

Method: CAL-3 Revision: 1 Final Revision Date: 06/02/03	Laboratory Instrument Calibration Method	INEOS Nitriles
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METHOD SUMMARY

The definition of pH is the instantaneous hydrogen ion activity of the sample as measured by an electrode. The electrode system is based on the fact that a change of 1 pH unit produces an electrical change of 59.1 mV at 25° C. The pH of the sample is measured electrometrically with an electrode in combination with a reference potential. This method is used to calibrate the pH scale of a pH meter. Precision of pH measurement has been demonstrated at + or - 0.11 units at the 6.86 level (95%CL), with an accuracy of 99.1% recovery at the 6.86 level.

SAFETY

Acrylonitrile and acetonitrile are hazardous to the health and dangerous to handle. Use acrylonitrile and acetonitrile in a well ventilated hood. Review the MSDS for detailed information concerning toxicity, first aid procedures and safety precautions.

Refer to the appropriate safety section or site manual for the necessary protective equipment to use when handling any reagents or samples.

REFERENCES

ASTM D1293-99, "Standard Test Methods for pH of Water" <http://www.astm.org/>

APPARATUS AND REAGENTS

1. **Electronic pH Measurement Apparatus:** Fisher Accumet 915 or equivalent.
2. **Stirring motor,** magnetic, variable speed with inert, plastic stirring bars.

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3. **Beaker**, 100 mL glass.
4. **Reference Buffer Solutions:** pH 4,7, and 10, traceable to NIST Standards.

CALIBRATION PROCEDURE

1. To calibrate the Accumet 915 pH meter:

For a Two Point Calibration:

- a. Put pH-4 buffer in a beaker and put the tip of the electrode in the solution.
- b. Press STBY/MEAS and check that the instrument is in pH mode.
- c. Press TWO POINT CAL. The upper screen will display 4.00 and the lower screen will read STD BUFFER 1. Press ENTER. When the millivolts have stabilized, press ENTER again.
- d. Put pH 10 buffer in a second beaker, rinse the electrodes with water and immerse the tip of the electrode in the pH 10. The upper screen will read 10.0 and the lower screen will read STD BUFFER 2. Press ENTER and allow the millivolts to stabilize (approx. 175 mV). Press ENTER again. The upper screen will now display the Efficiency of the electrode. If it is between 0.97 and 1.03 press ENTER. The meter will now be reading the pH of the buffer solution.

For Measuring pH Quality:

2. After standardization the meter will be in pH mode. Place pH 7 buffer into a clean beaker with a stirring bar.
3. Put the beaker on the stirring plate and mix at the same speed as the calibration measurements. Rinse the electrode with DI H₂O into a rinse beaker.
4. Put the electrode into the pH 7 buffer. Allow the reading to stabilize. Record the pH of the buffer solution.

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5. Record the reading in a calibration log book.

CALCULATIONS

$$\% \text{ Accuracy of the standard} = \frac{(\text{pH from Analysis}) * 100\%}{\text{pH of Standard}}$$

REPORT

Report the percent accuracy to the nearest whole number and record in the logbook.