The pH*Detect*™ Option for the Model 4660 Eclipse P&T Sample Concentrator

Application Note 26560706

Keywords

Eclipse Reporter Software
Eclipse Sample Concentrator
Model 4551A Water Autosampler
P&T
pH
pH*Detect*™
Purge-and-Trap
RTD
Resistance Temperature Detector
USEPA
VOC
Volatile Organic Compound

Introduction

The pH*Detect*™ option is an external module for the Model 4660 Eclipse Purge-and-Trap (P&T) Sample Concentrator that automatically measures the pH of Volatile Organic Compound (VOC) water samples to verify that the pH level meets the requirements specified in EPA, ISO, and other regulatory methods. Figure 1 shows the pH*Detect* option (patent pending) with the Model 4660 Eclipse P&T Sample Concentrator.

Figure 1. pH*Detect* Option and Model 4660 Eclipse P&T Sample Concentrator

The USEPA requires that water samples be preserved with acid to pH less than 2 for VOC methods, including USEPA methods 502, 524, 624, and 5030. Other regulatory methods, such as the Massachusetts VPH Method, provide the option of preserving the sample by raising the pH of the sample above 11. When following these methods, replicate samples must be collected in the field solely for measuring the pH. Without the pH*Detect* option, the analyst opens the extra sample vial, measures the pH manually, and records the measurement in a log; this procedure is both time-consuming and labor-intensive.

Alternatively, the technician or chemist must manually check the pH level of every vial after the VOC analysis is complete. The pH*Detect* option eliminates the need for collecting additional samples and the labor and materials associated with manual pH measurement. The pH*Detect* option electronically records all calibrations and sample pH measurements in the Eclipse database with a date and time stamp; the measurements can then be viewed, printed, or exported using the Eclipse Reporter software.

The automation of pH readings using the pH*Detect* module can greatly minimize labor costs and increase laboratory productivity.

pH*Detect* Description of Components

The pH*Detect* components are designed for easy maintenance, high reproducibility, reliable calibrations, and low carryover. The glass sample reservoir is visible through the front panel of the pH*Detect* module to provide visual confirmation of the filling and draining processes. The pH probe is designed with a small resistance temperature detector (RTD) and without a sheath over the pH electrode for accurate recording of all sample pH levels. All internal lines and materials in the sample pathway are either glass or Teflon® to minimize sample carryover and ensure reproducibility. Figure 2 shows the pH probe and the glass reservoir filled with pH 4.01 buffer.

The pH*Detect* includes two 500-mL reservoirs that contain the standard pH buffers for calibration. The reservoirs are easily accessible for refilling through the top of the pH*Detect* module and are connected to a valve manifold with color-coded Teflon® lines and fittings. The reservoirs are filled with a combination of two commercially available pH buffers:

- pH 4.01 and 7.00 for acidified samples (supplied in the instrument startup kit),
- pH 7.00 and 10.00 for basic samples, or
- pH 4.01 and 10.00 to cover the entire pH range.

Two simple connections between the Eclipse and the pH*Detect* module enable a fast and straightforward installation. The sample drain line from the Eclipse is connected to the inlet port on the back of the pH*Detect* module, and a waste line from the pH*Detect* module carries excess sample and rinse water to the waste receptacle. All power and communications for the pH*Detect* option are provided via a single I^2C communication cable that connects directly to the Eclipse.

