP ☐ MS ☐ GC ☐ AA ☐ CV ☐ PR ☐

STATUS: HERE ☐ ACQ ☐ WIP ☐ DONE ☐ BATCH ☐

ATYPE:

IFB/VOA, IFB/VO+1 ☐
IFB/BNA, IFB/BNA+2 OR 20 ☐
IFB/PST ☐
IFB/PCB ☐
IFB/MET ☐
IFB/CVAP ☐
IFB/FLAME ☐
IFB/FURNACE ☐
IFB/ICAP ☐
IFB/CN ☐
PP/VOA ☐
PP/BN ☐
PP/ACID ☐
PP/PST ☐
PP/PCB ☐
O&PST/PCB ☐
PP/MET ☐
RC/MET ☐
EPTX/MET ☐
TCLP/MET ☐
TAL/MET ☐
M601 ☐
M602 ☐
M618 ☐
SPEC___ ☐

ECRA/VOA, ECRA/VO+15 ☐
ECRA/BNA, ECRA/BNA+25 ☐
ECRA/PST(GC) ☐
ECRA/PCB(GC) ☐
ECRA/MET ☐
ECRA/CN ☐
ECRA/PN ☐
NJTSK/VOA, NJTSK/VO+15 ☐
NJTSK/BN, NJTSK/BN+15 ☐
NJTSK/ACID, NJTSK/AC+1 ☐
NJTSK/PST(GC) ☐
NJTSK/PCB(GC) ☐
NJTSK/MET ☐
NJTSK/CN OR NJTSK/CY ☐
NJTSK/PN ☐
AP__/PTVOA ☐
AP__/HPTVOA ☐
AP__/ACID ☐
AP__/BN ☐
AP__/HCP ☐
AP__/PST(EC) ☐
AP__/PCB(EC) ☐
AP__/PST(FPD) ☐
AP__/HERB ☐
AP__/MET ☐

OTHER: ____________________

SUBMITTED BY: ____________________

UPDATED BY: ____________________ DATE: ____________

ETC

CLP 1 WORK GROUP

REWORK REQUIRED FORM FOR SAMPLE PREPARATION

Q__ BATCH NO.__________

SAMPLE NO.	FRACTION	REASON (Refer to Key)

KEY:

(1) LOW/NO PEST SURR
(2) LOW/NO BN SURR
(3) LOW/NO ACID SURR
(4) HIGH PEST SURR
(5) HIGH BN SURR
(6) HIGH ACID SURR
(7) MS,MSD DO NOT MATCH
(8) BIPHASIC
(9) NEEDS DILUTION
  [] (1:5), [] (1:10)
  [] (   )(WRITE IN)
(10) CONTAINS PRECIPITATE

(11) EVAPORATED
(12) BLANK CONTAM.
  WITH_________
(13) NEEDS TBA
  SULFITE CLEAN-UP
(14) NEEDS COPPER
  CLEAN-UP
(15) LOW/NO SPIKE
(16) HIGH SPIKE
OTHER____________
____

REQUESTOR:______________________ DATE:______________
AUTHORIZED BY:_________________(MANAGER) DATE:______________
QA OFFICER:_____________________ DATE:______________

Current date: 90/09/24 *** MS SCHEDULING SHEET *** Page: 1

Sequence:

10/1

Batch#: MS5310
Sched: 90/09/24
Category: VOA

Job#	Scrn Amt	Fraction	LogInk	Hold	Xrnd	Batch#	Acq	Wip	Comments
CA4910		1NJTSK4/VO+15	100629	09/30	DA14	QV7035	✓	✓	SPECVOA: VERBAL DATA DUE 14 DAYS VTSR. HARDCOPY DUE 21 DAY VTSR. NO INDEP INTERNAL CHAIN OF CUSTODIES
		1NJTSK4/VOA	100629	09/30	DA14	↓	✓	✓	
		SPECVOA	100629	00/00	DA14		✓	✓	

------------------------------ END OF BATCH ------------------------------

Start date: ______ Signature: 9/25

Completion date: ______ Signature: 9/20

APPENDIX D-2

COMPUCHEM

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories (if for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples).

- Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat, safety glasses and gloves). These items need to be worn at all times when in the marked areas (colored line).
- Each sample container is inspected before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, these items are inspected to make sure that they are intact and this observation is recorded on the chain-of-custody form (Attachment 1). If the custody seals, tapes, or padlocks are broken, one must contact Customer Service (for commercial samples) or the Sample Management Office (for EPA samples) for permission to continue processing the sample.
- Each container is opened under the hood and checked for breakage. The condition of the refrigerant is checked (whether any ice remains or whether the cooling packs are solid) and the temperature of a representative sample (liquid samples only) is obtained by immersing a clean thermometer in the sample. The temperature is recorded on the Sample Record (Attachment 2).
- The temperature and pH are recorded on the Log-In Sheet. If a temperature or pH variance occurs, a QA Notice is written and associated with the sample (Attachments 3-4).
- Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.

- The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.

- When a CompuChem SampleSaver is received, the SampleSaver number is recorded on the Log-In Sheet and is entered into the LMS system by number.

- Samples are removed from the shipping container and the sample identification information on the sample bottles is compared to the sample information on the traffic sheets, packing lists, and Chain-of-Custody Form included in the container (Attachments 5-11).* For EPA samples, Form DC-1 is filled out as well, per USEPA SOW. If discrepancies exist, the problem is noted on the Chain-of-Custody Form; the Receiving Clerk notifies Customer Service (for commercial samples) and the Supervisor notifies SMO (for EPA samples).

- Each water VOA is checked for air bubbles and headspace, and noted on the Chain-of-Custody Form as well as the Log-In-Sheet.

- On each complete and correct <u>EPA Chain-of-Custody and Traffic Report</u> the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

- On each complete and correct <u>Commercial Chain-of-Custody</u> the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

* NOTE: For samples associated with NJDEP X-408, an NJDEP059 "Sample Analysis Request Form" will accompany the samples upon receipt (Attachment 12).

Received in Good Condition' is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. All sample tags (if received) regardless of condition are retained and stored in grey folders which are produced along with sample records, for CLP deliverables samples. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain-of-custody seals), the exceptions are noted on the appropriate documents, initialed and dated.

- The statement 'Received in Good Condition' does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.

- Incoming samples are checked against SMO scheduled receipts (for EPA samples).

- The sample is logged in on the Log-In Sheet Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

- For EPA samples, the samples' account data is entered into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, customer service is contacted to check for the existence of the order and to receive requisitions for analyses. The order is then completed in the CLMS, and the EPA Scheduling Log (Attachment 13) is also completed.).

- The sample is entered into the sample receipt portion of the CLMS in order to generate a CompuChem number for each sample. The CompuChem number is filled in on the accessioning log sheet (this completes the log sheet).

- The CompuChem number is a unique, six digit identity which is generated by the LMS system in numerical sequence. It can be cross-referenced to the Client ID.

- A CompuChem label is generated in numerical sequence, with the CompuChem number.

- The samples are labelled with the CompuChem number by wrapping each sample bottle with its computer-generated CompuChem sample label. Sample labels are color coded, and are to be rotated with a different color every 2 week period by the Supervisor of Receiving or the Supervisor designee.

