perfectION™ Guidebook

perfectION™
Combination Lead Electrode
Successful Ion Measurement

METTLER TOLEDO

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1. Introduction

This user guide contains information on the preparation, operation and maintenance for the lead ion selective electrode (ISE). General analytical procedures, electrode characteristics and electrode theory are also included in this user guide. Lead electrodes measure free lead ions in aqueous solutions quickly, simply, accurately and economically.

perfectION™ Combination Lead Electrode

The reference and sensing electrodes are built into one electrode, which decreases the amount of required solution and reduces waste. The built-in Click & Clear™ reference junction prevents clogging of the diaphragm and provides fast and stable readings.

The perfectION™ Combination Lead Electrode is available with a BNC connector (P/N 51344730) and a Lemo connector (P/N 51344830) for METTLER TOLEDO titrators.

2. Required Equipment

1. METTLER TOLEDO ISE meter, such as the SevenMulti™ benchtop meter or the SevenGo pro™ portable meter, or a METTLER TOLEDO titrator, such as the Tx (T50, T70, T90) Excellence or G20 Compact titrators.

   METTLER TOLEDO combined ISEs can be used on any ISE meter with a BNC connection.

2. perfectION™ combined lead ion selective electrode

3. Stirrer

4. Volumetric flasks, graduated cylinders, beakers and pipettes. Plastic labware is required for low-level lead analysis.

5. Distilled or deionized water

6. Ion Electrolyte B Reference filling solution (P/N 51344751)

7. Lead standard solution 1000 mg/L (P/N 51344780)

8. Methanol-formaldehyde. The methanol-formaldehyde solution decreases the solubility and retards the oxidation of the electrode sensing element. Add the methanol-formaldehyde solution in a 1:1 ratio to all samples and standards.

   Preparation note:
   Methanol-formaldehyde solution – Add 3 drops of 37% formaldehyde to 1 liter of reagent-grade methanol.

9. Lead ionic strength adjuster (ISA). ISA provides a constant background ionic strength for samples and standards.

   Preparation note:
   5 mol/L $NaClO_4$ – Add 80.25 g of reagent-grade $NaClO_4 \cdot H2O$ to a 100 mL volumetric flask. Add 50 mL of distilled water and mix the solution until the solids are dissolved. Dilute to volume with distilled water and mix the solution thoroughly.

3. Electrode and Measurement Setup

Electrode Preparation

Remove the protective shipping cap from the sensing element and save the cap for storage. Fill the electrode with Ion Electrolyte B Reference filling solution.

Electrode Filling Instructions:

1. Install the flip spout cap on the filling solution bottle and lift the flip spout on the bottle to a vertical position.

2. Insert the spout into the fill hole on the outer body of the electrode and add a small amount of filling solution to the reference chamber. Invert the electrode to moisten the O-ring and return the electrode to the upright position.

3. Hold the electrode body with one hand and use your thumb to push down on the electrode cap to allow a few drops of filling solution to drain out of the electrode.

4. Release the electrode cap. If the sleeve does not return to its original position, see if the O-ring is moist and repeat steps 2 through 4 until the sleeve returns to the original position.

5. Add filling solution to the electrode up to the filling hole.

Note: Add filling solution each day before using the electrode. The filling solution level should be at least 2.5 cm above the level of sample in the beaker to ensure a proper flow rate. The fill hole should always be open when taking measurements.
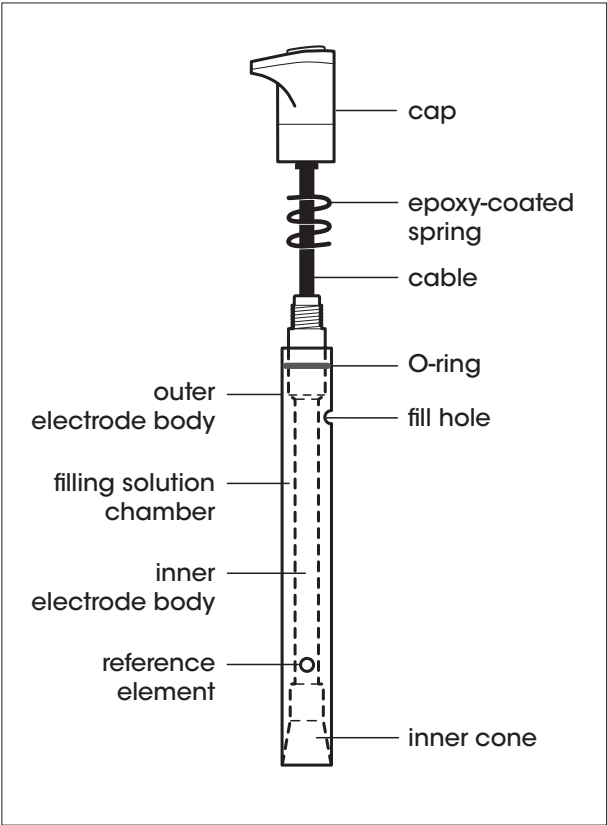
cap

epoxy-coated spring

cable

O-ring

outer electrode body

fill hole

filling solution chamber

inner electrode body

reference element

inner cone

Figure 1 – *perfectION™ Lead combination electrode*

Checking Electrode Operation (Slope)

These are general instructions that can be used with most meters to check the electrode operation.

This procedure measures the electrode slope. Slope is defined as the change in millivolts observed with every tenfold change in concentration. The slope value provides the best means for checking the electrode operation.

1. If the electrode has been stored dry, prepare the electrode as described in the **Electrode Preparation** section.
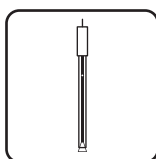
2. Connect the electrode to a meter with a mV mode. Set the meter to the mV mode.
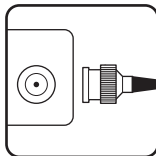
3. Add 50 mL of distilled water, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA into a 150 mL beaker. Stir the solution thoroughly.
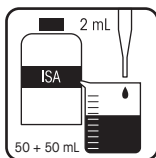
4. Rinse the electrode with distilled water and place the electrode into the solution prepared in step 3.
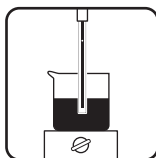
5. Select either a 0.1 mol/L or 1000 mg/L lead standard. Pipette 1 mL of the standard into the beaker and stir the solution thoroughly. When a stable reading is displayed, record the electrode potential in millivolts.
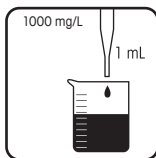
6. Pipette 10 mL of the same standard into the same beaker and stir the solution thoroughly. When a stable reading is displayed, record the electrode potential in millivolts.
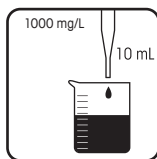
7. There should be a 25 to 30 mV difference between the two millivolt readings when the solution temperature is between 20 to 25 °C. If the millivolt potential is not within this range, refer to the **Troubleshooting** section.
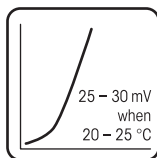
Sample Requirements

The epoxy body of the lead electrode is resistant to damage by aqueous solutions. The electrode may be used intermittently in solutions that contain methanol, benzene or acetone.

Samples and standards should be at the same temperature. A 1 °C difference in temperature for a 10^{-3} mol/L lead solution will give rise to about a 2% error.

The solution temperature must be less than 50 °C.

In all analytical procedures, ISA must be added to all samples and standards before measurements are taken.