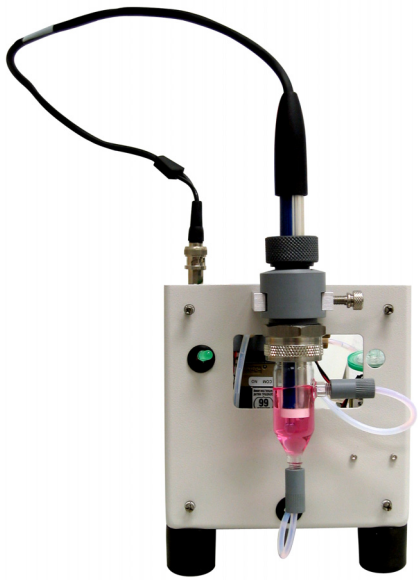


Figure 2. pH probe and glass sample reservoir filled with pH 4.01 buffer

pH*Detect* Principle of Operation

Overview

As the VOC water sample drains from the Eclipse sparge vessel during DESORB, it is collected in the glass reservoir on the front panel of the pH*Detect* module. A precise pH electrode measures the pH level, the pH reading is logged with a date and time stamp in the Eclipse database, and the sample drains from the reservoir. The Eclipse then prepares the sample pathway for the next sample by purging with gas and flushing with autosampler rinse water during the normal Eclipse rinse cycle.

Calibration of the pH electrode is fully automated using the two onboard buffer solutions. All calibrations and sample pH measurements are logged in the Eclipse database with a date and time stamp. This information is accessible from a networked PC for reporting using the Eclipse Reporter or for exporting to a spreadsheet, local area network (LAN), or LIMS.

Calibration

The automated calibration sequence of the pH*Detect* module can be initiated on demand any time the Eclipse is in standby by pressing the **Calibrate pH** button on the pH Settings screen in the Maintenance section of the software (refer to Figure 3), or the calibration can be programmed to occur during the PURGE state at pre-defined calibration intervals (as shown in Figure 4). During calibration, the pump on the pH*Detect* module supplies air pressure to the buffer bottles, the calibration valve opens, and the air pressure forces the buffer solution into the glass sample reservoir. The glass reservoir is rinsed twice with the buffer solution, and the pH of the calibration buffer is then measured on the third and final fill. The procedure is repeated with the second buffer, and the successful calibration is logged with a date and time stamp in the Eclipse database.

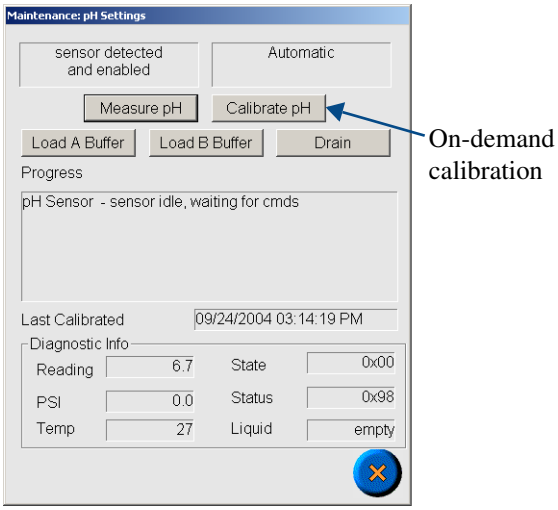


Figure 3. Maintenance: pH Settings Screen

The pH probe can be calibrated any time the Eclipse is in the Standby state by pressing the "Calibrate pH" button on the Maintenance: pH Settings screen.

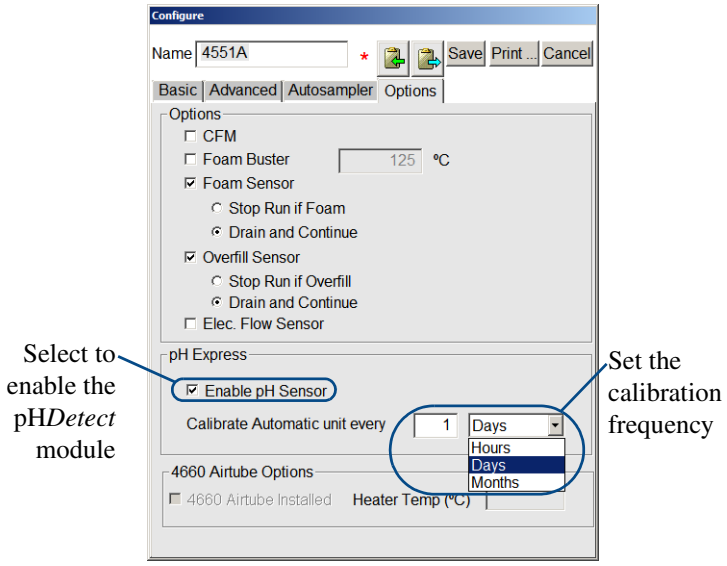


Figure 4. Eclipse Configuration Screen

The pHDetect option is enabled and configured from the Eclipse Configuration screen; the automatic calibration interval is selected from a drop-down menu.

