

Application Note

Static Sampling with the EGM-4 Using Our Sample Injection Kit

This application note outlines steps and procedures required to measure CO₂ concentration in syringe sample using the EGM-4 Environmental Gas Monitor.

Equipment Required

- Any model EGM-4 Environmental Gas Monitor running software version 1.54 and above.
- ACS037 Sample Injection Kit
- A Syringe with needle size ≤ 18 gauge (User supplied)

Measurement principle

The concentration of gas inside a syringe is measured by integrating CO_2 over a period of time (area under the curve) as the EGM-4 runs in a flow-through manner.

Before starting, you will need to know the syringe volume and the flow rate. (To obtain an accurate measurement of the flow rate, please refer to the Calibration section on Page 7.) During the measurement, CO₂-free gas is first passed through the EGM-4 to establish a baseline, and the sample is injected and CO₂ concentrations during this measurement phase are integrated. The final concentration in the syringe is calculated as:

$$CO_{2 INT} = \sum (CO_{2 m} - CO_{2 b}) \cdot \frac{\Delta t}{60} \cdot \frac{F}{V}$$

Where

 $CO_{2 \text{ INT}}(\text{ppm}) = \text{calculated } CO_2 \text{ concentration inside syringe ("integrated" } CO_2)$ $CO_{2 \text{ b}}(\text{ppm}) = \text{baseline } CO_2 \text{ readings before the measurement phase (averaged over 10 readings)}$ $CO_{2 \text{ m}}(\text{ppm}) = CO_2 \text{ readings during the measurement phase}$ $\Delta t \text{ (s)} = \text{sample interval, which is fixed at 1.6 sec per recording in EGM-4}$ $F \text{ (ml min}^{-1}) = \text{flow rate}$ V (ml) = syringe volume

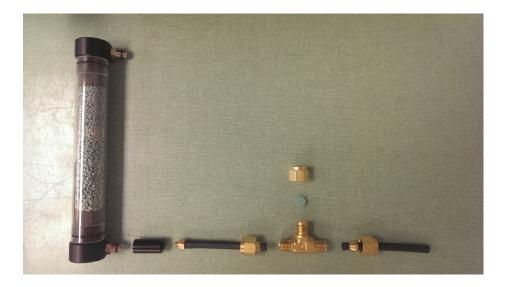
The 60 in the formula converts seconds to minutes.

To use the EGM-4 in injection mode, an injection kit accessory is needed (part number ACS037). This kit includes all the parts of the injection port assembled and leak tested, with 4 spare septa, as well as a soda lime column that provides the CO_2 -free baseline gas. The actual soda lime is not included and needed to be provided by the user.

Assembly

The ACS037 kit comes pre-assembled. If needed, the individual components can be taken apart and re-assembled as shown in the following pictures.

Parts



Assembly

Most connections are screwed on hand-tight. The soda lime column should be pushed on to the adaptor that includes an o-ring for sealing.



Assembled





The injection port includes a 9mm low-bleed septum that could take up to 50 injections, according to manufacturer certification. For best results, we recommend changing the septum every 25 injections.

The septum has a guiding hole and accommodates needles up to 22 gauge. For best results, we recommend needles with a size ≤ 18 gauge. The volume of the syringe does not matter as much, but it should be at least a size where you could comfortably inject at least 3 seconds. For this reason alone, syringes with a volume > 10 mL (e.g. 10- and 25-mL syringes) are recommended. If available, we recommend glass syringes (e.g. typical GC syringes), but plastic (polypropylene) syringes will work as well.

The injection mode works best for samples with concentrations > 100 ppm. At low concentrations, it will work but measurement errors as a percentage gets significantly larger.

Measurement

1. Set up the injection port as described in the previous Assembly section. Install the exit tubing of the injection port onto the "Gas In" port (upstream) of the EGM-4. Clamp the soda lime column vertically onto a ring stand or set it in a tube holder and or so it does not move around during the measurement and remains vertical.



- 2. Set the low CO_2 alarm limit on EGM-4 to 0 to prevent the CO_2 READ TOO LOW messages from appearing. From the main menu press 2SET, then 6ALM, and then 1 to adjust the low CO_2 limit. Enter 0, then press Y, Y to return to the main menu.
- 3. If the Probe type has not been set, set the probe type to 13 (Injection Mode) by going to the main menu, then choose 2SET, then 1EGM, then press N until "??" appears before: PROBE, then enter 13. Press Y to return to the 2SET menu, Press Y to return to the main menu.
- 4. Press 1REC to start an injection sampling run. The EGM-4 may take some time to warm up and do its auto-zeroing.
- 5. INJECTION DATA | 1ALL 2END: Press 1 to store every data point to internal memory, or press 2 to only store the final reading after the injection run is complete.