- Each Log-In Sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review each log sheet is stamped as reviewed and initialed and dated.

- The labelled samples are transferred to the secured, locked walk-in cooler facility.

- The CompuChem number is listed on the original Chain-of-Custody Form next to the associated client ID when possible.

- The Quiz portion of the CLMS is accessed to produce the worksheets for EPA sample analyses. For EPA samples the system will generate volatile, semi-volatile, and pesticide worksheets. For commercial and inorganic samples, the appropriate worksheets are pulled from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with Customer Service. All laboratory worksheets are distributed to the appropriate Production Planning and Control Planner.

- To produce EPA quality control worksheets for the QC samples associated with a batch of samples, EPA Water or EPA Solid programs of Quiz in the CLMS is accessed and the samples' CompuChem Numbers are entered; these worksheets are copied on green paper. To assemble commercial QC worksheets, the appropriate green fraction worksheets are pulled from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary, a copy of the Chain-of-Custody Record.

- Commercial file folders are assembled for Report Integration; included in the production sample's folder are the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; the QC Sample Record is included in the green Quality Control folder which also goes to Report Integration.

- EPA file folders are assembled for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-of-Custody, original air-bill, a copy of the Log-In Sheet, a copy of the EPA Scheduling Log,

  Custody Tags (if received) and a gray envelope contents sheet (Attachment 9). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (Attachment 10).

- If problems arise concerning received samples, Customer Service is contacted (for commercial samples) or the Technical Management Staff is contacted (for EPA samples see below).

- Samples are sometimes received from the EPA that should not have been sent. Therefore, they require a transfer. The following steps should be taken:

  1. Fill out a new Chain-of-Custody Form using the information on the sample tags.

2. Sign the Chain-of-Custody in the first section labeled "Relinquished By".

3. A Copy of Chain-of-Custody is kept for our records; the original is sent with samples. A copy of the paperwork received with the samples should also be sent.

4. Notations are made on the Traffic Reports stating samples are being sent to another laboratory.

Samples hand-delivered after business hours should be recorded as follows:

1. The actual date of sample receipt shall be recorded on the Chain-of-Custody (see Note).

2. The date and time of sample receipt are recorded on the TRs as follows:

   a. Organic Traffic Report

   Date and time of receipt in Column F, sample condition upon receipt.

   b. Inorganic Traffic Report

   Same as above

The notation "H.D." (Hand-Delivered) or "Received by Common Carrier" should be recorded next to the actual sample receipt time and date. The following calendar day shall be recorded in the block designated for Receipt Date on the Organic/Inorganic Traffic Report.

Samples received after business hours are logged in like samples received during regular business hours (page 1 of this SOP details the procedure for logging in samples).

Note: For New Jersey DEP Chain-of-Custody procedure, see Production Planning and Control SOP 3.2 and the note on page 2 of 19 of this SOP.

COMPUCHEM LABORATORIES

CHAIN-OF-CUSTODY RECORD

№ u00933

PROJECT NAME:

PROJECT NUMBER:

SAMPLERS (SIGNATURE)

PRINTED NAME

CLIENT ID (9 CHARACTERS)									#	GC/MS					GC							INORGANICS				OTHER											SAMPLING INFO		REMARKS
1	2	3	4	5	6	7	8	9	No. of Bottles/Vials	624-8240	625-8270	TCL-VOA	TCL-SVOA	Other.	601-8010	602-8020	608-8080	8140	TCL PEST/PCB's	Herbicides	Other.	Metals	Cyanide	TAL Metals	Other.	TOC	TOX	Oil & Grease	Pet. Hydro.	Phenols						MATRIX: Water/Soil	DATE	TIME	

RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time	SHIPPING INFO
COMPANY NAME:		COMPANY NAME:		COMPANY NAME:		Number of Shipping Containers-
RECEIVED BY:	Date/Time	RECEIVED BY:	Date/Time	RECEIVED BY:	Date/Time	Method of Shipment
COMPANY NAME:		COMPANY NAME:		COMPANY NAME:		
RELINQUISHED BY:	Date/Time	RELINQUISHED BY	Date/Time	RELINQUISHED BY.	Date/Time	Special Handling Requirements
COMPANY NAME		COMPANY NAME		COMPANY NAME		

ATTACHMENT 2

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:
Turnaround: Priority: Due:
Analysis Codes:

Lab Instructions

- -

Receiving Data

Sample Identification: SS Code:
SS Number: Receiving Codes:
Date Received: Time:
Receiving Information:
Containers Received:
Matrix: Temp: Sampling Date(s):

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date	
_______	____	( )
_______	____	( )
_______	____	( )
_______	____	( )

Company Name:

Sample Record

Requisition Number:	CompuChem Number:
Case:	Account Number:

- -

FOOTNOTES:

Applicable QA Notices:

- -

Company Name:

QUALITY ASSURANCE NOTICE

CompuChem#
Sample ID
Case#
Type of Analysis
Receipt Date

The pH reading for the sample above was ____, the required pH level is ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Preserve In-House
Analyze - Qualify with Notice
Dispose - Client will Resample
Subcontract Lab to Preserve

Supervisor Signature
Date

QANR2
900130

QUALITY ASSURANCE NOTICE

CompuChem# __________
Sample ID __________
Case# __________
Type of Analysis __________
Receipt Date __________

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C (+/-2C). The temperature on the sample above was ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Analyze - Qualify with Notice __________
Dispose - Client will Resample __________

Supervisor Signature __________
Date __________

QANR3
900130

COMPUCHEM LABORATORIES

RECEIVED ON:
CASE # ORG/INORG:
DELIVERED BY:
ACCOUNT #: CONTRACT #

ORDER#
FREIGHT #'s
REGION: $ AMT:

OF PAGES OF
CASE I'D
TAT C-OF-C Y or N
TAGS Y or N

#	REMARKS	REQ	SAMPLE ID	CC#	SD	TEMP	Sul	Chl	pH	ANAL CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY: RECEIVED BY: REVIEWED:

COMPUCHEM LABORATORIES

RECEIVED ON:
CLIENT:
DELIVERED BY:
SUBCONTRACT#

ORDER#
FREIGHT #'s
SS#

OF PAGES OF
CASE I'D
TAT C-OF-C Y or N

#	REMARKS	REQ	SAMPLE ID	CC#	SD	TEMP	Sul	Chl	pH	ANAL. CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY:
RECEIVED BY:
REVIEWED:

COMPUCHEM LABORATORIES

date shipped
to consignee:_______________ date received
from consignee: _______________

number of sample: ______________________________

consignee name: ______________________________

address: ______________________________

____

____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date ______________________

Time ______________________

To:

Date ______________________

Time ______________________

Company ______________________

Address ______________________

City & State ______________________ Zip Code ______________

Sample Name/Number ______________________

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

COMPUCHEM

CONTENTS OF FOLDER

DATE: ____________________

EPA: () ORGANIC () INORG　　COMMERCIAL: ()