Sample pH Measurement

The sample pH readings are enabled in the method using either the Eclipse Active Method screen or the Method Editor, as shown in Figure 5. As the water sample drains from the Eclipse sparge vessel during DESORB, it is collected in the glass reservoir on the front panel of the pH*Detect* module, and any excess sample flows out the drain line to the waste receptacle. The precise pH electrode measures the pH level of the sample, which is then logged in the Eclipse database with a date and time stamp. When configured with a 4551A Autosampler, the autosampler rinse water and helium flush the glass reservoir and sample lines. During the pH*Detect* option's drain cycle, the drain valve at the bottom of the glass reservoir opens, and the sample is removed through the bottom of the reservoir using pressurized air. The entire sample pathway, including the glass reservoir and all lines, is then flushed with pressurized air to prevent sample carryover.

The calibration and sample pH measurements are transferred to the Eclipse via the I^2C communication cable of the pH*Detect* module and are stored in the Eclipse database for subsequent reporting.

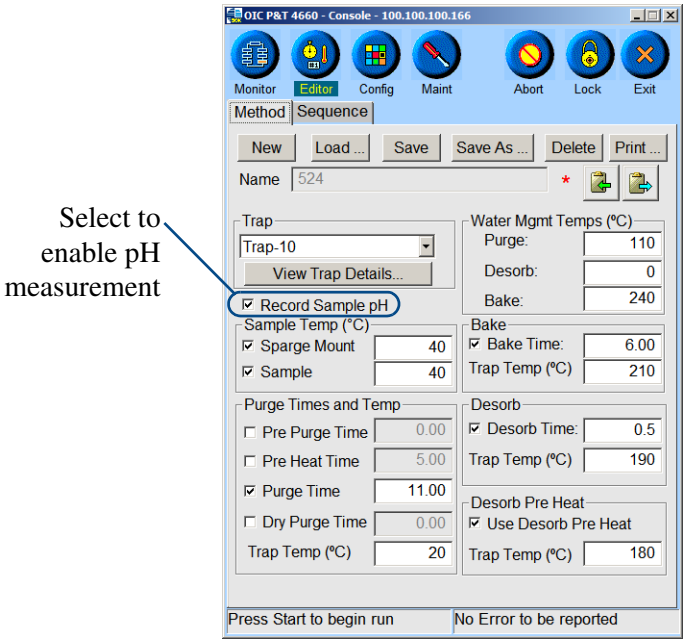


Figure 5. Eclipse Method Editor Screen

Select "Record Sample pH" in the Eclipse Method Editor or Active Method screen to enable pH measurement and recording for all VOC water samples.

Eclipse Reporter Software, Version 2.1

Once the pH data have been recorded and stored in the Eclipse database, they can be viewed directly from the Eclipse Instrument Log file. The data can also be imported to a PC for viewing, reporting, and exporting to a delimited text file using the Eclipse Reporter software. Figure 6 illustrates the pH readings as they are viewed from the Eclipse Instrument Log file (accessed through the Maintenance screen). This view displays the date and time that the sample pH was measured, which user was logged onto the instrument, the vial position when used with the Model 4551A Water Autosampler, the measured pH level for the sample, and the date of the last calibration.

