SemiChem APM 200 with H_2O_2 in CMP Process

Application guide

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INTRODUCTION

Our SemiChem APM 200 (advanced process monitor) Application guide provides step-by-step instructions for properly configuring the SemiChem APM 200 for the CMP application for measuring hydrogen peroxide. This guide is designed for trained and qualified personnel. It is complimentary to the SemiChem APM 200 Installation and Operating Manual, not a replacement.

A.1 PRECAUTIONS

Be sure any personnel working on the SemiChem APM 200 have read and understand the Installation and Operating Manual. There are inherent hazards to working with the SemiChem APM 200.

WARNING: Service of the SemiChem APM 200 may cause exposure to hazardous conditions including chemical, electrical, and mechanical dangers. Personnel should be properly trained in all areas before attempting to service or troubleshoot the analyzer.

WARNING: Electrical Precaution: High voltage electronics (110/220 VAC) are located throughout the electronics module. Printed circuit boards are powered with 24 VDC. All service should be performed by a qualified electrician/electrical technician.

WARNING: Chemical Precaution: Corrosive or poisonous liquids. Some reagents may be corrosive to the eyes, skin, and respiratory tract and may be hazardous if ingested. Wear chemical resistant gloves, face shield, and apron when changing the burette O-ring. See MSDS for further health hazard information. To minimize chemical exposure, purge the burette with air by removing the reagent intake line from the reagent bottle, then flush the appropriate burette. This will remove most of the reagent from the burette assembly. If replacing the burette glass, consult with on-site environmental personnel for proper disposal of remaining reagents. If replacing a burette O-ring, keep the burette glass assembly within the enclosure for proper exhaust of fumes.

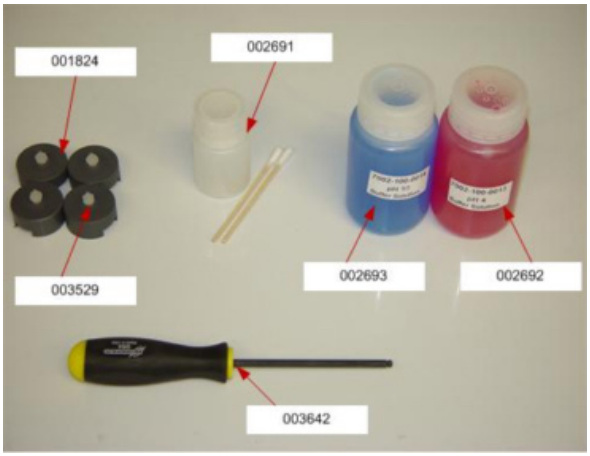
It is the responsibility of the user to fully understand the use, application, and hazards associated with the SemiChem APM 200. Refer to the SemiChem APM 200 Installation and Operating Manual for more information.

A.2 REASSEMBLY

A.2.1 START-UP KIT

To complete the start-up of the SemiChem APM 200, you will need:

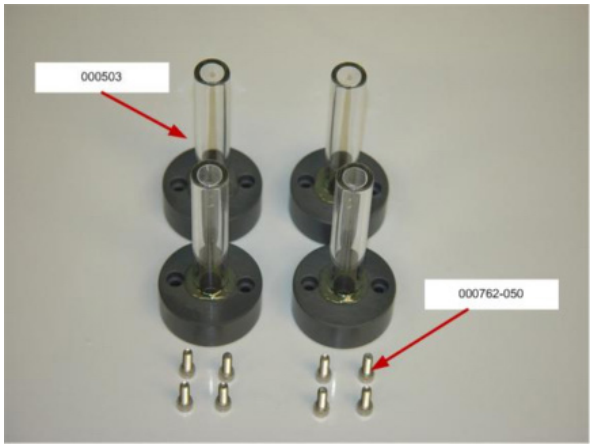
1. 1 ea. 9/64" ball driver (003642).
2. 4 ea. tube weights and barbs (001824 and 003529).
3. Silicone paste kit (002691).