Measuring Hints

Lead concentration can be measured in moles per liter (mol/L), milligrams per liter (mg/L) or any convenient concentration unit.

Table 1 – *Lead Concentration Unit Conversion Factors*

mol/L	mg/L Lead (Pb^{2+})
1.0	207200
10^{-1}	20720
10^{-2}	2072
4.83×10^{-3}	1000
10^{-3}	207.2
10^{-4}	20.72
4.83×10^{-6}	1

- Stir all standards and samples at a uniform, moderate rate. Place a piece of insulating material, such as Styrofoam or cardboard, between the magnetic stirrer and beaker to prevent measurement errors from the transfer of heat to the sample.

- Always use freshly prepared standards for calibration.

- Always use the methanol-formaldehyde solution when it is recommended.

- Always rinse the electrode with distilled water between measurements and shake the electrode to remove the water and prevent sample carryover. Do not wipe or rub the electrode sensing element.

- Allow all standards and samples to come to the same temperature for precise measurements.

- Concentrated samples (greater than 10^{-1} mol/L lead) should be diluted before measurement.

- Verify the electrode calibration every two hours by placing the electrode in a fresh aliquot of the least concentrated standard used for calibration. If the value has changed by more than 2%, recalibrate the electrode.

- After immersing the electrode in a solution, check the electrode sensing surface for air bubbles and remove air bubbles by reimmersing the electrode in the solution and gently tapping it.

- For high ionic strength samples, prepare standards with a background composition similar to the sample.

- The fill hole cover must be open during measurements to ensure a uniform flow of reference filling solution.

- If the electrode is used in dirty or viscous samples or the electrode response becomes sluggish, empty the electrode completely, hold the junction open and flush the junction with distilled water. Empty any water from the electrode and refill it with fresh filling solution. Press down on the electrode cap to let a few drops of the filling solution flow out of the electrode and then replenish any lost solution.

- Start the calibration or measurement with the lowest concentrated standard or sample.

Electrode Storage and Maintenance

Electrode Storage

For storage between measurements and up to one week, store the electrode in a 4 mol/L potassium chloride solution. Do not add ISA to the storage solution. The filling solution inside the electrode should not be allowed to evaporate, as crystallization will result.

For storage longer than one week, drain the electrode, flush the chamber with distilled water and store the electrode dry with the protective shipping cap covering the sensing element.

Polishing the Lead Combination Electrode

The sensing surface of solid state electrodes can wear over time, which causes drift, poor reproducibility and loss of response in low-level samples. The electrode can be restored by polishing the sensing surface with a polishing strip. The polishing strip can also be used if the sensing surface has been etched or chemically poisoned.

1. Cut off about an inch of the polishing strip.

2. Hold the electrode with the sensing surface facing up.

3. Place a few drops of distilled water on the sensing surface.

4. With the frosted side of the polishing strip facing down, use light finger pressure to place the polishing strip on top of the sensing surface.

5. Rotate the electrode for about 30 seconds.

6. Rinse the electrode with distilled water and soak the electrode in a 1 mg/L or 10^{-5} mol/L lead standard for ten minutes.

Flushing the Lead Combination Electrode

If the area between the electrode sleeve and inner cone becomes clogged with sample or precipitate, flush the area with filling solution or distilled water.

1. Hold the electrode body with one hand and use your thumb to push down on the electrode cap to drain the electrode. Push down on the cap until all the filling solution is drained from the chamber.

2. Fill the electrode with distilled water and then push down on the cap until all the water is drained from the chamber.

3. Fill the electrode with fresh filling solution up to the fill hole. Push down on the cap to allow a few drops of filling solution to drain out of the electrode and replenish the lost filling solution.

Disassembling and Reassembling the Lead Combination Electrode

Note: Disassembly is usually not required and should not be done unless a thorough cleaning is required.

1. Tip the electrode so the filling solution moistens the O-ring on the electrode body. Hold the electrode body with one hand and use your thumb to push down on the electrode cap to drain the electrode.

2. Unscrew the cap counterclockwise and then slide the cap and spring up the cable.

3. Hold the outer sleeve with one hand and firmly push down on the threaded portion with your thumb and forefinger to separate the inner body from the sleeve.

4. Grasp the inner cone with a clean, lint-free tissue and withdraw the body from the sleeve using a gentle twisting motion. Do not touch the pellet above the cone, as it will damage the pellet. Rinse the outside of the electrode body and the entire sleeve with distilled water. Allow it to air dry.

5. Moisten the O-ring on the electrode body with a drop of filling solution. Insert the screw-thread end of the electrode body into the tapered, ground end of the sleeve.

6. Push the body into the sleeve using a gentle twisting motion until the bottom surface of the inner cone is flush with the tapered end of the sleeve.

7. Place the spring onto the electrode body and screw on the cap. Refill the electrode with filling solution.

Serial Delutions

Serial dilution is the best method for the preparation of standards. Serial dilution means that an initial standard is diluted, using volumetric glassware, to prepare a second standard solution. The second standard is similarly diluted to prepare a third standard, and so on, until the desired range of standards has been prepared.

1. **To prepare a 100 mg/L lead standard** – Pipette 10 mL of the 1000 mg/L standard into a 100 mL volumetric flask. Dilute to the mark with deionized water and mix well.

2. **To prepare a 10 mg/L standard** – Pipette 10 mL of the 100 mg/L standard into a 100 mL volumetric flask. Dilute to the mark with deionized water and mix well.

3. **To prepare a 1 mg/L standard** – Pipette 10 mL of the 10 mg/L standard into a 100 mL volumetric flask. Dilute to the mark with deionized water and mix well.

To prepare standards with a different concentration use the following formula:

$$C_1 * V_1 = C_2 * V_2$$

C_1 = concentration of original standard
V_1 = volume of original standard
C_2 = concentration of standard after dilution
V_2 = volume of standard after dilution

For example, to prepare 1000 mL of a 100 mg/L lead standard from a 20720 mg/L lead standard:

C_1 = 20720 mg/L lead
V_1 = unknown
C_2 = 100 mg/L lead
V_2 = 1000 mL
20720 mg/L * V_1 = 100 mg/L * 1000 mL
V_1 = (100 mg/L * 1000 mL) / 20720 mg/L = 4.8 mL

4. Analytical Techniques

A variety of analytical techniques are available to the analyst. The following is a description of these techniques.

Direct Calibration is a simple procedure for measuring a large number of samples. Only one meter reading is required for each sample. Calibration is performed using a series of standards. The concentration of the samples is determined by comparison to the standards. ISA is added to all solutions to ensure that samples and standards have similar ionic strength.

Low-Level Calibration is similar to the direct calibration technique. This method is recommended when the expected sample concentration is less than 1 mg/L or 5×10^{-6} mol/L lead. A minimum three point calibration is recommended to compensate for the electrode's non-linear response at these concentrations. A special calibration standard preparation procedure is the best means of preparing low-level calibration standards.

Incremental Techniques provide a useful method for measuring samples, since a calibration is not required. The different incremental techniques are described below. They can be used to measure the total concentration of a specific ion in the presence of a large (50 to 100 times) excess of complexing agents. As in direct calibration, any convenient concentration unit can be used.

• **Known Addition** is useful for measuring dilute samples, checking the results of direct calibration (when no complexing agents are present), or measuring the total concentration of an ion in the presence of an excess complexing agent. The electrode is immersed in the sample solution and an aliquot of a standard solution containing the measured species is added to the sample. From the change in potential before and after the addition, the original sample concentration is determined.