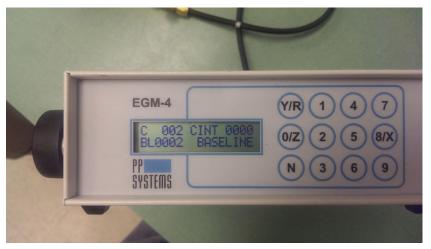
F/V: XX.XXX | 1=CAL 2=INPUT: F/V is the flow rate of the EGM-4 sample inlet (F in mL min⁻¹) divided by the volume of the syringe (V in mL). If the F/V value is correct, press Y to continue. If you know the correct value from previous testing, press 2 to input F/V manually. Flow rate of each EGM-4 is factory set to approximately 350 mL min⁻¹, but for best accuracy it is recommended that the actual flow rate be measured with an independent flow meter. Better yet, we recommend calibrating F/V using a "span gas" with a known CO₂ concentration. If you want to use the built-in calibration feature to compute a new F/V value press 1 (see calibration section at the end).

6. PLOT NO = 0 | Y OR NEW VALUE: Press Y to accept the default plot number or press number to change it.

This is also the "Ready" screen: pressing Y will now start sampling. Before you press Y, take your syringe sample (from e.g. your chamber or airbag) if you have not done so already. As with all good injection practices, it is always advisable (when possible) to take a little more than you need, and then push to the desired volume. For example, to take a 10mL sample, you should withdraw say 11 mL from your chamber, and then slowly push to the 10mL mark.

7. Baseline measurement: C 000 CINT 0000 BL0001 BASELINE

For the next 10 readings (16 seconds), the EGM-4 will record the baseline reading, which is the CO_2 concentration of the background. The "C" displays the current CO_2 concentration in the tubing, and BL displays the average baseline CO_2 concentration ($CO_{2 b}$ in the formula on page 1). Both "C" and BL will update in real time.



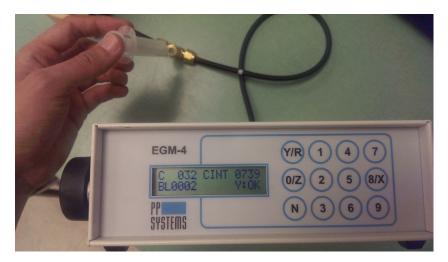
Simply **wait** during this phase. After 16 seconds, the average baseline concentration is displayed, for example BL0001, and the Injection phase begins.

If the baseline does not go down to 0 or a very small number (typically 1-3), then either there is a leak in the system, or the "zero" airflow has not been established. If it is the latter, go back to previous step, wait 2 - 3 minutes, and try again.

8. Injection phase:

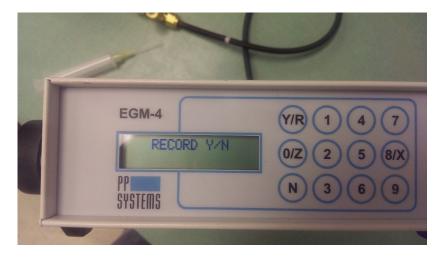
During this phase, the syringe should be slowly and steadily injected into the airstream through the septum. The injection speed should not exceed 3 mL min⁻¹ to avoid over-pressurization of the system (in other words, it should take you at least 3 seconds to inject a 10 mL syringe).

The "C" displays the current CO_2 concentration in the tubing. "CINT" displays the CO_2 concentration inside the syringe in ppm as calculated by integration ($CO_{2 \text{ INT}}$ in the formula on page 1). Both C and CINT will update in real time as the injection goes.



Typically, during an injection, "C" will go up quickly and drop back to zero, correspondingly CINT will increase quickly as the "C" peaks and then plateaus out at a constant value. Once "C" has dropped to 0 (or a very low background value), wait 3 - 5 seconds to make sure CINT concentration has stabilized, and then immediately press Y to end recording.

9. RECORD Y/N: Press Y to record the final readings into internal memory.



10. Make additional measurements as necessary, by repeating steps 5 to 9. Errors from injection measurements are typically larger than flow-through methods (larger errors could come from both instrumental and handling sources), so when possible it is always good to do repeats and take the average value.