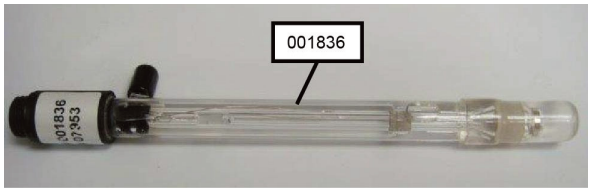
Start-up kit items.

In addition, you will need:

1. Glass burettes (000503) – the number of glass burette assemblies will vary depending on the application.
2. 8 – 32 × ½" SHCS (000762-050).



3. The sensor, electrode, ORP combination (001836).



A.2.2 GLASS BURETTES

Burette installation:

1. Using the silicone paste kit, apply a small amount of silicone to each burette piston's O-ring.

NOTE: Silicone paste is not needed on jacketed O-rings.

2. Install the glass burette into the piston and guideposts using a slight twist motion.
3. Secure the burette glass assembly with the 8 – 32 × ½" SHCS and 9⁄64" ball driver.
4. Repeat for burette 2.

Installed burettes.

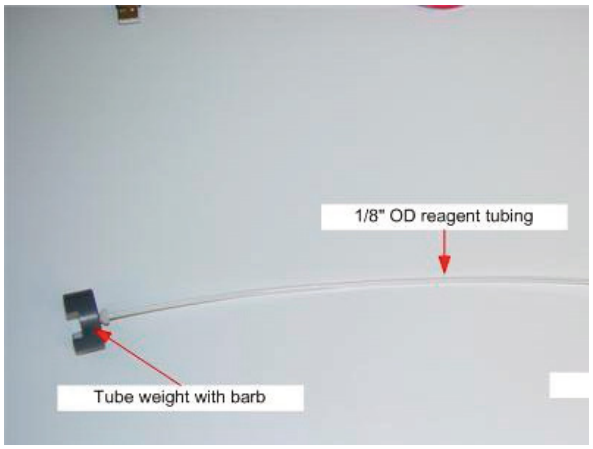
A.2.3 REAGENTS AND TUBING

Reagents installation:

1. Within the reagent storage, locate and uncoil the reagent tubing.

NOTE: That each reagent tube is labeled with its corresponding burette.

2. Insert the barb into the tube weight.
3. Remove the reagent cap from the reagent bottles and slide the reagent 1 tube through the cap.
4. Install the barb/tube weight combination onto the end of the reagent tube.
5. Repeat for reagent 2.



Installed tube weight.

6. Fill each reagent bottle with the reagents:
   - Reagent bottle 1: 4.0 N sulfuric acid
   - Reagent bottle 2: 0.1 N ceric sulfate in 1.0 N sulfuric acid
7. Carefully insert the tube weight/tube/cap combination into corresponding reagent bottle. Be sure the tube weight falls to the bottom and sits flat. Secure the cap. Place reagent bottles into the corresponding slots within the reagent storage area.

NOTE: To follow the recipe parameters it is important that the reagents are correctly assigned to the proper burettes.

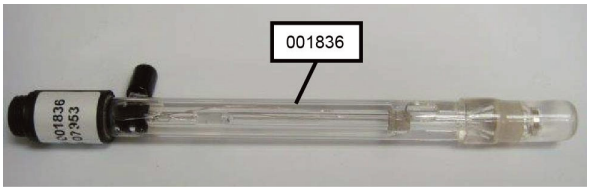
A.2.4 ORP SENSORS

1. Remove any caps and covers on the ORP sensor.

NOTE: There may be residual white crystals. These crystals are saturated potassium chloride solution. They are normal and can simply be wiped away.

2. Remove the electrode retaining nut(s) from the measuring cell. Remove the 3 mm x 11 mm O-ring from the electrode port. You can choose to use any of the open ports.
3. Slide the electrode into the retaining nut. Be careful, the electrode is glass and can break. Slide the O-ring onto the electrode shaft.
4. Insert the electrode/retaining nut/O-ring combination into the measuring cell.
5. Locate the electrode cable labeled "Input 1". connect the ORP electrode and the electrode cable.

NOTE: To follow the recipe parameters it is important that the electrodes are connected to the proper inputs.



