

PLANT OPERATIONS MANUAL

Volume 08

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Section 04

Revision 1

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CHEMISTRY INSTRUCTION

OPERATION OF ORION 701 ION ANALYZER

SAFETY RELATED

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1.0 INSTRUMENT DESCRIPTION

Orion Digital pH/Mv Meter 701A and Manual Electrode Switch Model 605

2.0 REFERENCES

- 2.1 Orion Research Instruction Manual IM701A, 460000949
- 2.2 Orion Research Instruction Manual IM605, 460001037
- 2.3 Chemistry Section Procedure 08-S-03-1, Qualification of Chemistry Program
- 2.4 Chemistry Section Procedure 08-S-03-7, Control of Chemistry Support Equipment

3.0 DEFINITIONS

- 3.1 Specific Ion Electrode - A device that develops an electrical potential proportional to the logarithm of the activity of an ion in solution.

4.0 PREREQUISITES

- 4.1 Specific ion electrode
- 4.2 Reference electrode
- 4.3 An assortment of reference standards
- 4.4 Reference electrode filling solution

5.0 PRECAUTIONS

- 5.1 Always make measurements or calibrations with the test solution in a non-metal container.
- 5.2 Place the instrument function switch in standby prior to removing the electrodes from solution.

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- 5.3 To eliminate warm-up time and increase component life, always leave the meter in the STDBY mode rather than disconnect from line power. Warm-up time is 30 minutes.
- 5.4 Check and ensure reference electrode filling solution is at least one inch above the solution being measured.
- 5.5 Allow all samples and standards to reach the same temperature before attempting a measurement.
- 5.6 Stir both standards and samples with a magnetic stirrer while a specific ion measurement is being made. Some magnetic stirrers generate enough heat to increase the temperature of the solution. To avoid this, place a piece of thermal insulating material between the stirrer and beaker.
- 5.7 Rinse electrodes with demineralized water between measurements. Do not blot dry.
- 5.8 Never use an individual reference and a common reference electrode in the same solution.

6.0 INSTRUCTIONS

6.1 Functional Check - 701A Ionalyzer

- 6.1.1 Connect the shorting strap between the input jack and reference jack, and turn the function switch to STDBY.
- 6.1.2 Connect the power cord of the 701A to a suitable power source. Let the meter warm up for thirty minutes.
- 6.1.3 With the function switch on STDBY, verify that a decimal point only is displayed in the middle of the display.
- 6.1.4 Turn the function switch to REL MV and verify a display of polarity sign and four digits.

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- 6.1.5 Using the calibration control, verify that the display is adjustable from about -200mv to +200mv. Adjust the calibration control for a display of 000.0.
 - 6.1.6 Turn the function switch to pH/.001.
 - 6.1.7 Turn the ISO ADJ screw to obtain the isopotential point (7.000). Verify that turning the temperature compensator knob does not change the reading by more than ± 0.002 pH.
 - 6.1.8 Turn the function switch to MV. Verify a display of 000.0 ± 0.1 . If the display is outside these limits, repeat steps 6.1.6 and 6.1.7. If still out, terminate test and notify Chemistry Supervisor.
 - 6.1.9 Turn the function switch to pH/.001. Set the slope indicator dial to 100% and turn the temperature compensator knob until the white arrow points to 29°C. At this temperature a change of one pH unit corresponds to a 60 mv change in potential.
 - 6.1.10 Turn the calibration control until a reading is obtained that is 0.50 pH units less than the isopotential point.
 - 6.1.11 Set function switch to REL MV and verify a display of 30.0 ± 0.3 mv. If the meter does not check out in any of the above steps, repeat the step. If the second attempt fails, contact the Lead Laboratory Chemist.
- 6.2 605 Electrode Switch
- 6.2.1 Operation
 - a. The Channel Selector Switch on the instrument is used to select from any channel 0 through 5. When channel 0 is selected, standardization is accomplished utilizing the pH/mV instrument standardization control and not one of the five channel controls on the electrode switch. Channel 0 must be standardized before switching the electrode switch on.

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- b. Standardize each channel used by placing the selector switch to the corresponding channel and rotating the calibrate control for that respective channel to the desired reading.
- c. To verify the original setting at any time, connect a shorting lead between channel 0 reference and sensing electrode input jacks and note the readings. If the readings do not coincide with the original readings, the controls have been moved and the channel will require restandardization.