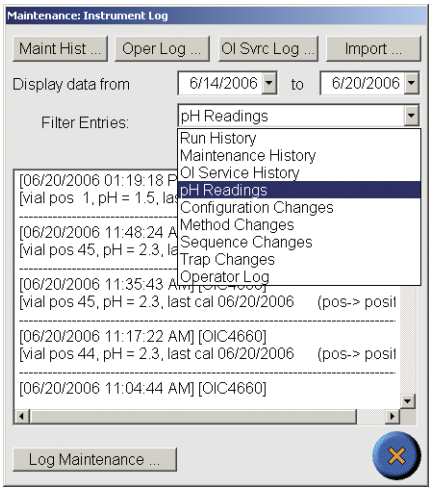


Figure 6. Maintenance: Instrument Log Screen

The Maintenance Instrument Log file can be used to view all of the pH calibrations and sample measurements that have been saved in the database.

The calibrations and sample pH measurements are stored electronically in the Eclipse database. To view, print, or export pH data using the Eclipse Reporter software, the files must first be imported to a PC that is connected to the Eclipse through a LAN network. Once the data have been imported to the PC, the reporting software feature is activated from the Eclipse Maintenance screen, as shown in Figure 7. The user defines a Start Date and End Date to select the range of pH data to be reported. Start and End times can also be specified to isolate data from specific analytical sequences for reporting. The **pH Log** button is selected from the list of available Reports, and the report of selected pH data is displayed on the PC for viewing or printing, as shown in Figure 8.

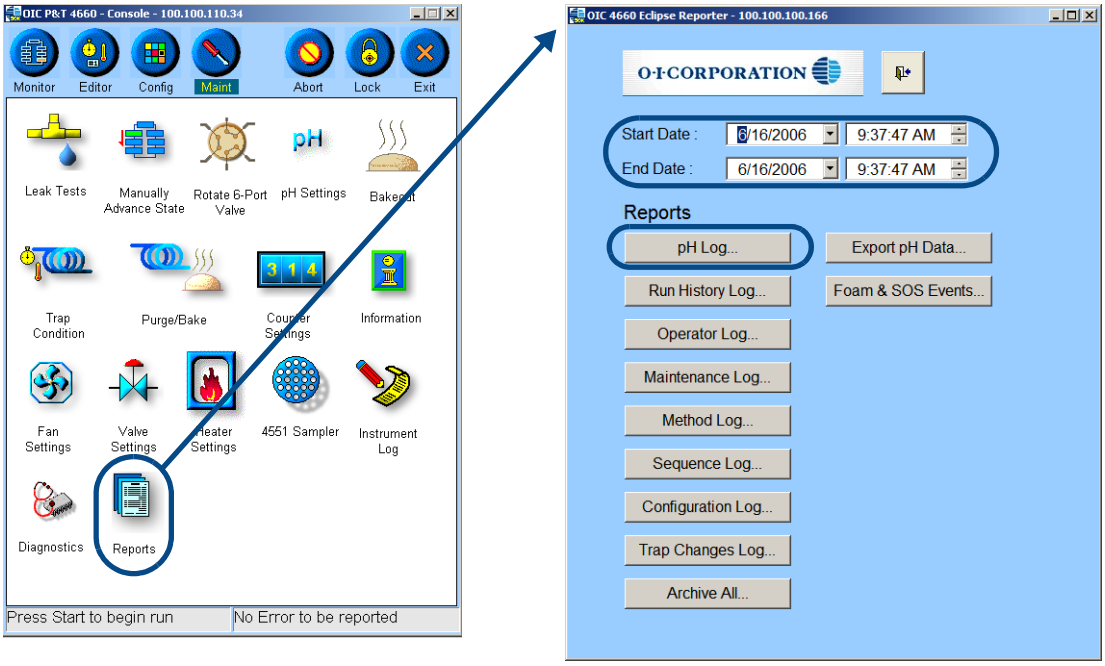
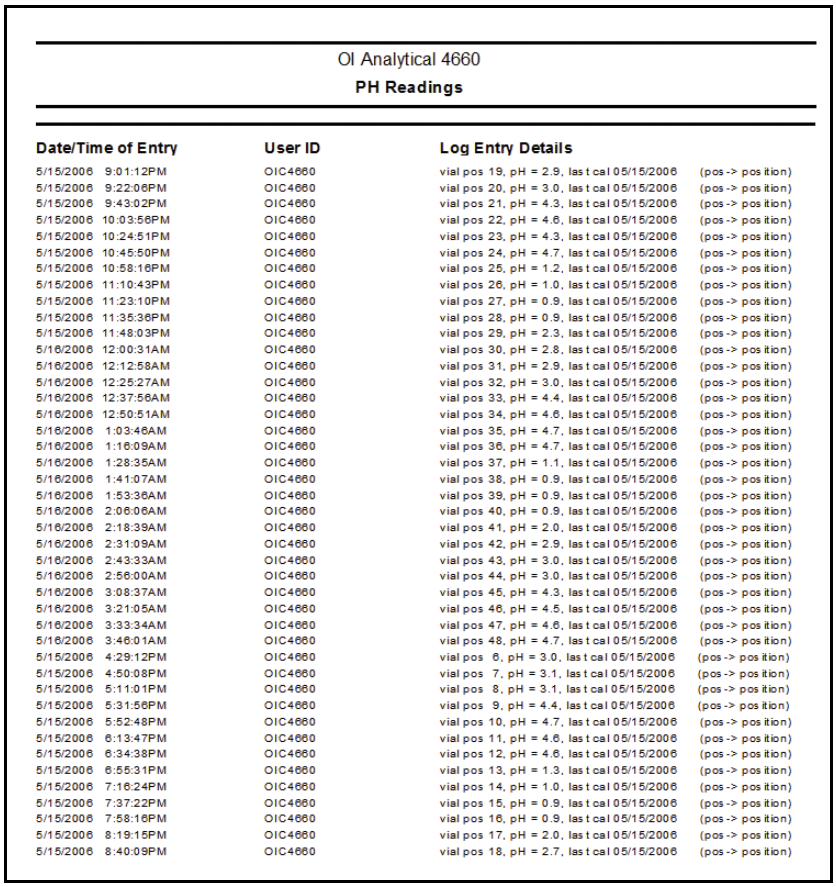