ORP Electrode with protective cap.

A.3 PREPARING TO RUN

A.3.1 FACILITIES

At this point, the SemiChem APM 200 should be fully installed and reassembled. All utilities can now be energized.

1. Close all doors and be sure the exhaust is connected and active.
2. Power up the SemiChem APM 200. The SemiChem APM 200 information system will turn on. The MAIN screen will be available shortly.

NOTE: If the system has been provided with safety interlocks, the screen will have a RED background indicating the safety interlocks require a reset. Press the "GREEN" button below the EPO button to initiate the safety circuits. Once pressed, and all safety interlocks have been satisfied, the system is now ready.

3. Turn on CDA, water, and sample. Visually verify there are no leaks.
4. From the MAIN screen, press "Service," then press "Recipes."
5. From the service screen, select the recipe "Flush Cell." Press "Run Now."
6. Verify water sprays into the cell from the top. It will spray into the cell with the drain open for about 20 seconds, the drain closes and the water level will reach the level sensor and stop the spray.

Service > Recipes.

A.3.2 FLUSH BURETTES

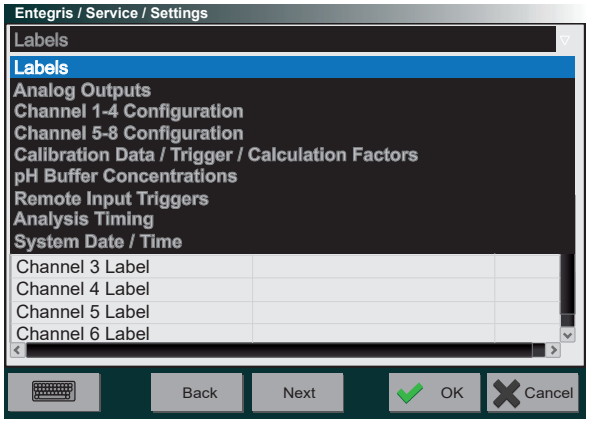
Flushing the burettes with the appropriate reagents:

1. From Recipes, choose "Refill_Burette_1." Press "Run Now." The burette will proceed to cycle up and down three times. During which, it will displace the air in the burette with the reagent. Once complete, the reagent tubing and glass burette will be completely filled with reagent. Repeat for burettes 2.
2. Carefully inspect the burette glass and all tubing to verify that all the air and bubbles have been purged from the burette system. If not, repeat the above step. For proper performance, the burette system must be completely free from air bubbles.
3. Select "Flush_cell." Press "Run Now."

A.3.3 SETTINGS

The SemiChem APM 200's operation and behavior within a process environment can be configured to suit specific user needs. The settings allow the SemiChem APM 200 to fit into the user's process control systems. There are eight setting screens. Use the following to program the settings. Refer to the SemiChem APM 200 Installation and Operating Manual for more information on each setting.

From the MAIN screen, press "Service," then press "Settings."



Service/Settings.

Labels: The "Labels" menu is used for labeling the SemiChem APM 200. As an example:

– Burette 1 Label: 5.0 N Sulfuric Acid
– Burette 2 Label: 0.1 N Ceric Sulfate
– Sample 1 Label: Peroxide Distribution Tank
– Sample 2 Label: Peroxide Day Tank
– Output 1 Label: Peroxide Distribution Tank
– Output 2 Label: Peroxide Day Tank

Analog Outputs: Sets the range for each output.

NOTE: The SemiChem APM 200 utilizes 4 – 20 mA analog signals. 4 mA will be 0, 20 mA will correspond to the high range.

Relays: Sets the behavior and threshold for each alarm.

Calibration Data/Trigger/Calculation Factors:

1. Trigger. If set to local, the SemiChem APM 200 will run independent and will run whichever recipe is set as default based on the internal analysis frequency. If set to remote, the SemiChem APM 200 will run with PLC handshaking. In addition, the user can select which recipe to run when using the recipe select feature. The SemiChem APM 200. If set to computer, the SemiChem APM 200 will respond to commands from the RS232. Refer to the SemiChem APM 200 Installation and Operating Manual, section 4.2.
2. Calculation Factors: These are set as part of the process calibration.

pH Buffer Concentration: When using an ORP electrode, these are not used.