Calibration

The flow rate on EGM-4 is preset to 350 mL min⁻¹ (\pm 25 mL min⁻¹), but its exact value also depends on the flow restriction of anything plumbed to the gas inlet and gas outlet ports. The following procedure outlines steps to calibrate the flow rate in order to enter a correct "F/V" value. Typically, the instrument only needs to be calibrated once every 3 months. Once calibrated, the calibration function is more like a diagnostics to make sure the pump is still running at the previous speed. If there are no major deviations it may not be necessary to change F/V.

There are three options for flow rate calibration:

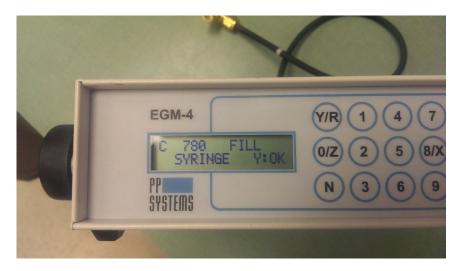
A) Measure flow rate with a flow meter

Measure the flow rate by connecting an independent flow meter in line with the EGM-4. Compute F/V manually and enter it directly at Step 5 in the measurement section.

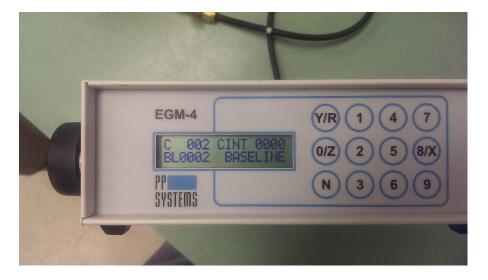
B) Calibrate flow rate with a span gas of known concentration by using the built-in calibration (recommended)

The procedures are as follows (we will use a 400 ppm span gas as an example):

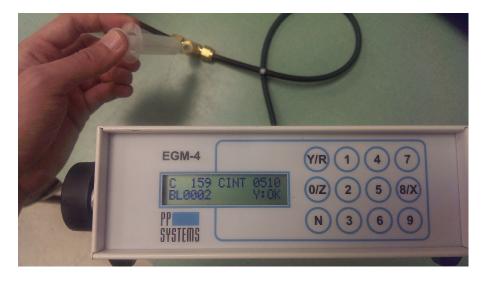
- 1. Set up the injection port as described in the previous Assembly section. Install the exit tubing of the injection port onto the "Gas In" port (upstream) of the EGM-4.
- 2. Begin the calibration routine by pressing 1CAL from the F/V menu (Step 5 in the Measurement Section above).
- 3. Ignore what is on this screen and press Y to continue.



- 4. Press N to change the concentration, and press in the concentration of a span gas (400 ppm). At this step, withdraw a syringe sample of the span gas, and then press Y to continue.
- 5. The EGM-4 now begins a 16 second baseline measurement period.



6. When the display changes to Y:OK, slowly inject the calibration gas and observe the CINT value rise. When you are done injecting, wait for the C00000 to return to the baseline level, then hit Y to stop the measurement.



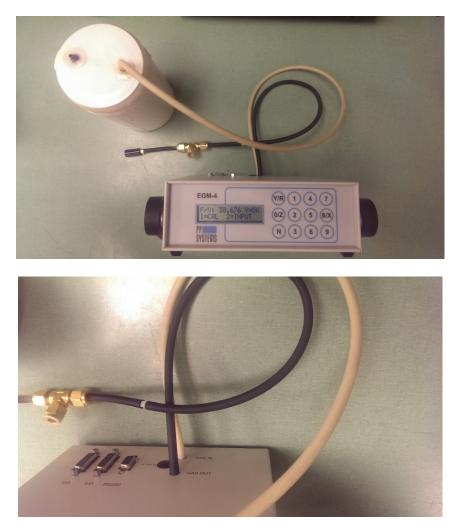
The last displays shows the calibration concentration C xxxx, the calculated concentration CINTxxxx with the original F/V factor, and the new F/V factor to get the calculated concentration to equal the calibration concentration. Press Y to accept and use the new F/V, or press N to ignore the new F/V and continue using the original F/V.