Figure 7. Eclipse Maintenance Screen and Eclipse Reporter Screen for Reports

Maintenance screen from the PC version of the Eclipse software with the "Reports" icon (left), and the Eclipse Reporter screen (right) for selecting the date/time range and type of report.



OI Analytical 4660

PH Readings

Date/Time of Entry	User ID	Log Entry Details
5/15/2006 9:01:12PM	OIC4660	vial pos 19, pH = 2.9, last cal 05/15/2006 (pos -> position)
5/15/2006 9:22:06PM	OIC4660	vial pos 20, pH = 3.0, last cal 05/15/2006 (pos -> position)
5/15/2006 9:43:02PM	OIC4660	vial pos 21, pH = 4.3, last cal 05/15/2006 (pos -> position)
5/15/2006 10:03:56PM	OIC4660	vial pos 22, pH = 4.6, last cal 05/15/2006 (pos -> position)
5/15/2006 10:24:51PM	OIC4660	vial pos 23, pH = 4.3, last cal 05/15/2006 (pos -> position)
5/15/2006 10:45:50PM	OIC4660	vial pos 24, pH = 4.7, last cal 05/15/2006 (pos -> position)
5/15/2006 10:58:16PM	OIC4660	vial pos 25, pH = 1.2, last cal 05/15/2006 (pos -> position)
5/15/2006 11:10:43PM	OIC4660	vial pos 26, pH = 1.0, last cal 05/15/2006 (pos -> position)
5/15/2006 11:23:10PM	OIC4660	vial pos 27, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/15/2006 11:35:36PM	OIC4660	vial pos 28, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/15/2006 11:48:03PM	OIC4660	vial pos 29, pH = 2.3, last cal 05/15/2006 (pos -> position)
5/16/2006 12:00:31AM	OIC4660	vial pos 30, pH = 2.8, last cal 05/15/2006 (pos -> position)
5/16/2006 12:12:58AM	OIC4660	vial pos 31, pH = 2.9, last cal 05/15/2006 (pos -> position)
5/16/2006 12:25:27AM	OIC4660	vial pos 32, pH = 3.0, last cal 05/15/2006 (pos -> position)
5/16/2006 12:37:56AM	OIC4660	vial pos 33, pH = 4.4, last cal 05/15/2006 (pos -> position)
5/16/2006 12:50:51AM	OIC4660	vial pos 34, pH = 4.6, last cal 05/15/2006 (pos -> position)
5/16/2006 1:03:46AM	OIC4660	vial pos 35, pH = 4.7, last cal 05/15/2006 (pos -> position)
5/16/2006 1:16:09AM	OIC4660	vial pos 36, pH = 4.7, last cal 05/15/2006 (pos -> position)
5/16/2006 1:28:35AM	OIC4660	vial pos 37, pH = 1.1, last cal 05/15/2006 (pos -> position)
5/16/2006 1:41:07AM	OIC4660	vial pos 38, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/16/2006 1:53:36AM	OIC4660	vial pos 39, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/16/2006 2:06:06AM	OIC4660	vial pos 40, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/16/2006 2:18:39AM	OIC4660	vial pos 41, pH = 2.0, last cal 05/15/2006 (pos -> position)
5/16/2006 2:31:09AM	OIC4660	vial pos 42, pH = 2.9, last cal 05/15/2006 (pos -> position)
5/16/2006 2:43:33AM	OIC4660	vial pos 43, pH = 3.0, last cal 05/15/2006 (pos -> position)
5/16/2006 2:56:00AM	OIC4660	vial pos 44, pH = 3.0, last cal 05/15/2006 (pos -> position)
5/16/2006 3:08:37AM	OIC4660	vial pos 45, pH = 4.3, last cal 05/15/2006 (pos -> position)
5/16/2006 3:21:05AM	OIC4660	vial pos 46, pH = 4.5, last cal 05/15/2006 (pos -> position)
5/16/2006 3:33:34AM	OIC4660	vial pos 47, pH = 4.6, last cal 05/15/2006 (pos -> position)
5/16/2006 3:46:01AM	OIC4660	vial pos 48, pH = 4.7, last cal 05/15/2006 (pos -> position)
5/15/2006 4:29:12PM	OIC4660	vial pos 6, pH = 3.0, last cal 05/15/2006 (pos -> position)
5/15/2006 4:50:08PM	OIC4660	vial pos 7, pH = 3.1, last cal 05/15/2006 (pos -> position)
5/15/2006 5:11:01PM	OIC4660	vial pos 8, pH = 3.1, last cal 05/15/2006 (pos -> position)
5/15/2006 5:31:56PM	OIC4660	vial pos 9, pH = 4.4, last cal 05/15/2006 (pos -> position)
5/15/2006 5:52:48PM	OIC4660	vial pos 10, pH = 4.7, last cal 05/15/2006 (pos -> position)
5/15/2006 6:13:47PM	OIC4660	vial pos 11, pH = 4.6, last cal 05/15/2006 (pos -> position)
5/15/2006 6:34:38PM	OIC4660	vial pos 12, pH = 4.6, last cal 05/15/2006 (pos -> position)
5/15/2006 6:55:31PM	OIC4660	vial pos 13, pH = 1.3, last cal 05/15/2006 (pos -> position)
5/15/2006 7:16:24PM	OIC4660	vial pos 14, pH = 1.0, last cal 05/15/2006 (pos -> position)
5/15/2006 7:37:22PM	OIC4660	vial pos 15, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/15/2006 7:58:16PM	OIC4660	vial pos 16, pH = 0.9, last cal 05/15/2006 (pos -> position)
5/15/2006 8:19:15PM	OIC4660	vial pos 17, pH = 2.0, last cal 05/15/2006 (pos -> position)
5/15/2006 8:40:09PM	OIC4660	vial pos 18, pH = 2.7, last cal 05/15/2006 (pos -> position)

Figure 8. Eclipse Reporter pH Data Report

Report of pH data displayed using the Eclipse Reporter. A hard copy of the report can also be printed from this screen.