Titrations are quantitative analytical techniques for measuring the concentration of a species by incremental addition of a reagent (titrant) that reacts with the sample species. Sensing electrodes can be used for determination of the titration end point. Ion selective electrodes are useful as end point detectors, because they are unaffected by sample color or turbidity. Titrations are approximately 10 times more precise than direct calibration.

	Direct	Small Volume Direct	Low-Level	Known Addition	Titration
$[Pb^{+2}] < 1.0$ mg/L			✔		
$[Pb^{+2}] > 1.0$ mg/L	✔	✔		✔	✔
Increased accuracy					✔
Occasional Sampling				✔	
Small sample volume		✔			
Large number of samples	✔		✔	✔	
Reduce chemical usage		✔			
Field measurement		✔			
Ionic strength greater than 0.1 mol/L				✔	
Complicated background					✔

Direct Calibration Technique

Typical Direct Calibration Curve

In the direct calibration procedure, a calibration curve is constructed either in the meter memory or on semi-logarithmic paper. Electrode potentials of standard solutions are measured and plotted on the linear axis against their concentrations on the log axis. In the linear regions of the curves, only two standards are needed to determine a calibration curve. In non-linear regions, more points must be taken. These direct calibration procedures are given for concentrations in the region of linear electrode response. Low-level measurement procedures are given in a following section for measurements in the non-linear electrode region.
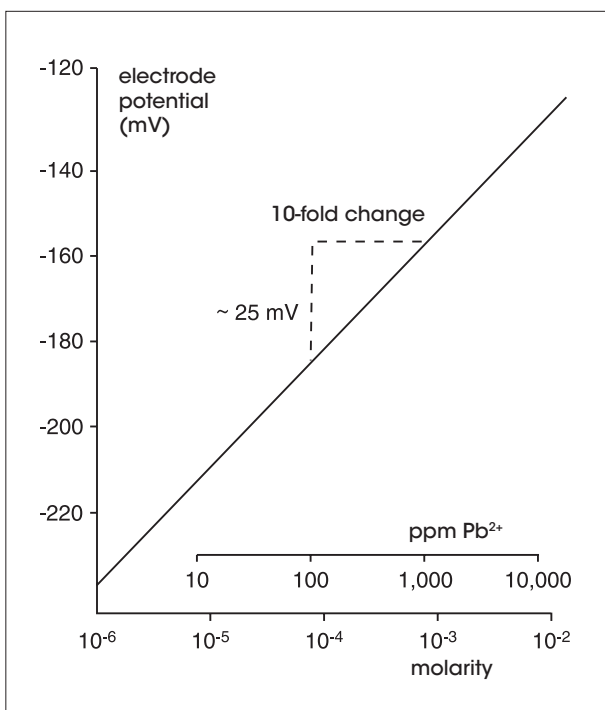

Figure 2 – *Typical Direct Calibration Curve*

Direct Calibration Overview

The following direct measurement procedures are recommended for moderate to high level measurements. Samples must be in the linear range of the electrode – greater than 1 mg/L or 5×10^{-6} mol/L lead. A two point calibration is sufficient, although more points can be used. When using an ISE meter, sample concentrations can be read directly from the meter. When using a mV meter, a calibration curve can be prepared on semi-logarithmic graph paper, or a linear regression (against logarithmic concentration values) can be performed using a spreadsheet or graphing program.

Calibration Hints

- Standard concentrations should bracket the expected sample concentrations.

- Always add 2 mL of ISA and 50 mL of the methanol-formaldehyde solution per 50 mL of standard or sample.

- For high ionic strength samples that have an ionic strength of 0.1 mol/L or greater, prepare standards with a background composition similar to that of the samples, or measure the samples using the known addition method.

- During calibration, measure the least concentrated standard first, and work up to the most concentrated standard.

Direct Calibration Setup

1. Prepare the electrode as described in the **Electrode Preparation** section.

2. Connect the electrode to the meter.

3. Prepare at least two standards that bracket the expected sample range and differ in concentration by a factor of ten. Standards can be prepared in any concentration unit to suit the particular analysis requirement. See the **Serial Dilution** section for instructions on how to prepare standards. All standards should be at the same temperature as the samples. For details on temperature effects on electrode performance, refer to the **Temperature Effects** section.

Direct Calibration Procedure Using a Meter with an ISE Mode

*Note***:** *See the meter user guide for more specific information.*

1. Add 50 mL of the less concentrated standard, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA to a 150 mL beaker and stir the solution thoroughly.

2. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the less concentrated standard. Wait for a stable reading and adjust the meter to display the value of the standard, as described in the meter user guide.

3. Add 50 mL of the more concentrated standard, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA to a second 150 mL beaker and stir the solution thoroughly.

4. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the more concentrated standard. Wait for a stable reading and adjust the meter to display the value of the second standard, as described in the meter user guide.

5. Record the resulting slope value. The slope should be between 25 and 30 mV when the standards are between 20 and 25 °C.

6. Add 50 mL of sample, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA to a clean 150 mL beaker and stir the solution thoroughly.

7. Rinse the electrode with distilled water, blot it dry and place it into the sample. The concentration of the sample will be displayed on the meter.

Note: *Other solution volumes may be used, as long as the ratio of solution to methanol-formaldehyde to ISA remains 50:50:2.*

Direct Calibration Procedure Using a Meter with a mV Mode

Note: See the meter user guide for more specific information.

1. Set the meter to the mV mode.

2. Add 50 mL of the less concentrated standard, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA to a 150 mL beaker and stir the solution thoroughly.

3. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the less concentrated standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.

4. Add 50 mL of the more concentrated standard, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA to a second 150 mL beaker and stir the solution thoroughly.

5. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the more concentrated standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.

6. Using semi-logarithmic graph paper, prepare a calibration curve by plotting the millivolt values on the linear axis and the standard concentration values on the logarithmic axis.

7. Add 50 mL of sample, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA to a clean 150 mL beaker and stir the solution thoroughly.

8. Rinse the electrode with distilled water, blot it dry and place it into the beaker. When a stable reading is displayed, record the mV value.

9. Use the calibration curve prepared in step 6 in order to determine the unknown concentration of the sample.

Note: Other solution volumes may be used, as long as the ratio of solution to methanol-formaldehyde to ISA remains 50:50:2.

Small Volume Direct Calibration Technique

Take advantage of special design features available with the perfectION™ combination lead electrode to meet your measuring needs. Due to the Click & Clear™ reference, this electrode is able to measure sample volumes as small as 5 mL using a modified direct measurement procedure. Because less solution volume is required, the chemical usage of lead standards and ISA is reduced. All samples should have a concentration greater than 1 mg/L or 5×10^{-6} mol/L lead. A two point calibration is sufficient, although more points can be used. The following procedure recommends using 25 mL of sample. Smaller sample volumes can be used, as long as the final volume of solution is sufficient to cover the bottom of the electrode.

Calibration Hints

- Standard concentrations should bracket the expected sample concentrations.

- Always keep the ratio of solution to methanol-formaldehyde to ISA at 50:50:2.

- For high ionic strength samples that have an ionic strength of 0.1 mol/L or greater, prepare standards with a background composition similar to that of the samples, or measure the samples using the known addition method.

- During calibration, measure the least concentrated standard first, and work up to the most concentrated standard.

- Calibrate with the same volume of standard as the volume of sample that is available for analysis.