CASE #: ____________________　　CLIENT: ____________________

NO. OF TRAFFIC RPTS: ____________________　　ORDER#: ____________________

#	ITEM	Y	N	NA	COMMENTS
1	CHAIN-OF-CUSTODY	()	()	()	
2	AIRBILL	()	()	()	
3	DAILY LOG SHEET	()	()	()	
4	TRAFFIC REPORT	()	()	()	
5	TAGS	()	()	()	
6	SMO COVER LETTER	()	()	()	
7	SCHEDULING LOG	()	()	()	

PROBLEMS/COMMENTS:

SAMPLE RECEIVING:

DOCUMENTATION CONTROL:

COMPUCHEM LABORATORIES

SAMPLE DELIVERY GROUP (SD
TRAFFIC REPORT (TR) COVER S

Lab Name: ______________________ **Contract No.:** 68-01-________

Lab Code: __________ **Case No.:** __________ **SAS No.:** ________-___

Full Sample Analysis Price in Contract: $________

SDG No./First Sample in SDG: __________ **Sample Receipt Date:** __________
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: __________ **Sample Receipt Date:** __________
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1		11	
2		12	
3		13	
4		14	
5		15	
6		16	
7		17	
8		18	
9		19	
10		20	

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order
(i.e., the order listed on this form).

______________________ ______________________
Sample Custodian Date

ATTACHMENT 11

SAMPLE LOG-IN SHEET

Lab Name: ____________________ Page ___ of ___

Received By (Print Name): ____________________ Log-in Date: ____________

Received By (Signature): ____________________

Case Number: ____________

Sample Delivery Group No.: ____________

SAS Number: ____________

REMARKS:

1. Custody Seal(s) Present/Absent* Intact/Broken
2. Custody Seal Nos.: ________ ________
3. Chain-of-Custody Records Present/Absent*
4. Traffic Reports or Packing List Present/Absent*
5. Airbill Airbill/Sticker Present/Absent*
6. Airbill No.: ________ ________
7. Sample Tags Present/Absent*
   Sample Tag Numbers Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: ________
11. Time Received: ________

Sample Transfer

Fraction: ________ ________

Area #: ________ ________

By: ________ ________

On: ________ ________

	CORRESPONDING		
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Reviewed By: ____________________

Date: ____________________

Logbook No.: ____________________

Logbook Page No.: ____________________

FORM DC-1

3/90

ATTACHMENT 11

Form DEP-050
10/86

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTE

SAMPLE ANALYSIS REQUEST FORM

SAMPLE LOCATION

NAME OF FACILITY/ COMPANY/PERSON	CODE	MAILING ADDRESS
SAMPLE LOCATION		

SAMPLE COLLECTOR

SAMPLER(S) Name	DIVISION		
Name	CONTRACT #	Task #	Report Form:

SAMPLE IDENTIFICATION

NJDEP SAMPLE NO.	SAMPLING DATE	ABSCISSA (Longitude)
SAMPLE POINT ID	TIME (MIL) SAMPLING BEGAN	ORDINATE (Latitude)
SAMPLE SOURCE CODE	TIME (MIL) SAMPLING FINISHED	ELEVATION
REFERENCE POINT		

SAMPLING PROCESS

COLLECTION METHOD	Code	Other	SAMPLE TYPE	Code	Other
TYPE OF SAMPLER	Code	Other	SAMPLE MATRIX	Code	Other
DESCRIPTION					

FIELD MEASUREMENTS

SAMPLE TEMP. °C	PARAMETER	PARAMETER	PARAMETER
AIR TEMP. °C	VALUE	VALUE	VALUE
WEATHER			

CHAIN-OF-CUSTODY IMPLEMENTED ☐ YES ☐ NO

PRESERVATION CODE(S) ___ ___ ___ ___ ___

REQUESTED ANALYSIS(ES)

PARAMETERS (by Compound or Group of Compounds)	CONTAINER NUMBER	DETECTION LIMIT (digits/units)	EMERG. (Check One)	NON-EMERG. (Check One)

ANALYSIS(ES) AUTHORIZED BY	TITLE

ANALYZING LABORATORY

CONTRACT LAB NAME	PERSON ACCEPTING SAMPLE		TITLE
LAB ID #	LAB CONTROL #	DATE	TIME (MIL)

ATTACHMENT 13

COMPUCHEM LABORATORIES

EPA SCHEDULING LOG

WEEK ENDING SATURDAY:

CASE#: () NEW CASE () OLD CASE () COMPLETED CASE

REGION: TURNAROUND TIME:

ACCOUNT #: CONTRACT #: CONTRACT $

****

QUANITY RECEIVED # OF SAMPLES

WATERS	
SEDIMENTS/SOILS	
OTHER	

CONCENTRATION: () LOW () MEDIUM () HIGH

ORDER#:	
AIRBILL#:	

****

PROBLEMS/COMMENTS:

SMO CONTACTED AT (TIME) SPOKE TO:

RESOLUTION FROM SMO:

APPENDIX D-3

WARZYN

CHAIN-OF-CUSTODY, LOG-IN AND TRACKING PROCEDURES

Scope and Application: This procedure is applicable to the log-in of samples and addresses such areas as: sample custody, log-in, labeling and preservation of samples. This procedure begins as the samples enter the laboratory and follows the samples to their destruction.

Procedure:

1. Chain-of-Custody Procedure: Samples arriving in the laboratory are accompanied by some type of custody record. The most common type of record is that shown in Figure 1. Follow the instructions below for all levels of chain-of-custody.

   A. Superfund Level Chain-of-Custody:

      1. Refer to the "Chain-of-Custody - Superfund Level" SOP.

   B. Routine Chain-of-Custody:

      1. Samples arriving in the laboratory are accompanied by a chain-of-custody record (refer to Figure 1).
      2. Review the custody form with the samples received and see that the spaces marked #1 - #12 have been accurately completed by the sampler.
      3. Any discrepancies with the chain-of-custody record or the samples should be immediately brought to the attention of the customer service relations person so that the problems can be solved in an efficient manner.
      4. If samples were shipped, note the courier name in the remarks section (#15) of the chain-of-custody record.
      5. Record any other comments such as: temperature upon receipt, preservation (if done in lab), problems, etc, in the remarks section (#15).
      6. Fill in the lab numbers in the section marked #16 (refer to the log-in portion of this procedure for how to obtain lab numbers).
      7. Sign and date the chain-of-custody record (#13 - #14).
      8. Route the custody record to the data management office when log-in is complete.

NOTE: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator and properly disposed of.

2. Log-In Procedure: Samples are logged in as follows (Refer to Figure 2):

A. Sample Information:

Care should be taken to document the condition of samples upon receipt. Anomalies such as broken or leaking bottles, expired holding times, improper labeling or preservation, and air bubbles in VOA vials should be noted on the chain-of-custody. It should also be noted whether the samples arrived cold.

B. Numbering Samples:

Number the samples on the chain-of-custody form. Record lab numbers used in the black lab book. If no chain-of-custody form accompanied the samples, the sampler must be contacted and one must be filled out. Blank forms are available in the log-in area.

NOTE:

- Sample preparation is required if the samples need to be filtered and/or preserved in the lab.
- If metals are requested, metal digestion is required for all matrices except filtered groundwaters or private well.
- Sample compositing is required if the lab is instructed to composite a series of samples received into one sample for analysis.