Remote Trigger Inputs: Each remote trigger input can be tied to a recipe. Therefore, when either a PLC (remote mode) or a PC (computer) send the input (as per the SemiChem APM 200 Installation and Operating Manual, section 4.2), the SemiChem APM 200 will run the recipe tied to the input. For example, Remote Recipe 0 may be the "Hydrogen Peroxide Sample 1" recipe, and Remote Recipe 1 may be the "Hydrogen Peroxide in Sample 2" recipe. In this case, the user can select which analysis to perform depending on the control needs of the process.

Analysis Timing: Sets the analysis frequency and sensor equilibration time.

NOTE: The SAMPLE TRANSFER VOLUME must be set to whatever the cell volume is in milliliters. This is typically set to 200.

System Date and Time: Sets the date and time.

A.3.4 RECIPES AND ADJUSTMENTS

A.3.4.1 Recipes

The SemiChem APM 200 is provided with a variety of recipes as a set of application defaults. These recipes should be considered "starting points" and will most likely need to be adjusted to meet individual needs.

The recipes available for the Hydrogen Peroxide in CMP are:

Hydrogen peroxide sample 1: This recipe will measure hydrogen peroxide (H_2O_2) on sample 1 only.

Hydrogen peroxide sample 2: This recipe will measure H_2O_2 on sample 2 only.

These recipes can be edited and adjusted. Additionally, recipes can be imported via the USB.

Text notations of the recipe assumptions are within each of the recipes. These assumptions are based on the SemiChem APM 200 configuration the sample loop volume, the burette assignments, the electrode input assignments, the reagent concentrations, and the process concentration range. Generally, it is assumed that:

1. Sample loop is 1.0 mL. This is the factory default.
2. Burettes and electrode are assigned as per A2.3 (page 5).
3. Ceric sulfate is 0.1 N.
4. The expected H_2O_2 expected concentration is 1-2%. If the expected concentrations vary, the system configuration and reagent concentration may need to be adjusted. Please contact Entegris for more information.

A.3.4.2 Adjustments

Once the desired recipe is selected, there are a number of parameters that can be adjusted to optimize the performance of the SemiChem APM 200. As an example, the recipe shown below is for HF and H_2O_2 for Sample 1.

```
#H2O2 for Sample 1 Only

#Loop 1 is 1.0 mL
#Reagent 1: 4.0 N Sulfuric Acid
#Reagent 2: 0.1 N Ceric Sulfate in 1.0 N H2SO4
#Sample: 1% Hydrogen Peroxide
#Sensor #1: ORP
function main

call HOME MOTORS motor1=yes motor2=yes motor3=no motor4=no
call BURETTE number=1 action=setvol volume=0.00 valve=EV01
call BURETTE number=2 action=setvol volume=0.00 valve=EV02
#Hydrogen Peroxide Analysis
call EMPTY_APM200_CELLA time=15
call FLUSH_APM200_CELLA time=25
call EMPTY_APM200_CELLA time=15
call SAMPLE time=10 valve=AV01
call CONDITION number=1 volume=2 valve=EV01
call TRANSFER_APM200_CELLA time=6000
call CONTROLIO io=MIXERA value=1 time=0
call TITRATE cell=1 number=2 channel_id=1 sensor_ch=1 endpoint=850 window=100 end=1050 sensor=mV
algorithm=seek_set direction=up_1 min_inc=0.03 max_inc=0.30 goal=3.5 formula=normal units=%
call CONTROLIO io=MIXERA value=0 time=0
call EMPTY_APM200_CELLA time=15
call FLUSH_APM200_CELLA time=25
call HOMEMOTORS motor1=yes motor2=yes motor3=no motor4=no
end
```

HOMEMOTORS: This brings the burettes motors to their uppermost position.

BURETTE number = 1 action = setvol volume = 0.00 valve = EV01: Resets the system's tracking of the volume of reagent injected.