Small Volume Direct Calibration Setup

1. Prepare the electrode as described in the **Electrode Preparation** section.

2. Connect the electrode to the meter.

3. Prepare at least two standards that bracket the expected sample range and differ in concentration by a factor of ten. Standards can be prepared in any concentration unit to suit the particular analysis requirement. See the **Serial Dilution** section for instructions on how to prepare standards. All standards should be at the same temperature as the samples. For details on temperature effects on electrode performance, refer to the **Temperature Effects** section.

Small Volume Direct Calibration Procedure Using a Meter with an ISE Mode

Note: See the meter user guide for more specific information.

1. Add 25 mL of the less concentrated standard, 25 mL of the methanol-formaldehyde solution and 1 mL of ISA to a 50 mL beaker and swirl the solution to mix.

2. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the less concentrated standard. Wait for a stable reading and adjust the meter to display the value of the standard, as described in the meter user guide.

3. Add 25 mL of the more concentrated standard, 25 mL of the methanol-formaldehyde solution and 1 mL of ISA to a second 50 mL beaker and swirl the solution to mix.

4. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the more concentrated standard. Wait for a stable reading and adjust the meter to display the value of the second standard, as described in the meter user guide.

5. Record the resulting slope value. The slope should be between 25 and 30 mV when the standards are between 20 and 25 °C.

6. Add 25 mL of sample, 25 mL of the methanol-formaldehyde solution and 1 mL of ISA to a clean 50 mL beaker and swirl the solution to mix.

7. Rinse the electrode with distilled water, blot it dry and place it into the sample. The concentration of the sample will be displayed on the meter.

Note: *Other solution volumes may be used, as long as the ratio of solution to methanol-formaldehyde to ISA remains 50:50:2.*

Small Volume Direct Calibration Procedure Using a Meter with a mV Mode

Note: *See the meter user guide for more specific information.*

1. Set the meter to the mV mode.

2. Add 25 mL of the less concentrated standard, 25 mL of the methanol-formaldehyde solution and 1 mL of ISA to a 50 mL beaker and swirl the solution to mix.

3. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the less concentrated standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.

4. Add 25 mL of the more concentrated standard, 25 mL of the methanol-formaldehyde solution and 1 mL of ISA to a second 50 mL beaker and swirl the solution to mix.

5. Rinse the electrode with distilled water, blot it dry and place it into the beaker with the more concentrated standard. When a stable reading is displayed, record the mV value and corresponding standard concentration.

6. Using semi-logarithmic graph paper, prepare a calibration curve by plotting the millivolt values on the linear axis and the standard concentration values on the logarithmic axis.

7. Add 25 mL of sample, 25 mL of the methanol-formaldehyde solution and 1 mL of ISA to a clean 50 mL beaker and swirl the solution to mix.

8. Rinse the electrode with distilled water, blot it dry and place it into the beaker. When a stable reading is displayed, record the mV value.

9. Using the calibration curve prepared in step 6, determine the unknown concentration of the sample.

Note: *Other solution volumes may be used, as long as the ratio of solution to methanol-formaldehyde to ISA remains 50:50:2.*

Low-Level Calibration Technique

These procedures are for solutions that have a lead concentration of less than 1 mg/L lead (5×10^{-6} mol/L lead). For solutions low in lead but high in total ionic strength (greater than 10^{-1} mol/L), perform the same procedure by preparing a calibrating solution with a composition similar to the sample.

Accurate results require that the following conditions be met:

- Prepare at least three calibration standards that bracket the expected sample concentration.

- Always use low-level ISA for standards and samples.

- Plastic labware must be used for all low-level lead measurements.

- Adequate time must be allowed for electrode stabilization. Longer response time will be needed at low-level measurements.

- Stir all standards and samples at a uniform rate.

Low-Level Setup

1. Prepare the electrode as described in the **Electrode Preparation** section.

2. Connect the electrode to the meter. Set the meter to the mV mode.

3. Prepare the low-level ISA by pipette 20 mL of the ISA into a 100 mL volumetric flask and diluting to the mark with distilled water. Use low-level ISA for low-level measurements only.

4. Select a standard solution. Use either a 10 mg/L lead standard or a 10^{-4} mol/L lead standard.

   To prepare the 10 mg/L standard, pipette 10 mL of the 1000 mg/L standard into a 1 liter volumetric flask. Dilute to the mark with distilled water and mix the solution thoroughly.

Low-Level Calibration and Measurement

1. Add 50 mL of distilled water, 50 mL of the methanol-formaldehyde solution and 1 mL of low-level ISA to a 150 mL beaker.

2. Rinse the electrode with distilled water, blot it dry and place it into the beaker. Stir the solution thoroughly.

3. Add increments of the 10 mg/L or 10^{-4} mol/L lead standard mixed with low-level ISA to the beaker using the steps outlined in **Table 2**. Record the stable millivolt reading after each increment.

4. On semi-logarithmic paper, plot the concentration (log axis) against the millivolt potential (linear axis). Prepare a new calibration curve with fresh standards each day.

5. Add 50 mL of sample, 50 mL of the methanol-formaldehyde solution and 1 mL of low-level ISA to a clean 150 mL beaker. Rinse the electrode with distilled water, blot it dry and place the electrode into the sample.

6. Stir the solution thoroughly. When a stable reading is displayed, record the mV value.

7. Determine the sample concentration corresponding to the measured potential from the low-level calibration curve.

Table 2 – *Calibration Curve For Low-level Calibrations*
Additions of standard to 100 mL distilled water and 1 mL low-level ISA solution

Step	Pipette Size	Volume Added	Concentration (mg/L)
1	1 mL	1.0 mL	0.20
2	1 mL	1.0 mL	0.39
3	2 mL	2.0 mL	0.77
4	2 mL	2.0 mL	1.13

Step	Pipette Size	Volume Added	Concentration (mol/L)
1	1 mL	0.5 mL	1.0×10^{-6}
2	1 mL	0.5 mL	2.0×10^{-6}
3	1 mL	1.0 mL	3.9×10^{-6}
4	1 mL	1.0 mL	5.8×10^{-6}

Known Addition Technique

Known addition is a convenient technique for measuring samples in the linear range of the electrode (greater than 0.6 mg/L lead) because no calibration curve is required. It can be used to verify the results of a direct calibration or to measure the total concentration of an ion in the presence of a large excess of a complexing agent. The sample potential is measured before and after addition of a standard solution.

Accurate results require that the following conditions be met:

- Concentration should approximately double as a result of the addition.

- Sample concentration should be known to within a factor of three.

- Either no complexing agent or a large excess of the complexing agent may be present.

- The ratio of the uncomplexed ion to complexed ion must not be changed by addition of the standard.

- All samples and standards should be at the same temperature.

- With double or multiple known addition, the final addition should be 10 to 100 times the sample concentration.

- Add 2 mL of ISA and 50 mL of the methanol-formaldehyde solution to every 50 mL of sample before analysis.

- The standard addition volume should be no more than 10% of the sample volume, or the standard should be pretreated with ISA in a 50:1 ratio. See **Table 3**.

Known Addition Setup

1. Prepare the electrode as described in the **Electrode Preparation** section.

2. Connect the electrode to the meter.

3. Prepare a standard solution that will cause the lead concentration of the sample to double when added to the sample solution. Refer to **Table 3** for guidelines.