Finally, using the Eclipse Reporter Software, the pH data can also be exported to a delimited text file for use with other software programs—such as Microsoft® Excel or LIMS programs. The export function allows the user to select the output text delimiter (such as a tab, comma, or space) and to specify the directory and filename where the file is saved. The delimited data are exported and saved in the specified location as a text file with a `txt` extension. Figure 9 shows the Export options and an example of the text file after exporting the data to a tab-delimited file. Once in a delimited text format, the information is easily imported to a spreadsheet program or LIMS system.

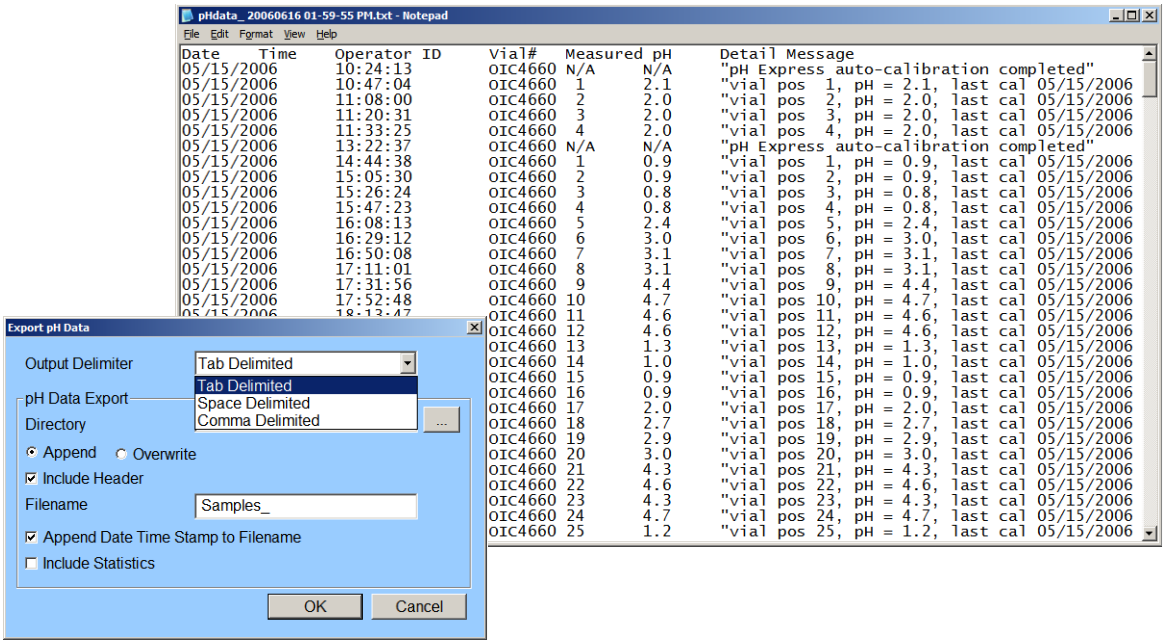


Figure 9. Export pH Data Dialog Box and Example of a Tab-Delimited Data File

The menu used to define the options for exporting data to a delimited text file (bottom left) and an example of the data text file after export (top right).

Summary and Conclusion

The pH*Detect* option for the OI Analytical Model 4660 Eclipse P&T Sample Concentrator improves laboratory productivity and profitability by eliminating the time and materials required for manual measurement of pH levels in VOC water samples.

The USEPA and other regulatory VOC methods require that VOC water samples be preserved with acid or base to prevent biodegradation. Without the pH*Detect* module, the pH level of these samples must be confirmed manually after P&T analyses using pH indicator strips, and the results must further be transcribed by hand to the final customer report. The pH*Detect* option fully automates the measuring, recording, and reporting of pH measurements for VOC water samples, increasing laboratory productivity and reducing costs by eliminating manual pH measurement steps.

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Publication 26560706