C. Subcontracted Work Required:

Refer to the subcontracting SOP for further instructions on subcontracting samples.

D. Special Accounting Notes:

If there is a discount or a special proposal for the sample analyses to be charged, record that information on the chain-of-custody.

E. Labeling Bottles:

1. Record the lab number on the sample bottle label and caps using a water-proof marker.

2. Check that the appropriate preservation and preparation has been circled.

3. If metals and total hardness are both required, using a red water-proof marker, place a dot on the bottle cap of that bottle to be used for those analyses.

F. Preserving Samples:

1. Samples which have been preserved in the field must be checked via pH paper in the laboratory to insure they were preserved to the correct pH. Check preserved samples as follows:

   a. Acid-preserved (HNO_3 or H_2SO_4) samples must be preserved to a pH <2. Using a pH stick, check the pH. If the pH is not <2, note this on the chain-of-custody, then add additional acid until a pH is obtained. IT IS CRITICAL THAT THE CORRECT TYPE OF ACID BE ADDED.

   b. Base-preserved NaOH samples must be preserved to a pH > 12. Using a pH stick, check the pH. If the pH is not > 12, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 12 is obtained.

   c. Base-preserved NaOH with ZnAc samples must be preserved to a pH > 9. Using a pH stick, check the pH. If the pH is not > 9, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 9 is obtained.

2. Samples which have not been preserved in the field need to be preserved in the laboratory. Record any preservation done in the lab on the field sheets or custody records. Refer to Figure 3 for preservation requirements and preserve accordingly. It is important that samples arrive in appropriate containers (see Figure 3). If they do not, contact the inorganic/organic supervisor. They will determine which analyses, if any, can be run.

3. It is important that samples be received and maintained cold for most analyses. If a sample was received warm, make note of this on the field sheet or custody record.

G. Computer Log-In:

Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

1. Turn on the computer terminal.
2. Enter: C CHICO (return).
3. Enter: ACSORA (return).
4. Enter: Password (return).
5. At the VMS $ prompt, enter LABMENU (return).
6. Enter: LOGIN (return).
7. Enter: Password (return).
8. The Log-In screen will appear as shown in Figure 2. Using information on the chain of custody, proceed as follows:
    a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the chain of custody, press return.
    b. Sample date automatically defaults to yesterday's date. If this is correct, press return or override with correct date.
    c. Enter sample identification from station location on the chain of custody and press return.
    d. Enter proper matrix number and press return.
    e. Desc: This is used if sample identification is longer than the number of characters in the sample identification field. (Return to override or enter information and return.)
    f. Date received automatically defaults to today's date. If correct, press return or override with correct date.
    g. Days to process automatically defaults to 14 days. If this is correct (standard turnaround time) press return or if rush work, override with the number of days until due.
    h. Duedate: First date listed is report date, second date is the lab due date which can be changed if necessary.

i. Comments: Enter special tests or other information needed to analyze samples correctly and press return.

j. Manager: Filled in automatically; press return if correct or over ride.

k. Price/Cost: Return if correct or over ride.

l. % Markup/Discount: For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.

m. To enter parameters, press **Next Screen** (see Figure 2A).

n. Matrix automatically defaults to matrix entered above (return or override).

o. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.)

p. To enter address for the report, press **Next Screen** (see Figure 2B).

q. Type: Report (return).

r. If an address has already been entered and matches the chain-of-custody press **Commit** and return.

s. If no address appears, press return until cursor is in the City field. Type in the city and press return.

t. Type in state (press **Commit** and return). After city and state has been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.

9. To duplicate the entry for another sample on the same chain-of-custody:

   a. If next sample to be entered has all the same information as the previous sample entered, press **Duplicate Record** key (F7).

   b. Enter sample identification and press **Commit** key, return.

10. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):

    a. Press **Create Record** key (Insert Here) and enter sample identification.

b. Press **Previous Screen** (F12) if sample date needs to be changed.

c. Press **Next Screen**, return. Then press **Next Screen** again to enter new product codes for the analyses requested.

d. Press **Previous Screen** to enter new sample or to quit.

e. Press **Clear Form** (F17) to enter a new chain-of-custody.

f. Press **Exit/Cancel** (PF4) to exit from the log-in screen.

   1. Press **Exit/Cancel** to exit from the LIMS system.
   2. At the VMS $ prompt, enter LO (return) to log off the VAX computer.
   3. Enter N (return).

13. If in Step 8a. the computer says project number is invalid:

   a. Press **Exit/Cancel** (PF4).

   b. Enter "Y" (return).

   c. On the LIMS Main menu, use the up arrow key to position the cursor on "Seedpak Management". Press **Commit** key.

   d. Use the down arrow key to move to project entry. Press **Commit** key.

   e. Enter project number (return).

   f. Enter account number (9999) and return.

   g. Enter description for chain of custody (project name).

   h. Enter manager from chain-of-custody (i.e. Finner, K).

   i. Press **Exit/Cancel** (PF4), twice.

   j. At the LIMS main menu, select log-in and press **Commit**.

14. To print daily log-in reports:

   a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:

   b. Select "Seedpak Reporting" from the main menu, and press **Commit**.

c. Select FS02 and press Commit.

d. Enter today's date, press Commit and return.

e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.

f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.

15. Sample Storage:

a. Proper handling and storage is necessary in order to maintain the integrity of the samples. Refer to Figure 3 for storage requirements.

b. Refrigerated Storage:

1. Store routine custody samples in the walk-in refrigerator. Separate samples by preservative type. Samples with organic analyte requests are stored on a separate shelf in the walk-in.

c. Non-Refrigerated Storage:

1. Metal samples are stored in the log-in area in the appropriate cabinets.

2. Other samples not requiring refrigeration are stored on the appropriate shelves next to the walk-in refrigerator.

d. Any samples removed for analysis must be put back in the original location when the analysis is complete.

16. Sample Handling:

a. Samples are handled in and out of the walk-in coolers and black cabinets by the sample custodian.

b. Analysts are responsible for filling out sample request forms (see Figure 4) to request needed samples.

c. Forms are given to the sample custodian.

d. Sample custodian gathers requested samples and leaves yellow copy of form with samples, and keeps the white copy.

e. Analysts return samples to the walk-in accompanied by the yellow copy of the request form.

f. Next morning, sample custodian puts samples back in their proper place on the shelves.

g. Sample custodian signs off on white sheet that samples were returned. Any remarks are also noted on the white sheet.

17. Storage/Disposal:

Due to limited sample storage facilities available, the following guidelines have been created for sample storage/disposal. Sample storage time may be extended upon client's request.

a. One week after the report issue date, inorganic samples which are in cold storage are to be transferred to warm storage.

b. One month after the report issue date, organic samples which are in cold storage are to be transferred to warm storage.

c. Samples in warm storage should be disposed of as follows:

- Aqueous Samples: Dispose 1 month after report issue date.
- Non-Aqueous Samples: Dispose 6 months after report issue date.