4. Determine the electrode slope by performing the procedure in the **Checking Electrode Operation (Slope)** section.

5. Rinse the electrode with distilled water.

Table 3 – *Guideline For Known Addition*

Volume of Addition	Concentration of Standard
1 mL	100 times sample concentration
5 mL	20 times sample concentration
10 mL*	10 times sample concentration

* Most convenient volume to use

Known Addition Using a Meter with a Known Addition Mode

Note: *See the meter user guide for more specific information.*

1. Set the meter to measure in the known addition mode.

2. Measure 50 mL of the sample, 50 mL of the methanol-formalde-hyde solution and 2 mL of ISA and pour the solutions into a 150 mL beaker. Rinse the electrode with distilled water and place it into the sample solution. Stir the solution thoroughly.

3. When a stable reading is displayed, set the meter as described in the meter user guide, if required.

4. Pipette the appropriate amount of the standard solution into the beaker. Stir the solution thoroughly.

5. When a stable reading is displayed, record the sample concentration.

Known Addition Using a Meter with a Millivolt Mode

1. Set the meter to the relative millivolt mode. If a relative millivolt mode is not available, use the millivolt mode.

2. Measure 50 mL of the sample, 50 mL of the methanol-formaldehyde solution and 2 mL of ISA and pour the solutions into a 150 mL beaker. Stir the solution thoroughly.

3. Rinse the electrode with distilled water, blot it dry and place the electrode into the beaker. When a stable reading is displayed, record the actual mV value.

4. Pipette the appropriate amount of standard solution into the beaker. Stir the solution thoroughly.

5. When a stable reading is displayed, record the mV value. Subtract the first reading from the second reading to calculate ΔE.

6. Use **Table 5** to find the Q value that corresponds to the change in potential, ΔE. To determine the original sample concentration, multiply Q by the concentration of the added standard:

$$C_{sample} = Q * C_{standard}$$

$C_{standard}$ = standard concentration
C_{sample} = sample concentration
Q = value from **Table 5**

The table of Q values is calculated for a 10% volume change. The equation for the calculation of Q for different slopes and volume changes is given below.

$$Q = (p * r) / \{[(1 + p) * 10^{\Delta E/S}] - 1\}$$

Q = value from **Table 5**
ΔE = $E_2 - E_1$
S = slope of the electrode
p = volume of standard / volume of sample and ISA
r = volume of sample and ISA / volume of sample

Calculating Known Addition for Samples using Excel Spreadsheets

If it is more convenient, a simple spreadsheet can be set up to calculate the known addition results, using any ratio of sample to addition. A typical worksheet is shown in **Table 4**. The numbers shown are examples, but the formulas and their locations should be copied exactly.

Table 4 – *Known Addition Calculations using Excel Spreadsheets*

A	B	C
1		Enter Value
2	Volume of sample and ISA (mL)	102
3	Volume of addition (mL)	10
4	Concentration of addition	10
5	Volume of sample	50
6	Initial mV reading	45.3
7	Final mV reading	63.7
8	Electrode slope	28.2
9		
10		Derived Values
11	Delta E	=C7 - C6
12	Solution volume ratio	=C3/C2
13	Antilog term	=10^ (C11/C8)
14	Sample volume ratio	=C2/C5
15	Q term	=C12*C14/ (((1+C12)*C13)-1)
16	Calculated initial concentration in same units as addition	=C15*C4

Table 5 – *Q Values for a 10% volume change, slopes*
(in column heading) are in units of mV/decade

ΔE	Q Concentration Ratio			
	28.6	**29.1**	**29.6**	**30.1**
5.0	0.3114	0.3170	0.3225	0.3281
5.2	0.2990	0.3044	0.3098	0.3152
5.4	0.2874	0.2927	0.2979	0.3032
5.6	0.2764	0.2816	0.2867	0.2919
5.8	0.2661	0.2712	0.2762	0.2812
6.0	0.2564	0.2614	0.2663	0.2711
6.2	0.2473	0.2521	0.2569	0.2616
6.4	0.2386	0.2433	0.2480	0.2527
6.6	0.2304	0.2350	0.2396	0.2441
6.8	0.2226	0.2271	0.2316	0.2361
7.0	0.2152	0.2196	0.2240	0.2284
7.2	0.2082	0.2125	0.2168	0.2211
7.4	0.2015	0.2058	0.2099	0.2141
7.6	0.1952	0.1993	0.2034	0.2075
7.8	0.1891	0.1932	0.1972	0.2012
8.0	0.1833	0.1873	0.1912	0.1951
8.2	0.1778	0.1817	0.1855	0.1894
8.4	0.1725	0.1763	0.1801	0.1839
8.6	0.1674	0.1712	0.1749	0.1786
8.8	0.1626	0.1662	0.1699	0.1735
9.0	0.1579	0.1615	0.1651	0.1687
9.2	0.1535	0.1570	0.1605	0.1640
9.4	0.1492	0.1527	0.1561	0.1595
9.6	0.1451	0.1485	0.1519	0.1552
9.8	0.1411	0.1445	0.1478	0.1511
10.0	0.1373	0.1406	0.1439	0.1471
10.2	0.1337	0.1369	0.1401	0.1433
10.4	0.1302	0.1333	0.1364	0.1396
10.6	0.1268	0.1298	0.1329	0.1360
10.8	0.1235	0.1265	0.1296	0.1326
11.0	0.1203	0.1233	0.1263	0.1293
11.2	0.1173	0.1202	0.1231	0.1261
11.4	0.1143	0.1172	0.1201	0.1230
11.6	0.1115	0.1143	0.1172	0.1200
11.8	0.1087	0.1115	0.1143	0.1171
12.0	0.1061	0.1088	0.1116	0.1143
12.2	0.1035	0.1062	0.1089	0.1116
12.4	0.1010	0.1037	0.1063	0.1090
12.6	0.0986	0.1012	0.1038	0.1064
12.8	0.0963	0.0988	0.1014	0.1040
13.0	0.0940	0.0965	0.0991	0.1016
13.2	0.0918	0.0943	0.0968	0.0993
13.4	0.0897	0.0922	0.0946	0.0971
13.6	0.0876	0.0901	0.0925	0.0949
13.8	0.0856	0.0880	0.0904	0.0928
14.0	0.0837	0.0860	0.0884	0.0907
14.2	0.0818	0.0841	0.0864	0.0887
14.4	0.0800	0.0823	0.0845	0.0868
14.6	0.0782	0.0804	0.0827	0.0849
14.8	0.0765	0.0787	0.0809	0.0831

