

CALIBRATION OF CO₂ ANALYZERS

Every gas measuring instrument requires a method for calibration or verification. There are several means for calibrating a CO₂ analyzer. The easiest way is a gas calibration: pure CO₂ gas at a known pressure is injected on the sensor membrane. This is a perfect method if the unknown sample is a gas. However, if the analyzed sample is a liquid, a slight deviation from the actual value can be noticed: Even though the CO₂ sensor measures the CO₂ partial pressure in the liquids, the presence of the liquid can have some effect on the temperature of the sensitive membrane and on the access of CO₂ molecules to the membrane. In practice, the CO₂ measurements in liquid will indicate 1 or 2% lower CO₂ level than expected after a gas phase calibration. One way to prevent this problem is to calibrate in the liquid phase, that's why Hach has developed the Orbisphere CO₂ calibration kits.

Orbisphere CO₂ Calibration Kits

The aim of this kit is to provide the end user with an accurate and easy-to-use liquid calibration method. A known and precise amount of sodium bicarbonate powder is combined in a precise PET bottle, with a ready-made dilute citric acid solution to provide defined, accurate carbon dioxide content in the liquid. During the calibration procedure, this solution can then be circulated through the flow chamber of a thermal conductivity sensor.

The dose of sodium bicarbonate powder is filled into a plastic vial with repeatability better than 0.3%. Final packages are controlled statistically and the average weight as well as the standard deviation for a batch can be guaranteed. The liquid content is also precisely under control, since the chemicals are supplied ready for use. The precise PET bottles for the preparation of the test sample have shown variability on their volume under 0.1% during quality checks. This is mainly due to the use of a single mold.

The expansion coefficient with temperature and with pressure has also been measured and taken into account. The combination of the previous elements will provide a traceable test sample with a unique accuracy of +/- 0.5%. This is an acceptable condition in order to calibrate or verify our analyzer with a specified accuracy of 1%.

Most CO₂ analyzers, including Orbisphere, measure the partial pressure of the CO₂. Since the solubility of CO₂ is different in pure water and in sodium citrate solution, some corrective factor should be applied to the theoretical calculation.



Hach has measured the solubility of CO₂ in the final calibration solution at different temperatures and computed correction tables for various analyzer settings (patent pending). The calibration tables will be delivered in the starter kit and will tell the user which number to enter in their instrument during calibration or which value they should read on their display during a verification, depending on analyzer settings.

APPLICATION: CALIBRATION OF CO₂ ANALYZER

Example:

Select column according to instrument unit setting			
Tem. (°C)	V/V in Water	V/V in Beer	V/V in Cola
6	3.353	3.149	3.266
8	3.332	3.151	3.275
10	3.310	3.150	3.284
12	3.289	3.145	3.292
14	3.268	3.137	3.300
16	3.248	3.126	3.307
18	3.227	3.112	3.313
20	3.207	3.096	3.318
22	3.187	3.076	3.321
24	3.167	3.054	3.323
26	3.147	3.029	3.323
28	3.127	3.002	3.320
30	3.108	2.973	3.315
32	3.088	2.942	3.308
34	3.069	2.908	3.298
36	3.050	2.873	3.286
38	3.031	2.836	3.271
40	3.012	2.798	3.252

At 25 °C (77 °F) in sodium citrate liquid, the CO₂ concentration computed from known chemical quantities, and volumes will be 3.038 V/V or 5.861 g/kg (equilibrium pressure: pCO₂ = 4.208 bar).

For an analyzer configured to measure CO₂ in beer, the concentration corresponding to this pressure at same temperature will be 3.042 V/V.

For the instrument set for CO₂ in water, the value will be 3.157 V/V and 3.323 V/V for cola units.

How to Use the Calibration Kit:

A "starter kit" containing all the items required for preparing 10 samples (including the calibration bottle), and a "refill kit" containing 10 spare samples (no bottle) are available with two carbonation levels.

Preparation Steps:

- The dilute citric acid is poured in the calibration bottle.
- Its temperature measured roughly (the effect of the temperature is not highly critical and a 2 °C error in temperature will not generate more than a 0.5% error on the CO₂ concentration). The table has temperature increments of 2 °C and a coarse linear interpolation between these values will further reduce the error.
- The adaptor piece is screwed on the calibration bottle, and the bicarbonate test tube is gently inserted through the adapter making sure the tube floats on the liquid.
- The bottle is then tightly closed. At this time, the bottle can be tilted bottom up and vigorously shaken. The goal is to make sure no bicarbonate powder is left in the tube.
- The bottle can then be left sitting on a table for 3 minutes or more (intermittent shaking would speed up the process). Once all the powder is dissolved, the bottle can be shaken for two or three strokes just before use.
- The calibration sample has to be used the same day it was prepared, preferably within one hour. A CO₂ loss of about 1% per day has been typically observed..

The analyzer settings should be clearly identified in order to use the suitable column of the conversion table.

As with any other sample, the piercing process is critical for a reliable measurement..

Before piercing, we have to make sure the forcing gas pressure is higher than the sample pressure (depends on the temperature: about 5 bar forcing pressure at 25 °C). Also make sure the flow chamber exit is closed. During the piercing step, make sure there are no leaks around the needle and no degassing in the bottle. In case there is a burst of bubble in the bottle at that time, do not attempt to calibrate the analyzer. Prepare another calibration sample instead.



Systems Components:

Model	Description
32313	CO ₂ calibration: starter kit, high level. Calibration bottle + adapter + reagents from model 32314.
32314	Refill kit. High level. Reagents to prepare a reference solution to calibrate CO ₂ analyzers. High carbonation 3 to 8 g/kg.
32315	CO ₂ calibration: starter kit, low level. Calibration bottle + adapter + reagents from model 32316.
32316	Refill kit. Low level. Reagents to prepare a reference solution to calibrate CO ₂ analyzers. Low carbonation 0 to 3 g/kg.

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