EMPTY_APM200_CELLA time = 15: Opens the drain valve for 15 seconds. The time can be adjusted to ensure that the drain fully empties prior to the next step.

FLUSH_APM200_CELLA time = 25: Spray rinses the cell for 25 seconds. This time can be adjusted to ensure the cell is completely rinsed prior to the next step.

SAMPLE time = 10 AV01: This command prepares the 6-port valve to take a sample. Additionally, it opens valve AV01, the sample 1 inlet valve, and diverts sample to the 6-port valve for eight seconds. This time can be adjusted to accommodate varying sample inlet flow rates. Note, the sample time only has to be long enough for a fresh sample to go from the 3-way recirculating AV01 to the 6-port valve.

TRANSFER_APM200_CELLA time = 6000: Prepares the 6-port valve to deliver sample to the measuring cell. Opens AV03 to transfer the contents of the sample loop to the measuring cell. AV03 will remain open until either the cell level sensor trip or the time is reached. If the level sensor is not tripped within the defined time (6000 seconds), the system will respond with an error code: 64.

CONTROLIO = MIXERA value = 1 time = 10: Turns the mixer motor on. This is not adjustable.

TITRATE (for the H_2O_2 measurement):

– cell = 1: Cell 1 in single cell system, cell 1 or 2 in dual cell system.

– number = 3: Defines which burette is used to perform the titration.

– channel_id = 2: Defines which displayed and analog output is used for this measurement.

– sensor_ch = 1: Defines the ORP electrode is connected to sensor input 1.

– endpoint = 850: Defines the titration endpoint value Can be optimized for the chemistry's endpoint.

– window = 100: Defines the tolerance in which the SemiChem APM 200 will seek for the actual endpoint. In this case, the SemiChem APM 200 will search from 750 to 950 mVs for the endpoint.

– end = 10.50: Defines when the titration is terminated.

– sensor = mV: Defines the displayed sensor response graph scale.

– algorithm = seek/set: Defines how the SemiChem APM 200 determines the endpoint. Seek allows the SemiChem APM 200 to determine the endpoint within the window. Setpoint assigns the endpoint at a fixed value. Seek/set allows the SemiChem APM 200 to first search for the endpoint within the window. If unable, then it will revert to the setpoint value.

– direction = up_1: Defines the direction of the titration. It also defines the method of reagent injection. Up value configures the reagent injection volumes based on the slope of the sensor response curve using the min. and max. increments. Up-1 value configures the reagent injection volumes based on a goal volume of where the endpoint is expected.

– min_inc = 0.03 & max_inc = 0.4: Sets the range of reagent volume injections. Min. increment is usually 0.03. Max. increment is usually 10% of the expected goal volume.

– goal = 3.5: When using the up-1 (or down-1) titration direction, the SemiChem APM 200 will optimize reagent volume injections based on the expected endpoint volume. As the total reagent volume injections get closer to the goal volume, the system will inject smaller and smaller increments as it slowly approaches the endpoint. Thus, giving the SemiChem APM 200 more data points in the critical portion of the sensor response curve to determine the most precise endpoint. This value can be adjusted to match the expected endpoint value. This feature is only suitable for applications in which the process is fairly stable.

– formula = normal: The calculation formula simply takes the endpoint volume and multiplies by a calibration factor to report sample concentration. Other formulas can be used, but are not applicable for the DSP+ application.

– units = %: Defines the units labeled on the display output.

– offset = 0.00: Defines if there is a reagent injection offset. This volume is subtracted from the overall reagent used in the recipe from a given burette to that point.

– equib = 3: Defines the amount of time (seconds) between reagent injection, mix, and sensor reading. In this case, the system is allowed three seconds for the solution to be mixed prior to a sensor reading. This can be adjusted if the sensor response curve is not smooth.

The recipe shown above is for the above stated assumption. However, these parameters can be adjusted to optimize the system. Additionally, if the process concentrations vary from the above stated assumption, then there may need to be further adjustments in the recipe, reagent concentrations, and/or sample loop size.

A.3.5 ORP SENSOR CALIBRATION

The ORP does not require calibrations.