ΔE	Q Concentration Ratio			
	28.6	**29.1**	**29.6**	**30.1**
15.0	0.0748	0.0770	0.0792	0.0813
15.5	0.0708	0.0729	0.0750	0.0771
16.0	0.0671	0.0691	0.0711	0.0732
16.5	0.0636	0.0655	0.0675	0.0695
17.0	0.0603	0.0622	0.0641	0.0660
17.5	0.0573	0.0591	0.0609	0.0627
18.0	0.0544	0.0561	0.0579	0.0597
18.5	0.0517	0.0534	0.0551	0.0568
19.0	0.0491	0.0508	0.0524	0.0541
19.5	0.0468	0.0483	0.0499	0.0515
20.0	0.0445	0.0460	0.0476	0.0491
20.5	0.0424	0.0439	0.0454	0.0469
21.0	0.0404	0.0418	0.0432	0.0447
21.5	0.0385	0.0399	0.0413	0.0427
22.0	0.0367	0.0380	0.0394	0.0407
22.5	0.0350	0.0363	0.0376	0.0389
23.0	0.0334	0.0346	0.0359	0.0372
23.5	0.0318	0.0331	0.0343	0.0355
24.0	0.0304	0.0316	0.0328	0.0340
24.5	0.0290	0.0302	0.0313	0.0325
25.0	0.0277	0.0288	0.0300	0.0311
25.5	0.0265	0.0276	0.0286	0.0297
26.0	0.0253	0.0263	0.0274	0.0285
26.5	0.0242	0.0252	0.0262	0.0273
27.0	0.0231	0.0241	0.0251	0.0261
27.5	0.0221	0.0231	0.0240	0.0250
28.0	0.0211	0.0221	0.0230	0.0239
28.5	0.0202	0.0211	0.0220	0.0229
29.0	0.0193	0.0202	0.0211	0.0220
29.5	0.0185	0.0194	0.0202	0.0211
30.0	0.0177	0.0185	0.0194	0.0202
30.5	0.0170	0.0178	0.0186	0.0194
31.0	0.0162	0.0170	0.0178	0.0186
31.5	0.0155	0.0163	0.0171	0.0178
32.0	0.0149	0.0156	0.0163	0.0171
32.5	0.0143	0.0150	0.0157	0.0164
33.0	0.0137	0.0143	0.0150	0.0157
33.5	0.0131	0.0137	0.0144	0.0151
34.0	0.0125	0.0132	0.0138	0.0145
34.5	0.0120	0.0126	0.0133	0.0139
35.0	0.0115	0.0121	0.0127	0.0134
35.5	0.0110	0.0116	0.0122	0.0128
36.0	0.0106	0.0111	0.0117	0.0123
36.5	0.0101	0.0107	0.0112	0.0118
37.0	0.0097	0.0102	0.0108	0.0114
37.5	0.0093	0.0098	0.0104	0.0109
38.0	0.0089	0.0094	0.0099	0.0105
38.5	0.0086	0.0090	0.0095	0.0101
39.0	0.0082	0.0087	0.0092	0.0097
39.5	0.0079	0.0083	0.0088	0.0093

ΔE	Q Concentration Ratio			
	28.6	29.1	29.6	30.1
39.0	0.0082	0.0087	0.0092	0.0097
39.5	0.0079	0.0083	0.0088	0.0093
40.0	0.0075	0.0080	0.0085	0.0089
40.5	0.0072	0.0077	0.0081	0.0086
41.0	0.0069	0.0074	0.0078	0.0082
41.5	0.0067	0.0071	0.0075	0.0079
42.0	0.0064	0.0068	0.0072	0.0076
42.5	0.0061	0.0065	0.0069	0.0073
43.0	0.0059	0.0063	0.0066	0.0070
43.5	0.0056	0.0060	0.0064	0.0068
44.0	0.0054	0.0058	0.0061	0.0065
44.5	0.0052	0.0055	0.0059	0.0062
45.0	0.0050	0.0053	0.0057	0.0060
45.5	0.0048	0.0051	0.0054	0.0058
46.0	0.0046	0.0049	0.0052	0.0055
46.5	0.0044	0.0047	0.0050	0.0053
47.0	0.0042	0.0045	0.0048	0.0051
47.5	0.0041	0.0043	0.0046	0.0049
48.0	0.0039	0.0042	0.0044	0.0047
48.5	0.0037	0.0040	0.0043	0.0046
49.0	0.0036	0.0038	0.0041	0.0044
49.5	0.0034	0.0037	0.0040	0.0042
50.0	0.0033	0.0035	0.0038	0.0041
50.5	0.0032	0.0034	0.0036	0.0039
51.0	0.0030	0.0033	0.0035	0.0038
51.5	0.0029	0.0031	0.0034	0.0036
52.0	0.0028	0.0030	0.0032	0.0035
52.5	0.0027	0.0029	0.0031	0.0033
53.0	0.0026	0.0028	0.0030	0.0032
53.5	0.0025	0.0027	0.0029	0.0031
54.0	0.0024	0.0026	0.0028	0.0030
54.5	0.0023	0.0025	0.0027	0.0029
55.0	0.0022	0.0024	0.0026	0.0027
55.5	0.0021	0.0023	0.0025	0.0026
56.0	0.0020	0.0022	0.0024	0.0025
56.5	0.0019	0.0021	0.0023	0.0024
57.0	0.0019	0.0020	0.0022	0.0024
57.5	0.0018	0.0019	0.0021	0.0023
58.0	0.0017	0.0019	0.0020	0.0022
58.5	0.0017	0.0018	0.0019	0.0021
59.0	0.0016	0.0017	0.0019	0.0020
59.5	0.0015	0.0017	0.0018	0.0019
60.0	0.0015	0.0016	0.0017	0.0019

Lead Titration Technique

The lead electrode makes a highly sensitive endpoint detector for titration with EDTA of lead samples. With careful technique, titrations accurate to $\pm$ 0.3% of the total lead ion concentration of the sample can be performed.

EDTA complexes other cations besides lead ion. Interferences from alkaline earths and other ions, whose EDTA complexes are stable only at certain pH values, can be eliminated by adjusting the sample pH to the proper range before performing the titration. In many cases, other interferences can be eliminated by a suitable choice of sample pH and the addition of masking agents to the sample solution. A comprehensive list of methods is given in the Handbook of Analytical Chemistry, L. Meites, (ed.) McGraw Hill Book Co., New York, (1st edit.), pp. 3-76, 3-225.

Lead Titration Setup

1. Prepare the electrode as described in the **Electrode Preparation** section.

2. Connect the electrode to the mV sensor input of the titrator.

3. Prepare a 0.01 mol/L EDTA stock solution by adding 3.772 g of reagent-grade Na_4EDTA to a 1 liter volumetric flask. Add 50 mL of the methanol-formaldehyde solution and swirl the solution to dissolve the solids. Dilute to the mark with distilled water and mix the solution thoroughly.

Lead Titration Procedure

1. Place 50 mL of sample and 50 mL of the methanol-formaldehyde solution into a 150 mL beaker. Place the electrode in the sample and stir the solution thoroughly.

2. Perform an equivalence point titration using a standard EQP (equivalence point) titration method template available in the Tx Excellence and G20 Compact titrators. The EQP of the titration is the point of the greatest slope (inflection point). See **Figure 3**.

3. The concentration of the sample solution before dilution is calculated using the following equation:

R (mol/L) = Q*C/m

where
Q = VEQ*c*TITER
VEQ = Volume at the EQP
c = nominal concentration of the EDTA titrant
$TITER$ = Titer of the EDTA titrant
C = $1/z$, $z=1$ (equivalent number of the EDTA titrant)
m = volume of the sample solution

Sulfate Titration Technique

The lead electrode makes a sensitive endpoint detector for the titration of sulfate ions with lead perchlorate. The method is simpler and less time consuming than sulfate determinations by the gravimetric or turbidimetric methods, yet offers the same or greater precision in solutions as dilute as 10^{-4} mol/L (10 mg/L) sulfate.

The following ions are known to interfere with the titration if present in amounts (on a molar basis) in excess of the following:

NO_3^- $> 50 \times SO_4^{2-}$
HCO_3^- $> 100 \times SO_4^{2-}$ at pH 4
Cl^- $> 50 \times SO_4^{2-}$
Phosphate and calcium must be absent

A 0.1 mol/L $Pb(ClO_4)_2$ standard solution is used as titrant and can be diluted to the proper range for the expected concentration in the sample. As a general rule, the concentration of lead perchlorate should be about ten times greater than the expected sulfate ion concentration of the sample. With a sodium sulfate solution the titrant can be standardized.