Michael Linskens
Michael J. Linskens
Director, Technical Services

Kim Finner
Kim D. Finner
Laboratory Manager

Revision Date
08/18/87
08/15/88
3-18-90

APPENDIX D-4

ENSECO - AIR TOXICS LABORATORY

Enseco
A Corning Company

STANDARD OPERATING PROCEDURE

Subject or Title: Sample Identification for Enseco - Air Toxics		**Page** 1 **of** 4
SOP No.: LP-ATL-0002	**Revision No.:** Original	**Effective Date:** November 22, 1991
Supersedes: None		

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Prepared By: Valerie Zugates-Wildman	**Date:** November 19, 1991
Management Approval:	**Date:** 11/21/91
QA Officer Approval: Walter Berger	**Date:** 11-20-91

Subject or Title: Sample Identification for Enseco - Air Toxics	Page 2 of 4

SOP No.: LP-ATL-0002	Revision No.: Original	Effective Date: November 22, 1991

1. SCOPE AND APPLICATION

1.1 To describe the procedures used in the assignment of laboratory identification numbers to samples.

2. POLICIES

2.1 All Enseco Safety policies will be strictly adhered to while performing the following duties.

2.2 Ensure client confidentiality at all times.

2.3 Enseco security policy will be adhered to for all non-Enseco visitors entering the sample control receiving door.

3. SAFETY

3.1 All Enseco safety policies will be adhered to at all times.

3.2 All samples are considered hazardous. Always handle samples in the most safe condition as possible.

3.3 Always ask when there is a question about safety.

4. PROCEDURE

4.1 Each sample is assigned a unique identification number. The numbers are assigned when the project is logged into the laboratory.

4.2 Samples are logged into a logbook that is used to keep track of the next available project number. The following information is recorded in the logbook:

Date Received
Time Received
Sample Description
Analysis Required
Container Type
Date/Time Sampled
Client/Project
Initials of Person Logging in Project

Subject or Title: Sample Identification for Enseco - Air Toxics		Page 3 of 4
SOP No.: LP-ATL-0002	Revision No.: Original	Effective Date: November 22, 1991

4.3 The laboratory sample ID numbers consist of five parts:

4.3.1 "A-"

Every sample ID number begins with "A-" to identify that the project is from the Air Toxics laboratory of Enseco-CRL.

4.3.2 The year.

The last two digits of the current year.

4.3.3 The day.

The number of the day of the year that the samples are logged in. January 1 is "001" and December 31 is "365" ("366" in leap years).

4.3.4 The project.

Starting with "01", each project logged in is given a unique number.

4.3.5 Sample numbers.

Starting with "-001", each sample in the project is given a unique number.

Examples: Sample # A-9100503-006 would be sample #6 of the third project logged in on January 5, 1991. Sample # A-9036115-013 would be sample #13 of the 15th project logged in on December 27, 1990.

4.4 Each sample is labeled with its sample ID number by either attaching a sample ID tag or affixing a label.

Enseco
A Corning Company

STANDARD OPERATING PROCEDURE

Subject or Title: Sample Identification for Enseco - Air Toxics		Page 4 of 4
SOP No.: LP-ATL-0002	Revision No.: Original	Effective Date: November 22, 1991

5. Responsibilities

5.1 Sample Control Technician

5.1.1 The Sample Control Technician is responsible for following the procedures and policies set forth in this SOP in a safe and timely manner.

5.2 Laboratory Director

5.2.1 The Laboratory Director is responsible for ensuring the procedures and policies set forth in this SOP are followed in a safe and timely manner.

7. SAMPLE CUSTODY

Upon receipt by Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

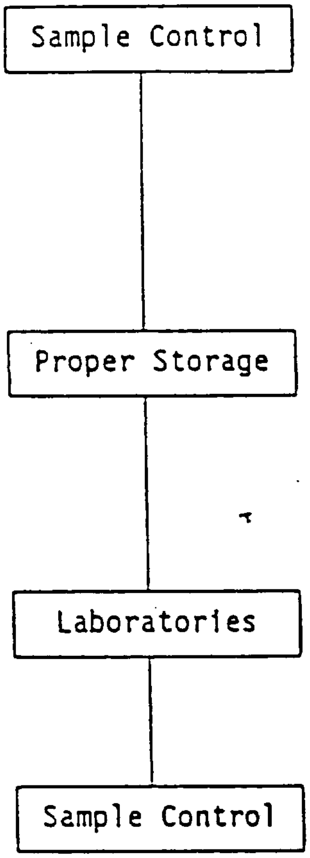
All samples are received by Enseco's Sample Control Group and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2. The Chain-Of-Custody Record (Interlaboratory Analysis Form) used to transmit samples between laboratories within Enseco is given in Figure 7-3.

Sample bottles provided to the client by Enseco are transmitted under custody using the Enseco "Sample Safe™".

Figure 7-1

ENSECO SAMPLE PROCESSING FLOW CHART



- * Check and document physical condition of sample
- * Verify documentation and parameter assignment
- * Log into LIMS
- * Send acknowledgement letter to client

- * Store sample according to preservation guidelines
- * Transfer sample to lab with proper documentation

- * Document analytical work
- * Return unused samples to Sample Control

- * Return sample to client or arrange for sample disposal

Enseco

CHAIN OF CUSTODY

No. 5068

SAMPLE SAFE™ CONDITIONS

1. Packed by: ____________ Seal # ________
2. Seal Intact Upon Receipt by Sampling Co.: Yes No
3. Condition of Contents: ____________
4. Sealed for Shipping by: ____________
5. Initial Contents Temp.: ________ °C Seal # ________
6. Sampling Status: Done Continuing Until ________
7. Seal Intact Upon Receipt by Laboratory: Yes No
8. Contents Temperature Upon Receipt by Lab: ________ °C
9. Condition of Contents: ____________

Attn: ____________

Enseco Client ____________

Project ____________

Sampling Co. ____________

Sampling Site ____________

Team Leader ____________

Date	Time	Sample ID/Description	Sample Type	No. Containers	Analysis Parameters	Remarks

CUSTODY TRANSFERS PRIOR TO SHIPPING

	Relinquished by: (signed)	Received by (signed)	Date	Time
1				
2				
3				

SHIPPING DETAILS

Delivered to Shipper by: ____________

Method of Shipment: ____________ Airbill # ____________

Received for Lab: ____________ Signed: ____________ Date/Time ____________

Enseco Project No. ____________

FIGURE 7-2

FIGURE 7-3

Page _______ of _______
File Index _______

Enseco

INTERLABORATORY ANALYSIS

SHIP TO: *(circle one)*

CAL ERCO CLE GAS MAR HOU

Attention:

SEND RESULTS TO:
Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611 FAC: (303) 431-7171

Attention:

CLIENT NAME

PROJECT NO.