C) Calibrate flow rate using buffered room air as a span gas (this is used when a span gas of known concentration is not readily available)

The idea here is to use EGM-4 to measure ambient air in a flow-through manner, and then use the ambient air as a span gas. A large mixing volume is needed. The procedures are as follows:

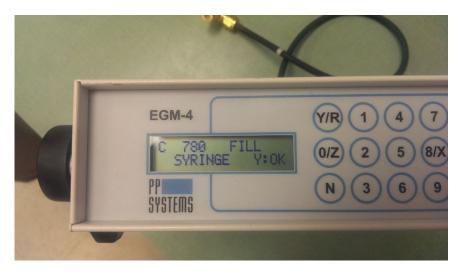
1. Set up the injection port as described in the previous Assembly section, but without the soda lime column. This injection port (the tee) is now being used as a sampling port. Install the injection setup on to the "Gas **Out**" port (downstream) on the EGM-4.

Install a large mixing volume on the "Gas In" (upstream) side of the EGM-4. The mixing volume needs to be at least ~ 1L to smooth out variations in CO_2 concentrations in room air. Place the inlet of the mixing volume away from the operator (or any other humans nearby), ideally in a fume hood with the sash closed all the way down. The ideal tubing length is about 0.25 - 0.5 m, or just enough length to comfortably operate but at the same time creating minimal backpressure.



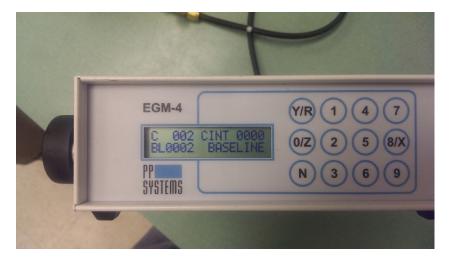
- 2. Begin the calibration routine by pressing 1CAL from the F/V menu (Step 5 in the Measurement Section above).
- 3. The EGM-4 now displays the measured CO_2 concentration of the gas inlet (with averaging enabled). Observe the CO_2 concentration until it has stabilized. Slowly and steadily, draw an air sample from the tee. The speed at which you draw should not exceed 3 ml sec⁻¹, or else there is the possibility of drawing in unbuffered and

unknown gas from the exhaust. Once again, draw a little more than you need, and then adjust to the right volume.

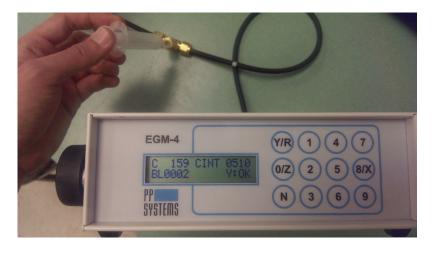


Our room air shown above is somewhat CO_2 -rich, but it was stable. At this point, you have a syringe full of calibration gas of a known concentration. Close the cap on the syringe needle to minimize diffusion.

- 4. Press Y to continue. Check to make sure that the concentration displayed on this screen is reasonable.
- 5. Disconnect the mixing volume on the "Gas In" port on EGM-4, and now re-plumb the injection port (the tee) back to the "Gas In" port, with the soda lime column attached (i.e. back to the Measurement setup). Once this is set up, wait approximately 60 seconds for the line to clear. Press Y to accept the calibration concentration and continue.
- 6. The EGM-4 now begins a 16 second baseline measurement period.



7. When the display changes to Y:OK, slowly inject the calibration gas and observe the CINT value rise. When you are done injecting, wait for the C00000 to return to the baseline level, then hit Y to stop the measurement.



The last displays shows the calibration concentration C xxxx, the calculated concentration CINTxxxx with the original F/V factor, and the new F/V factor to get the calculated concentration to equal the calibration concentration. Press Y to accept and use the new F/V, or press N to ignore the new F/V and continue using the original F/V.

Cheat Sheet

- 1. Set CO_2 alarm to 0
- 2. Set Probe type to 13
- 3. Press 1Rec to start a run
- 4. Calibrate F/V as needed
- 5. PLOT screen: Get ready, take your sample
- 6. BASELINE screen: Wait
- 7. INJECTION: Inject slowly and steadily over at least 3 seconds
- 8. CINT is the CO₂ concentration in your syringe
- 9. Press Y to record

If you would like to learn more about this application or would like to speak with one of our experienced technical staff, please feel free to get in direct contact with us at:

PP Systems 110 Haverhill Road, Suite 301 Amesbury, MA 01913 U.S.A.

Tel: +1 978-834-0505 Fax: +1 978-834-0545

Email: <u>support@ppsystems.com</u> URL: <u>www.ppsystems.com</u>