



EOSystems, Inc.

## PRELIMINARY RESULTS: VOC MONITORING OF CEROX SYSTEM 4 OPERATION AT UNR

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### THE EQUIPMENT

The VOC (volatile organic compounds) measurements reported here as preliminary results were made using a SXC-20 VOC monitor manufactured by Spectrex Corporation in Redwood City, CA. The unit has a broad spectrum sensor for organic compounds which develops a voltage signal proportional to the carbon content of the gas sensed. The signal level can be set with a known organic concentration using a standard calibration gas so that the output of the unit can be quantified. This voltage signal is stored in an onboard data logger in a form that can be downloaded to a computer for the permanent record.

Since the SXC-20 unit only senses carbon equivalents without regard to the chemical identity of the actual material sensed, the SXC-20 is equipped with a secondary sampling loop that is activated if the preset threshold for VOC's is reached. This secondary loop pulls a second gas sample from a second gas loop through an activated carbon sampling tube for off-line de-sorbition and analysis by gc/ ms (gas chromatography/mass spectrometry) if needed. This feature allows for possible identification of any overthreshold VOC materials detected.

### THE SAMPLING SET-UP

The Experimental Protocol calls for the sampling of the CerOx System 4 exhaust gas streams from the reactor units before passage through the Axi-Shear scrubber unit and after the Axi-Shear unit to verify compliance with of the CerOx unit with air quality regulations. The preliminary results were obtained by sampling only the exhaust stream from the Axi-Shear to simplify the development and testing of the water removal method from the gas stream prior to analysis. The gas stream exiting from the Axi-Shear unit is much cooler (ambient temperature) with a lower water content compared to the exhaust gas stream exiting the gas phase reactor (about 100° C) and saturated with water vapor (and a little nitric acid vapor).

The exhaust gas stream was dried using a Perma Pure membrane sample gas dryer. This unit allows the wet exhaust gas to pass through a Nafion™ membrane which has a counter current flow of dry air passing over the outside surface of the tube. This unit only removes water from the sample gas stream while permitting the free passage of the other gaseous components of the gas stream.

The sampling protocol for the use of the SXC-20 unit is given in detail in an attachment at the end of this Note. The calibration of the SXC-20 unit at the beginning of the operation and at the end of the operation being monitored puts the calibration record in the data log and verifies the proper operation of the unit over the period of operation. This protocol was developed and finalized &

during the first week of operation as familiarity and operating skill with the instrument was refined with experience.

## REPRESENTATIVE RESULTS

The SXC-20 unit was set with a input sample flow rate 150 ml/min (0.15 l/min) which passes over the VOC sensor element. This sample stream is pulling from the Axi-Shear exhaust stream that has a flow of 28 SCFM (800 l/min), This volumetric flow has an air mass flow of  $\pm 1.1$  kg/min of air. Some representative data are presented below that confirms the performance specifications of the CerOx System.

Figure Dec 09 97: The drum of waste formaldehyde was finished in the time period shown. The SXC-20 showed considerable drift during the day. The VOC levels detected were in the 10-15+ range. The cause of the drift is unknown. The unit appeared to be more stable on the next day (December 10).

Figure Dec 10 97: The processing of a drum of waste ethanol from the UNR Histology Laboratory was started in the time period shown. The SXC-20 showed a lower drift compared to December 09. The VOC levels detected during processing were below 10ppm.

## QUANTITATIVE EVALUATION OF RESULTS

The SXC-20 VOC monitor is calibrated with a standard gas having 100ppm methane in nitrogen. The unit is calibrated in C<sub>1</sub> units. That is, 50 ppm of ethanol, as a C<sub>2</sub> molecule, would have the same signal as the 100ppm calibration gas. In like manner 33ppm of a C<sub>3</sub>, such as acetone, would give rise to a signal of the same strength as the 100ppm of methane.

For the CerOx exhaust stream of 800 l/min, the mass flow as air is 1.1 kg/min (as air). A 100ppm level of methane in this stream would have a C<sub>1</sub> mass flow of 55mg/min (of methane). For an 8 hour day this level works out to about 0.1 Lb of methane (or C<sub>1</sub> equivalent).