A.4 RUNNING

A.4.1 FIRST RUN

If in local mode:

1. From the service screen, press "Recipes." Select recipe “HomeMotors”. This recipe will reset the motors. Press "Run Now."
2. From the Recipe screen, choose the appropriate recipe for the situation. Press "Save As Main." This will make this the default recipe.
3. Press "Run Now." The SemiChem APM 200 will proceed to perform the recipe. It will flush the cell, drain the cell, fill the sampling loop, and transfer the sample to the cell with DI water until it trips the level sensor. Then it will then add 5.0 mL of TISAB solution to bring the pH to about 5. The system will continue with the HF measurement. Once complete, the cell will drain, flush, and drain again. The SemiChem APM 200 will flush and rinse the cell, take another sample and it will begin with the titration of the hydrogen peroxide. After completion, the cell will again empty and flush, the burettes will reset and the displayed results will update.

If in remote mode:

1. Using the host PLC system, initiate a measurement sequence.
2. If using recipe select function, the SemiChem APM 200 will perform that recipe. The SemiChem APM 200 will proceed to perform the recipe. It will flush the cell, drain the cell, fill the sampling loop, and transfer the sample to the cell with DI water until it trips the level sensor. Then it will then add 5.0 mL of TISAB solution to bring the pH to about 5. The system will continue with the HF measurement. Once complete, the cell will drain, flush, and drain again. The SemiChem APM 200 will flush and rinse the cell, take another sample and it will begin with the titration of the hydrogen peroxide.After completition, the cell will again empty and flush, the burettes will reset and the displayed results will update. It will close the “READY” contact.

If in computer mode:

1. Using the host PC system, initiate a measurement sequence.
2. If using recipe select function, the SemiChem APM 200 will perform that recipe. The SemiChem APM 200 will proceed to perform the recipe. It will flush the cell, drain the cell, fill the sampling loop, and transfer the sample to the cell with DI water until it trips the level sensor. Then it will then add 5.0 mL of TISAB solution to bring the pH to about 5. The system will continue with the HF measurement. Once complete, the cell will drain, flush, and drain again. The SemiChem APM 200 will flush and rinse the cell, take another sample and it will begin with the titration of the hydrogen peroxide. After completion, the cell will again empty and flush, the burettes will reset and the displayed results will update. It will close the "READY" contact.

A.4.2 PROCESS CALIBRATION

The following procedure calibrates the SemiChem APM 200 to the process. The system can be calibrated with either the factor or process calibration method.

Factor Calibration:

1. The analyzer's calculation factor can be derived from first principals using the following equation:

Calculation Factor = (Ctit × RR × U × MW)/Vsamp

Ctit = Titrant concentration (normality)

RR = Reaction ratio (moles of sample that will react with each mole of titrant)

U = Unit factor (typical values: "1" for g/L, "0.1" for %, "1000" for mg/L, etc.)

MW = Molecular weight of sample species (e.g, H_2SO_4 = 98)

Vsamp = Volume of sample taken (typically 0.25 – 1 mL)

For example:

Ctit = 0.2

RR = 0.5

U = 0.1

MW = 98

Vsamp = 0.5

4.900 = (Ctit × RR × U × MW)/Vsamp

This factor is used in the Settings/Calculation Factors for output 1.

Process Calibration:

1. At the same time the SemiChem APM 200 takes a sample, take a grab sample.
2. Once the SemiChem APM 200 is complete, note the result. For example: 0.816 mL.
3. Make a note of a trusted laboratory's result. For example: 4.00% H_2SO_4.
4. Divide the laboratory result by the SemiChem APM 200 result. For example: 4.00/0.816 = 4.900.
5. In this case, 4.900 is the factor to be used in the Settings.

The calculation factor converts the SemiChem APM 200's milliliters of titrant to reach the endpoint into a concentration that the customer can use for process control. Once the SemiChem APM 200 is calibrated to the process, it should not have to be repeated unless maintenance is performed on the burette O-rings, there is a there is a change in reagent, or there is a change in electrode.

At this point, the SemiChem APM 200 is reassembled, calibrated, and ready for use.

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