Relinquished by: *(Signature)* Received by: *(Signature)* Date Time

Relinquished by: *(Signature)* Received by: *(Signature)* Date Time

Import Lab ID	Enseco ID	Client ID	Matrix (a, s, w)	Date Sampled	Date Rec'd	Date Auth.	Analysis Requested/ P.L. Item #	Sample Condition Upon Receipt

a. Written results required by (date): ______________ Verbal results required by (date): ______________

b. QC: ☐ Standard Enseco ☐ CLP Protocol ☐ Project-Specific ______________

c. Sample Disposal: ☐ Enseco ☐ Return to Client ☐ Phone RMAL

d. Raw Data Copies Needed: ☐ Yes ☐ No

e. Detection Limits: ☐ Standard Product ☐ Other*

f. Holding Times: ☐ Enseco ☐ EPA-CLP ☐ Other*

g. *Special Instructions: ______________

____

____

**h. Intercompany Rebate: *(circle one)* 0% 5% 10% i. P.O. Number ______________

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title:
BUILDING SECURITY

Page 1 of 2

SOP No.:
LP-RMA-0001

Revision No.:1.0
Original

Effective Date:
09/28/90

Supersedes:Original

1. Purpose:

The purpose of building security is to guarantee data security and confidentiality for the client as well as providing analytical data which is legally defensible.

2. Policies:

RMAL's security policy includes controlled access to the building, testing areas and data files, confidentially agreements with all personnel, identification badges for all personnel, electronic security and fire alarm systems. All visitors are also assigned visitor badges and are accompanied by RMAL staff during their stay in the facility.

3. Safety Issues: Not Applicable

4. Procedure:

<u>Building Security</u>

a. All exterior doors to the facility will remain locked at all times with the exception of the front entrance.
b. During the hours of 7:00 a.m. to 6:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entries. The alarm system is not activated during this time period.
c. The last employee to leave the facility must page the building to ensure he or she is the last person in the facility, they then call the alarm company to identify themself by name and code. The alarm system is activated, during this time period to prevent all other exterior doors from being usable, including sample receiving and the patio doors.

Prepared By: Date: 10-03-90

Management Approval: Date: 10-3-90

QA Officer Approval: Date: 10-3-90

STANDARD
OPERATING
PROCEDURE

SOP No.: LP-RMA-0001	Revision No.1 Original	Effective Date: 09/28/90

d. Sample receiving during the hours of 6:00 p.m.to 7:00 a.m. is permitted only with the assistance by a member of sample control.

Personnel Identification

a. All employees and visitors are required to wear security badges at all times while on the premises of all ENSECO divisions.

b. The personnel administrator is responsible for issuing a picture I.D. badge to an employee on the employee's first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file.

c. The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by an ENSECO employee before access to any building will be allowed.

Building Alarm System

a. Each employee will receive a copy of the security manual, personal security code and security training at the time of their orientation provided by a member of the Human Recorces department. The procedure is confidential information and can only be obtained from the Personnel Department.

5. Responsibilities:

a. It is the responsibility of each employee to maintain confidentiality of all clients data.

b. The Personnel Department is responsible for issuing employee identification badges and having signed "Confidentiality Agreements" in each employee's personnel file.

c. The receptionist is responsible for issuing visitor badges and for visitor sign-in during normal business hours.

d. Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

6. Comments:

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title:
Corrective Action - Refrigerator Ranges

Page 1 of 3

SOP No.:
LP-RMA-0052

Revision No.:
Revision 1.0

Effective Date:
May 28, 1992

Supersedes:
Original

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1. Purpose

The purpose of this standard operating procedure is to establish a policy in the event that refrigerator temperatures exceed the acceptable range.

2. Policies

2.1 The refrigerator temperature must be recorded daily and documented in a temperature logbook.

2.2 The temperature logbook must have acceptable temperature ranges posted in the front of the logbook (4°C +/- 2°C).

Prepared by:
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Date:
May 28, 1992

Management Approval:

Date:
7/2/92

QA Officer Approval:
Cindy Patterson

Date:
6/17/92

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0052	Revision 1.0	May 28, 1992

2.3 If the laboratories are not staffed during the weekend or on holidays, it is not necessary that the temperatures be monitored.

3. Safety Issues

3.1 If a refrigerator is not functioning properly, chemicals and samples may release volatile compounds. In some cases, chemicals can become reactive at these higher temperatures. Exposure to any chemicals or samples that are not refrigerated properly must be minimized. Proper safety apparatus includes, but is not limited to, gloves, safety glasses, breathing apparatus, and laboratory coats.

3.2 The toxicity and carcinogenicity of all chemicals must be considered. Each chemical should be treated as a health hazard, and proper procedures should be taken. A complete file of material safety data sheets (MSDS) is available in the standards lab.

4. Procedure

4.1 Calibrated thermometers must be used to monitor refrigerator temperatures. See RMAL SOP No. LE-RMA-002 for the calibration procedure for thermometers.

4.2 If the refrigerator temperature is out of the acceptable range this must be documented in the logbook. A decision is then made to either readjust the temperature, fill out a work order or move the samples to another cold storage area. The temperature is checked again within 24 hours and documented. Care should be taken to show that it is not out of range due to the frequent opening and closing of the refrigerator door. It is recommended that the temperature be checked the first time the door is opened in the morning.

4.3 If the decision is made to readjust the temperature in the refrigerator, this is to be documented in the logbook.

4.4 If the problem with the refrigerator can be fixed immediately, and the decision is made that sample and/or chemicals can stay in the refrigerator, this decision must be documented in the refrigerator logbook.

4.5 If refrigeration completely fails, all chemicals and samples must be immediately relocated to another appropriate refrigerator for storage. Corrective action and interim storage locations must be documented in the refrigerator logbook.

5. Responsibilities

5.1 One person will be assigned to monitor the refrigerator temperatures daily.

5.2 The team associate that is assigned is responsible for the adjustment or repair of the refrigerator.

5.3 The assignment of monitoring the refrigerators will be coordinated among the teams.

6. Comments

None.

7. Definitions

None.

APPENDIX E

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

Scope and Application:

This standard operating procedure (SOP) describes Warzyn's procedure for evaluating inorganic data according to procedures specified in a Quality Assurance Project Plan (QAPP). Each analytical procedure is specific to a project and is found or referenced in the QAPP. The reviewer must evaluate whether the procedure was adhered to and that the required Quality Control (QC) requirements were met as described in the QAPP and evaluate the usability of the data.

References:

1. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, Hazardous Site Evaluation Division, U.S. EPA, July, 1988 (or most current).

2. Statement of Work for Inorganics Analysis, U.S. EPA Contract Laboratory Program, SOW No. 788, July, 1988 (or most current).

3. Quality Assurance Project Plan (QAPP) containing the analytical procedures required for evaluation. Each QAPP is specific to a project.

Requirements:

1. It is recommended that the reviewer be familiar with the RAS Inorganics validation process (refer to the Validation Guidelines and Statement of Work above) prior to evaluating this data. The evaluation and action criteria described in this SOP are very similar to the RAS Inorganics validation process.

2. All completed validated data packages must include the following:

   a. Data Validation Narrative
   b. Validated Results; with data qualifiers added and pages stamped "VALIDATED".

NOTE: It is not necessary to include the raw data with the completed validated data package, since a copy of the raw data package already exists in the project file. Only include raw data if there are major concerns or deficiencies with the data set.

Procedure:

1. Screen the data package to see that the samples and analytes undergoing evaluation concur with what was requested on the "Request for Data Validation" form (refer to Figure 1). If discrepancies occur, contact the data management coordinator for clarification prior to continuing.