The measured levels in the preliminary data reported in the previous section range up to the 10-15 ppm

C<sub>1</sub> equivalent range with most below the 10ppm level. The upper level (10ppm) would have about 10% of the mass flux calculated for the 100ppm calibration gas or about 0.01 lb. C<sub>1</sub> per 8 hour shift. This

is well below any established *de minimis* level.

## FURTHER WORK

The SXC-20 unit will be deployed during all processing of organic materials in the CerOx System 4 unit. The SXC-20 will be calibrated with the standard calibration gas before and after each run. The instrument drift, if it persists, will be checked by the factory. The process gas streams will be sampled by the SXC-20 before and after the Axi-Shear scrubber to correlate with the TOC (total organic carbon) analyses of the Axi-Shear water effluent and to determine the fraction of measured VOC (in the post Axi-Shear gas stream) that is externally (to the CerOx System 4) generated.

## OPERATION PROTOCOL FOR THE SPECTREX SXC-20 VOC MONITOR

The Spectrex SXC-20 is a broad range monitor/sensor for VOC's in ambient air. It contains a time based onboard data logger which records time (interval is selectable) vs. detector output (in volts). The unit can be set to yield semi-quantitative measurements through calibration with a gas

having a known VOC composition. This is the approach used here. The SXC-20 also has the capability to trap any VOCs, that exceed a pre-set threshold, by pulling a slipstream through an activated carbon trap. The carbon trap, if used, can then be used to identify the detected component(s) by off-line gc/ms analysis.

## SXC-20 SET-UP

The unit can be operated using either its integral rechargeable battery for 8-10 hrs of operation (after charging) or its ac adapter if a longer operation time is desired. The unit is calibrated with a 100ppm methane in nitrogen certified calibration gas at the start and at the end of each run. This dual calibration verifies operation and allows for a reasonably quantitative estimate of the levels of detected VOC's, if any. The flow rates of both pumps have been set at 150ml/min. These values can be adjusted and/or reset, if necessary, by following the instructions in the manual.

## INITIALIZATION

1. Turn on unit and allow to warm up for 5 minutes.
2. Attach the SXC-20 to a PC Computer running the StowAway™ program by using the pin connector attached to that computer.

Double click the Bxcpro icon on the desktop to start the StowAway™ program.

Double click the Launch Logger toolbar (In the Stowaway Program) to access the SXC-20.

Select the sample time/interval and give the data file a name; use the day's date.

Initiate the Logger and disconnect from the computer.

3. Open the SXC-20 case by removing the 4 screws from the back.

4. Attach a gas line from the 100ppm methane in nitrogen calibration gas using a "Tee" connection.

Make sure that there is a gas flow exiting from the open end of the "Tee". This insures that the SXC-20 sample pump inlet is "flooded" with the calibration standard gas.

With the calibration gas flowing, adjust the Detector Sensitivity "POT" (First pot on the left) so that the first (from the left) red LED remains lighted. Keep this configuration for several minutes, 6 to 10 times the sampling interval being used, to record the calibration on the data logger. Be careful not to turn any of the other adjusting pots as this would change sample flow rates and trigger level for the second pump.

Disconnect the gas line. Be sure to shut the cylinder valve on the calibration gas cylinder.

5. Close the SXC-20 case.

## OPERATION

1. Select a new glass enclosed carbon trap, break off the ends to open the tube and place in the clear plastic tube holder. Be sure to note gas direction arrow on trap, the flow direction is from the outside to the inside of the SXC-20, and place the tube in the correct orientation.
2. Position the unit for desired sampling. Attach an adapter if a long run time is anticipated or the battery charge level is low.
3. Turn on second pump enable switch, on front of the SXC-20. The unit is now in full operation.

## SHUTDOWN

1. At the end of the sampling period, remove the SXC-20 from its sampling port. Do not turn the unit off.
2. If the second sampling pump was enabled, turn it off at this time. Remove the carbon trap tube, cap it tightly, and label it with date and run identifier.
3. Attach the unit to the calibration gas cylinder and repeat the process for calibration. It is not necessary to open the unit. Allow the unit to "read" the calibration gas for several minutes, use the same time period as for calibration.
4. Reattach the SXC-20 to the computer and access the Bxcpro program. Double click the download toolbar icon and follow the instructions. Save the file. Print the graph if desired. Save the data in an appropriately labeled file in the SXC-20 Data folder.
5. Detach from the Digital computer, turn off the SXC-20 and connect the unit to its battery charger.

DONE