6.2.2 Battery Check

- a. To perform an instrument battery check, verify that the electrode switch is connected to the respective meter, i.e., 701A.
- b. Set the Meter Selector Switch to the MV position.
- c. Switch the electrode switch off.
- d. Connect a shorting lead between the battery check jack on the rear panel and the sensing electrode input jacket of channel 0.
- e. Verify that the electrode switch channel selector points to channel 0.
- f. Read the battery voltage on the meter. A reading of 250 millivolts or greater indicates proper battery voltage. If less, contact the Chemistry Supervisor.

6.3 Operation

6.3.1 The 701A Ion Analyser can be used for both pH measurements or specific ion analysis.

- a. pH Measurements

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- (1) Select two buffers whose pH values bracket the expected sample pH, with one buffer between pH 6 and pH 8. Check the label on the buffer packet or bottle for the exact pH at the solution temperature.
- (2) Set the isopotential point to the pH of the buffer between pH 6 and 8. (To change the isopotential point from the factory setting of 7.000, connect the shorting strap between the input and reference jacks. Turn the function switch to REL MV and set the reading to 000.0 with calibration control. Turn the function switch to pH/.01. Turn the ISO ADJ screw on the back of the meter until the desired isopotential point is displayed.)
- (3) Remove the shorting strap. Attach the combination pH electrode or pH electrode and reference electrode securely to the meter. Turn the function switch to pH/.01.
- (4) Place electrodes in the buffer whose pH is closest to isopotential point. Turn the calibration control until the isopotential point is displayed.
- (5) Remove the electrodes from the isopotential buffer and rinse.
- (6) Place the electrodes in the second buffer solution.
- (7) If necessary, turn the temperature compensator until the pH value of the second buffer is displayed. Turn the slope indicator dial until the temperature compensator arrow points to the buffer temperature. Read the percent of the theoretical Nernst slope at the "% slope" index line. If the % Nernstian slope is less than 90%, repeat the standardization with fresh buffers. If the % slope is again less than 90%, see MEASUREMENT DIFFICULTIES, page 16 of Reference 2.1.

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- (8) Remove electrodes, rinse, and place in the unknown sample.
- (9) Allow about one minute for the reading to stabilize, and record the pH measurement directly from the display.
- (10) Place the instrument function switch in standby, rinse the electrodes and store in demineralized water.

b. Specific Ion Measurements

NOTE

Individual instructions are written for each specific ion measurement. The following is to be used as a general instruction for use of the 701/605 combination. Where conflicts exist, specific ion measurement instructions take precedence:

- (1) Multiple specific ion measurements can be made by connecting the Orion Model 605 manual electrode switch to the ion analyser.
- (2) Prepare three standards that bracket the unknown sample concentration. Add the ionic strength adjustor, or pH adjustor, recommended by the electrode instruction manual. For greater accuracy, more standards may be prepared and measured.
- (3) Place electrodes in the mid range standard and select proper channel on the electrode switch.
- (4) Set the function switch to the mV (absolute) position. Wait for the prescribed time, read, and record.
- (5) Set the function switch to the REL mV position. Wait for a stable reading. Adjust the individual calibration control until the meter reads 000.0. Since the offset potential in the REL MV mode is about +200mv, it is

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impossible to zero the meter when the electrode potential is outside these limits; in these cases, simply turn the function switch to the MV position and record the electrode potentials to 0.1mv.

- (6) Rinse electrodes and place in the most dilute standard. Wait for the prescribed time, read and record.
- (7) Rinse electrodes and place in the most concentrated standard. Wait for the prescribed time, read, and record.
- (8) Using semilogarithmic graph paper, prepare a calibration curve by plotting the electrode potentials measured (millivolt readings) on the linear axis and the activity or concentration value of the standards on the logarithmic axis.
- (9) Rinse electrodes and place in the unknown sample. Wait for the prescribed time, read and record.
- (10) Using the calibration curve, determine the concentration of the unknown.

7.0 DOCUMENTATION/CORRECTIVE ACTION

- 7.1 Enter operator's initials and date check performed on standardization card attached to the instrument.
- 7.2 Document results on proper reporting forms as per Chemistry Procedure 08-S-03-10.