2. Review the QAPP and the specific analytical procedure for the analyte undergoing evaluation. They will contain:

   a. A summary of the method required and method reference,
   b. The applicable matrices and recommended hold times,
   c. An approximate number of samples and matrix,
   d. Special technical instructions,
   e. Required deliverables,
   f. Data Requirements (Detection Limits, Precision, etc.), and
   g. Quality Control Requirements.

3. The reviewer must evaluate the data according to the quality control criteria specified in the QAPP. The evaluation of each analyte, in most cases, will include a review of the following criteria:

   a. Methodology,
   b. Raw Data/Quality Control Deliverables,
   c. Detection Limits,
   d. Holding Times,
   e. Calibration (Initial and Continuing),
   f. Blanks (Calibration and Preparation),
   g. Laboratory Control Samples (EPA Reference Samples),
   h. Laboratory Duplicates,
   i. Matrix Spikes,
   j. Sample Result Verification,
   k. Field Duplicates,
   l. Other (as specified in the procedure),
   m. Overall Assessment for a Case.

4. Evaluate the data for the criteria above as follows:

A. METHODOLOGY

Evaluation

1. Examine the data to determine if the acceptable analytical methodology was used as indicated in the QAPP.

2. Determine if the analytical procedure was followed.

Action

1. If an acceptable analytical method was not used, contact the Project Manager. It is the Project Manager's responsibility to inform the reviewer to continue the validation process of the data as received, or to reject the data. In some cases reanalysis or

resampling may be required, however the decision is left up to the Project Manager. The Project Manager may require that the reviewer determine the "usability" of the data package as submitted prior to deciding what formal action should be taken.

2. If the analytical procedure was not followed as stated, use professional judgement in determining whether the data should be qualified as estimated or unusable.

B. RAW DATA/QUALITY CONTROL DELIVERABLES

Evaluation

1. The QAPP will specify what information should be submitted as part of the data package. Determine whether the data package submitted is complete as described in the QAPP. Usually the following will be required:
   - Method used,
   - Calibration/standardization information,
   - Bench records tabulating the order of analysis of samples, standards, blanks, duplicates and spikes with the resulting instrument readouts and final concentrations. Records submitted should be complete enough such that all results could be recalculated from this raw data.
   - Photocopy of all instrument readouts (strip charts, printer tapes, etc.)
   - Reference (QC) samples to be identified by source, lot number and sample number, with corresponding true values and 95% confidence limits provided.
   - Custody records

Action

1. If the data package is not complete, contact the data management coordinator. The data management coordinator will contact the appropriate laboratory or project manager and obtain the information necessary to complete the evaluation process.

C. DETECTION LIMITS

Evaluation

1. Evaluate whether the detection limits required in the QAPP were achieved by the method used.

Action

1. If detection limits were not reported at least to what was required in the QAPP, review the raw data to determine if the detection limits were achievable. If they were, recalculate the data and report results to the required detection limits.

2. If detection limits were not achievable by the laboratory, determine why (laboratory sensitivity problem or detection limits unobtainable for the method used. Inform the project manager of

the situation. The project manager will determine whether the evaluation should be completed.

D. HOLDING TIMES

Evaluation

1. Determine whether the established holding times were met. The holding time is established by comparing the DATE SAMPLED with the DATE OF ANALYSIS found on the raw data. Required hold times should be noted in the QAPP.

2. Examine the digestion/distillation logs to determine if samples were preserved at the proper pH.

Action

1. If hold times were not noted in the QAPP, use the EPA-recommended hold times and apply these hold times to both water and soil samples.

2. If holding times and preservation criteria are not met, qualify all results $>$ Instrument Detection Limit (IDL) and estimated (J) and results $<$ IDL as estimated (UJ).

3. Use professional judgement in cases where the holding time is grossly exceeded. The expected bias would be low and the reviewer may determine that results $<$ IDL are unusable (R).

E. CALIBRATION

Evaluation

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank. In cases where a curve set-up is not applicable (i.e. a titrimetric procedure), verify that the titrants were standardized as required in the procedure.

2. Verify that the correlation coefficient is ≥ 0.995 (unless otherwise specified).

3. Verify that the calibration verification standards (ICV/CCV) were run at the appropriate frequency and that results were within acceptable limits as stated in the QAPP.

4. Recalculate approximately 10% of the ICV/CCV using the following equation:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows.

Action

1. If the minimum number of standards as required in the QAPP were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).

2. If the correlation coefficient is < 0.995 (or as specified in the QAPP), qualify results > IDL as estimated (J) and results > IDL as estimated (UJ).

3. If standardization of titrants was required and not performed, use professional judgement in qualifying the data. The reviewer should review ICV/CCV, EPA reference sample results and other criteria prior to determining if the data should be qualified as estimated (J if > IDL; UJ if < IDL) or unusable (R).

4. If the ICV/CCV %R falls outside the acceptable limits, use professional judgement to qualify associated samples. If possible, indicate the bias in the review. The following guidelines may assist the reviewer in qualifying the data:

   - If the ICV/CCV %R is significantly greater than the upper control limit (UCL), qualify associated results > IDL as unusable (R); results < IDL are acceptable.

   - If the ICV/CCV %R is significantly lower than the lower control limit (LCL), qualify all associated data as unuseable (R)

   - If the ICV/CCV %R are outside the acceptable limits, yet relatively close to the limits, qualify associated results as estimated (J if > IDL; UJ if < IDL and the %R is below the LCL).

F. BLANKS

Evaluation

1. Review the raw data for all blanks and verify the results were accurately reported.

2. No contaminants should be found in the blanks (i.e. all blank results should be < IDL).

3. Positive blank results must not be corrected by subtracting off the blank value from the sample results.

Action

1. Sample results ≥ IDL but less than 5 times the maximum concentration found in any blank should be qualified as not detected (U).

2. Any blank with a negative result whose absolute value is > IDL must be carefully evaluated to determine if the sample data is biased in any way.

G. LABORATORY CONTROL SAMPLES (EPA REFERENCE SAMPLES)

Evaluation

1. The laboratory control sample (LCS) serves as a monitor of the overall performance of the analysis including all preparation steps. Review the LCS and verify that the results fall within the control limits required. (If no limits noted, use 80-120% for the control limits.)

2. Check the raw data to verify the reported recoveries. Recalculate one or more recoveries (%R) using the following equation:

$$\text{LCS \%R} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Action

1. If results are < IDL and the LCS recovery is above the UCL, the data are acceptable.

2. If the LCS recovery for any analyte falls within the range of 50-LCL, or > UCL, qualify results > IDL as estimated (J).

3. If results are < IDL and the LCS recovery falls within the range of 50-LCL, qualify the affected results as estimated (UJ).

4. If the LCS recovery results are < 50%, qualify the data for the affected analytes as unusable (R).

5. If a LCS was required and not run, use professional judgement to determine whether the data is estimated (J) or unusable (R).

H. LABORATORY DUPLICATES

Evaluation

1. Review the duplicate data and verify that the results fall within the criteria required. If no criteria exist, use the limits of 20% for the Relative Percent Difference (RPD) or ± IDL if results are < 5x IDL (35 RPD or ± 2x IDL if > 5x IDL for soils).