The samples are diluted 1:1 with isopropanol before performing the titration.

The method outlined below is suitable for samples containing about 10^{-3} mol/L sulfate ion. For lower sulfate concentrations, the titratnt should be diluted correspondingly.

1. Prepare a 0.01 mol/L $Pb(ClO_4)_2$ titrant by adding 100 mL of the 0.1 mol/L $Pb(ClO_4)_2$ standard solution to a 1 liter volumetric flask and dilute to mark with distilled water.

2. Place 50 mL of sample and 50 mL of isopropanol into a 150 mL beaker. Add 1 mL of 1 mol/L $HClO_4$ to acidify the sample. Place the electrode in the sample and stir the solution thoroughly.

3. Perform an equivalence point titration using a standard EQP (equivalence point) titration method template available in the Tx Excellence and G20 Compact titrators. The EQP of the titration is the point of the greatest slope (inflection point). See **Figure 3**.

4. The concentration of the sample solution before dilution is calculated using the following equation:

R (mol/L) = Q*C/m

where
Q   = VEQ*c*TITER
VEQ   = Volume at the EQP
c   = nominal concentration of the EDTA titrant
TITER = Titer of the EDTA titrant
C   = 1/z, z=1 (equivalent number of the EDTA titrant)
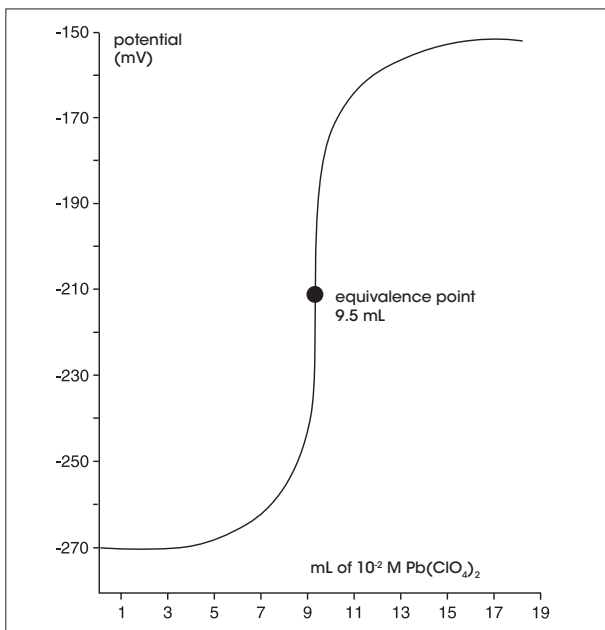m   = volume of the sample solution

Figure 3 – *Typical Sulfate Titration*

5. Electrode Characteristics

Electrode Response

The electrode potential plotted against concentration on semi-logarithmic paper results in a straight line with a slope of about 25 to 30 mV per decade change in concentration.

The time response of the electrode (the time required to reach 99% of the stable potential reading) varies from several seconds in concentrated solutions to several minutes near the limit of detection.

Limits of Detection

In neutral solutions, lead ion concentrations can be measured down to 10^{-6} mol/L (0.2 mg/L). Extreme care must be taken in making determinations below 10^{-5} mol/L (0.6 mg/L) to avoid sample contamination or adsorption of lead ion on container walls.

Reproducibility

Reproducibility is limited by factors such as temperature fluctuations, drift and noise. Within the operating range of the electrode, reproducibility is independent of concentration. With hourly calibrations, direct electrode measurements reproducible to $\pm$ 4 % can be obtained.

Temperature Effects

Since electrode potentials are affected by changes in temperature, samples and standard solutions should be within $\pm$ 1 °C ($\pm$ 2 °F) of each other. At the 10^{-3} mol/L level, a 1 °C difference in temperature results in errors greater than 4 %. The absolute potential of the reference electrode changes slowly with temperature because of the solubility equilibria on which the electrode depends. The slope of the electrode also varies with temperature, as indicated by the factor S in the Nernst equation. Theoretical values of the slope at different temperatures are given in **Table 6**. If the temperature changes, the meter and electrode should be recalibrated.

The electrode can be used at temperatures from 0 to 80 °C, provided that temperature equilibrium has occurred. For use at temperatures substantially different from room temperature, calibration standards should be at the same temperature as samples. The electrode must be used only intermittently at solution temperatures above 80 °C.

Table 6 – *Theoretical Slope vs. Temperature Values*

Temperature (°C)	Slope (mV)
0	27.1
10	28.1
20	29.1
25	29.6
30	30.1
40	31.1
50	32.1

The Ion Electrolyte B Reference filling solution that is included with the electrode will minimize junction potentials and provide optimum temperature and time response.

Interferences

The electrode does not respond to anions or to most cations. Copper, mercury and silver ions poison the lead electrode sensing element and must be absent from the sample solution. Ferric and cadmium ions also affect the membrane surface, if the level of ferric or cadmium ions exceed the level of lead ions present in the sample. If the level of ferric or cadmium ions are less than the level of lead ions, no interference will occur. Ferric ions can be eliminated by adjusting the sample pH to above 4 with sodium hydroxide.

If the electrode is exposed to high levels of interfering ions, it may become unstable and sluggish in response. If this happens, restore normal electrode performance by polishing it. Refer to the **Electrode Maintenance** section.

Complexation

Lead ion forms complexes with many common chemical species in solution, such as acetate, citrate, thiosulfate, pyrophosphate, and hydroxide ions. The electrode does not respond to lead ions that are bound to complexing agents, but it will respond to the free ions remaining in the solution. In some instances, the complexes can be broken by suitable pH adjustment. See **Figure 4**. If the level of complexing agent is known, measurements for total concentration can be made by using standards with the same background. If a large excess of complexing agent is present, the known addition technique can be used to measure total lead concentration.

pH Effects

The electrode response to lead ions in solutions at various pH values is shown in **Figure 4**. Although the electrode can be used over a wide pH range, hydrogen ions interfere with measurements of low-levels of lead ions. The edge of the shaded area to the left in **Figure 4** indicates the minimum pH at which low-level lead measurements can be made without hydrogen ion interference.
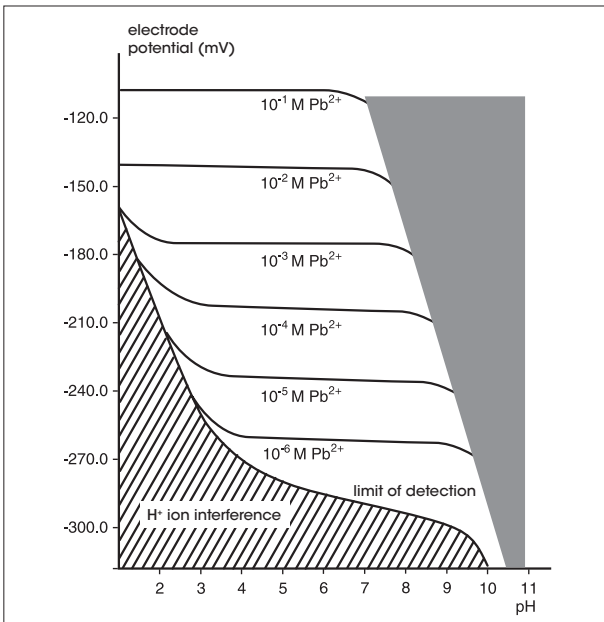