2. Review the data and verify that the duplicate analysis was not performed on a field blank.

3. Check the raw data and recalculate one or more RPD using the following:

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

where, S = Original sample value
D = Duplicate sample value.

Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same sample matrix as estimated (J).

2. If the field blank was used for duplicate analysis, carefully evaluate all other quality control data and use professional judgement in evaluating the data.

I. MATRIX SPIKES

Evaluation

1. Review the matrix spike data and verify that the results fall within the limits specified. If no criteria exists, use the limits of 75-125% for both water and soil matrices.

2. Check the raw data and recalculate the % Recovery of at least 10% of the data. Use the following equation to verify that the results were calculated correctly:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Review the data and verify that the field blank was not used for the spike analysis.

Action

1. If the spike recovery is $>$ 125% (or $>$ UCL stated in the procedure) and the sample results are $<$ IDL, the data is acceptable for use.

2. If the spike recovery is $>$ 125% or $<$ 75% ($>$ UCL or $<$ LCL stated in the procedure) and the sample results are $>$ IDL, qualify the data for these samples as estimated (J).

3. If the spike recovery falls within the range of 30-74% (30-LCL) and the sample results are $<$ IDL, qualify the data for these samples as estimated (UJ).

4. If any spike recovery results are $<$ 30% and the sample results are $<$ IDL, qualify the data for these samples as unusable (R).

5. In cases where more than one spike analysis was performed and one or more %R results were outside acceptable ranges, use professional judgement when qualifying the data. Matrix spikes should be performed on each sample matrix. Qualify data of similar matrix as the spiked sample if different matrices in the data package.

6. If the field blank was used for the spike analysis, carefully evaluate all other quality control criteria and use professional judgement in evaluating the data.

J. SAMPLE RESULT VERIFICATION

Evaluation

1. Examine the raw data to verify the analyte quantitation was calculated as stated in the procedure. Re-calculate a minimum of 10% of the sample results to verify results were calculated correctly.

2. Examine the raw data for anomalies such as baseline drift, negative absorbances (indicative of interfences), omissions, etc.

3. Verify there are no transcription or reduction errors (dilutions, percent solids, sample weights) on at least 10% of the samples.

4. Verify that all results fall within the calibrated range.

Action

1. If any discrepancies are found and can be corrected, make the corrections and note. Increase the level of quality control if necessary. If the discrepancy cannot be resolved by the reviewer, contact the data management coordinator. The laboratory or appropriate agency will be contacted and the information necessary to complete the evaluation will be requested. The project manager should be informed of the delay in the validation process. If any discrepancy remains unresolved, use professional judgement in qualifying the data.

K. FIELD DUPLICATES

Evaluation

1. Field duplicate samples measure field and lab precision and therefore the results may have more variability than lab duplicates. Review the field duplicate data and calculate the RPD. No criteria exists for evaluating the data.

Action

1. If the RPD is > 50%, note in the narrative. Use professional judgement in qualifying any data. Review the raw data to verify that no reduction errors exist. This is to verify that the discrepancy is due to sampling techniques (sampling, preservation,

filtering, etc.) rather than laboratory error. Make notation of the discrepancy in the narrative.

L. OTHER (as specified in the procedure)

Evaluation

1. Review the procedure for any other quality control criteria not covered under the above sections. Examples of additional information required may be: ICP interference check samples, special AA quality control checks, distilled/digested blanks and standards, standardization, specific methodology requests for different concentration levels, etc..

Action

1. If specific quality control criteria were required and not followed, the project manager should be informed.

2. If the data was outside the acceptable ranges, use professional judgement in qualifying the data. Clearly identify your action and justification of the action in the narrative.

M. OVERALL ASSESSMENT FOR A CASE

1. It is appropriate for the reviewer to make professional judgements and express concerns and comments on the validity of the overall data package. This is particularly appropriate when several QC criteria are outside specifications. It is the reviewers responsibility to thoroughly document and explain all data validation qualifiers added to the data.

2. The following is a summary of data validation qualifier definitions which can be used in evaluating the data:

U	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
J	The associated value is an estimated quantity due to quality control criteria not being met.
R	The data are unusable. The analyte may or may not be present.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

5. Once the data has been evaluated against all of the points described in part 4, the evaluator can prepare the data validation narrative.

The data validation narrative should be a summary of the data quality indicating any problems or deficiencies found with the data set. If possible, on data qualified as estimated, note if the data is biased high or low. It is easiest to discuss each analyte separately. Refer to Figure 2 for the form to be used when writing the data validation narrative.

6. Record any data validation qualifiers (DVQ) to the left of the results on the analytical report. The laboratory qualifiers (LQ) will usually recorded to the right of the result by the laboratory who performed the analytical work. A copy of the lab qualifier definitions should be supplied with the data. Data entry people need to be able to distinguish between DVQ and LQ, so note location of qualifiers if located other than above.

7. Record the sample ID and date sampled as noted on the Chain of Custody on the analytical report.

8. Hilight POSITIVE-HIT data with a hilighter.

9. Stamp all of the analytical reports "VALIDATED". A "VALIDATED" stamp can be obtained from the data management coordinator.

10. Sign and date the Data Validation Request Form.

11. A quality control review needs to be performed on the validated data package. The entire validated data package (including raw data, evaluator's notes and narrative) should be submitted to the QC reviewer for a final review.

Quality Control Review:

1. The purpose of this quality control review is to verify that the data were validated according to the guidelines above, that all data validation qualifiers were added and also that the narrative is complete.

2. Review the validated data package for completeness. The data package should contain the following:

   - Data Validation Request Form
   - Inorganic Data Validation Narrative
   - Validated Analytical Results

3. Verify that results exist for all samples and all analytes requested in the data package.

4. Review the data validation narrative with the validated results for consistency, i.e., if the narrative stated that a particular analyte was qualified, verify that the qualifier has been recorded with the associated data. Check approximately 10% of the qualifiers. If discrepancies are found, increase the level of QC checks.

5. Review the narrative; check that the data has been evaluated according to the quality control criteria specified in the QAPP.

6. Verify that all positive-hit data have been hilighted and that the sample descriptions and sampling dates have been recorded on every result page.

7. Verify that the reviewer has signed and dated the validated data package.

8. If no discrepancies exist, sign and date the Data Validation Request Form. If the data package requires clarification, review the raw data and/or obtain clarification from the reviewer.

9. Deliver the final validated data package to the Data Management Coordinator when the quality control review is completed.

10. Recycle any raw data. Raw data is not submitted with the final validated data package.

INORGANICS
DATA VALIDATION NARRATIVE

Site Name ______________ Project # ______________

SMO Case # (SAS #) ______________ #Samples/Matrix ______________

Laboratory ______________ Hours for Review ______________

SOW # ______________ EPA Validation Guidelines # ______________

Sample Numbers ______________

Validated By ______________ Date ______________

Reviewed By ______________ Date ______________

SUMMARY OF REVIEW:

CAW/dlk/KJD
[dlk-601-22]
60721-MD