Figure 4 – Electrode Potential Behavior vs. Solution pH in Pure $Pb(ClO_4)_2$ Solutions at 25 °C

Theory of Operation

The lead electrode consists of a sensing element bonded into an epoxy body. When the sensing element is in contact with a solution containing lead ions, an electrode potential develops across the sensing element. This potential, which depends on the level of free lead ion in solution, is measured against a constant reference potential with a digital pH/mV meter or ISE (concentration) meter. The measured potential corresponding to the level of lead ion in solution is described by the Nernst equation.

$$E = E_o + S * \log (A)$$

E = measured electrode potential
E_o = reference potential (a constant)
A = lead ion activity level in solution
S = electrode slope (about 28 mV per decade)
S = (2.3 R T) / nF
R and F are constants, T = temperature in kelvin and
n = ionic charge

The level of lead ions, A, is the activity or "effective concentration" of free lead ions in solution. The lead ion activity is related to free lead ion concentration, C_f, by the activity coefficient, γ.

$$A = γ * C_f$$

Ionic activity coefficients are variable and largely depend on total ionic strength. The ionic strength of a solution is determined by all of the ions present. It is calculated by multiplying the concentration of each individual ion by the square of its charge, adding all these values up and then dividing by two.

Ionic strength = $1/2 \sum (C_i Z_i^2)$

C_i = concentration of ion i
Z_i = charge of ion i
$\sum$ symbolizes the sum of all the types of ions in solution

If background ionic strength is high and constant relative to the sensed ion concentration, the activity coefficient is constant and activity is directly proportional to the concentration. Ionic strength adjustor (ISA) is added to all lead standards and samples so that the background ionic strength is high and constant relative to variable concentrations of lead. For lead, the recommended ISA is 5 mol/L $NaClO_4$. Other solutions can be used as long as they do not contain ions that would interfere with the electrode response to lead.

If samples have a high ionic strength (above 0.1 mol/L), standards should be prepared with a composition similar to the samples.

Reference electrode conditions must also be considered. Liquid junction potentials arise any time when two solutions of different composition are brought into contact. The potential results from the interdiffusion of ions in the two solutions. Since ions diffuse at different rates, the electrode charge will be carried unequally across the solution boundary resulting in a potential difference between the two solutions. In making electrode measurements, it is important that this potential is the same when the reference is in the standardizing solution as well as in the sample solution; otherwise, the change in liquid junction potential will appear as an error in the measured specific ion electrode potential.

The most important variable that analysts have under their control is the composition of the liquid junction filling solution. The filling solution should be equitransferent. That is, the speed with which the positive and negative ions in the filling solution diffuse into the sample should be nearly as equal as possible. If the rate at which positive and negative charge is carried into the sample solution is equal, then no junction potential can result.
perfectION™ reference filling solutions are specifically designed to meet all reference electrode conditions.

6. Troubleshooting

Follow a systematic procedure to isolate the problem. The measuring system can be divided into four components for ease in troubleshooting: meter, electrode, sample/application and technique.

Meter/Titrator

The meter/titrator is the easiest component to eliminate as a possible cause of error. Consult the meter/titrator user guide for directions.

Electrode

1. Rinse the electrode thoroughly with distilled water.

2. Verify the electrode performance by performing the procedure in the **Checking Electrode Operation (Slope)** section.

3. If the electrode fails this procedure, review the **Measuring Hints** section. Clean the electrode thoroughly as directed in the **Electrode Maintenance** section. Drain and refill the electrode with fresh filling solution.

4. Repeat the procedure in the **Checking Electrode Operation (Slope)** section.

5. If the electrode passes the procedure, but measurement problems persist, the sample may contain interferences or complexing agents, or the technique may be in error.

6. Before replacing a faulty electrode, review this user guide and be sure to thoroughly clean the electrode; correctly prepare the electrode; use the proper filling solution, ISA, and standards; correctly measure the samples and review the **Troubleshooting Checklist** section.

Sample/Application

The quality of results depends greatly upon the quality of the standards. Always prepare fresh standards when problems arise, it could save hours of frustrating troubleshooting! Errors may result from contamination of prepared standards, accuracy of dilution, quality of distilled water, or a mathematical error in calculating the concentrations.

The best method for preparation of standards is serial dilution. Refer to the **Serial Dilution** section. The electrode and meter may operate with standards, but not with the sample. In this case, check the sample composition for interferences, incompatibilities or temperature effects. Refer to the **Sample Requirements, Temperature Effects, Interferences** and **pH Effects** sections.

Technique

If trouble persists, review operating procedures. Review calibration and measurement sections to be sure proper technique has been followed. Verify that the expected concentration of the ion of interest is within the limit of detection of the electrode.

Check the method of analysis for compatibility with your sample. **Direct measurement** may not always be the method of choice. If a large amount of complexing agents are present, **Known Addition** may be the best method. If working with low-level samples, follow the procedure in the **Low-level Calibration** section.

Troubleshooting Checklist

- No reference filling solution added – Fill the electrode with filling solution up to the fill hole. Refer to the **Electrode Preparation** section for details.

- Incorrect reference filling solution used – Refer to the **Electrode Preparation** section to verify the correct electrode filling solution.

- Electrode junction is dry – Push down on the electrode cap to allow a few drops of filling solution to drain out of the electrode.

- Electrode is clogged or dirty – Refer to the **Electrode Maintenance** section for cleaning instructions.

- Sensing element is dirty or etched – Refer to the **Electrode Maintenance** section for cleaning instructions.

- Standards are contaminated or made incorrectly – Prepare fresh standards. Refer to the **Measurement Hints** and **Analytical Techniques** sections.

- ISA not used or incorrect ISA used – ISA must be added to all standards and samples. Refer to the **Required Equipment** section for information on the ISA.

- Samples and standards at different temperatures – Allow solutions to reach the same temperature.

- Air bubble on sensing element – Remove air bubble by reimmersing the electrode in solution.

- Electrode not properly connected to meter/titrator – Unplug and reconnect the electrode to the meter/titrator.

- Meter/Titrator or stir plate not properly grounded – Check the meter/titrator and stir plate for proper grounding.

- Static electricity present – Wipe plastic parts on the meter/titrator with a detergent solution.

- Defective meter/titrator – Check the meter/titrator performance. See the meter/titrator user guide.

7. Ordering Information

Parts	Order No.
Combined Lead electrode with BNC connector perfectION™ comb Pb^{2+}:	**51344730**
Combined Lead electrode with Lemo connector perfectION™ comb Pb^{2+} Lemo:	**51344830**
Ion Electrolyte B:	**51344751**
Lead Standard Solution 1000 mg/L:	**51344780**
Removable cone:	**00022986**

8. Electrode Specifications

Membrane type

solid state

Concentration Range

10^{-6} mol/L to 0.1 mol/L
0.2 mg/L to 20'700 mg/L

pH Range

pH 4 to 7

Temperature Range

0 to 80 °C continuous use

Electrode Resistance

Less than 1 MΩ

Reproducibility

± 4%

Minimum Sample Size

5 mL in a 50 mL beaker

Size

Body Diameter: 13 mm
Cap Diameter: 16 mm
Cable Length: 1.2 m

* Specifications are subject to change without notice

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For more information