

Calibration of a GC system for the quantification of CO₂ capture in aqueous solutions of alcanolamine

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Introduction

MINES ParisTech is one of France's oldest higher education institutions. While its inspirational model has not changed over the years, its teaching methods and research have been at the leading edge since 1783. Two Nobel Laureates trained at the School: Georges Charpak (Physics, 1992) and Maurice Allais (Economics, 1988).

MINES ParisTech is established in five major fields: Earth sciences and the environment; Energy and processes; Mechanical and materials engineering; Mathematics and systems; Economics, management and society.

The Center of Energy and Processes (CEP) studies the transformation of matter and energy in many fields, focussing on complex energy systems, particularly in transient conditions, and on controlling their emissions.

The Thermodynamique des équilibres entre phases (TEP) laboratory specializes in the measurement of thermodynamic properties, providing industry with specialists skilled in both experimental and theoretical aspects of handling fundamental and applied works.

Description

A key area of study for the TEP laboratory is the measurement of partition coefficients (ELV) of mercaptans for aqueous amines in the presence of carbon dioxide and/or hydrogen sulfide.

The release of CO_2 is measured using a GC method with a thermal conductivity detector (TCD). A calibration regime is essential because of the quantitative method of analysis. Samples may be either liquid or gaseous.

Calibrations are required before, during and after the testing process which takes place over several months. The accuracy and precision of the calibrations are critical.

The GC system is calibrated by injecting a known volume of CO₂ aspirated directly from a gas bottle through a septum. This is done with manual gas tight syringes and is operator dependent, often resulting in inaccurate measurements.

To reduce discrepancies a magnifying tool (see figure 1) was developed to improve the accuracy, precision and consistency of syringe use by different operators. Even with the magnifying tool, the calibration process remains time consuming and operator dependent.



Figure 1. Magnifying tool developed by MINES ParisTech

To address these methodology issues, the CEP TEP laboratory has evaluated and now use eVol®, the world's first digital analytical syringe.

Method

eVol is used to collect CO_2 from a gas bottle through a regulator equipped with a septum port. Using the 50 µL and 500 µL eVol syringes, volumes of gas are aspirated and injected into the GC system injection port. The CO_2 concentration is directly proportional to the sample volume. It elutes on a packed column in less than 50 seconds with levels detected by a TCD.

Each time the total aspirated volume is injected. Increasing volumes of aspirated sample are successively injected to set up the calibration curve with the test for each volume repeated several times.

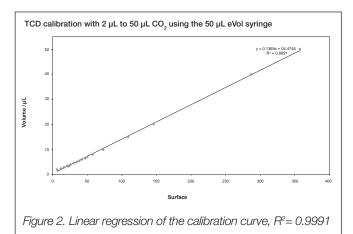
Tests are performed in a stable and controlled environment at 23°C and an atmospheric pressure of 1006.3 mbar (100.63 kPa).

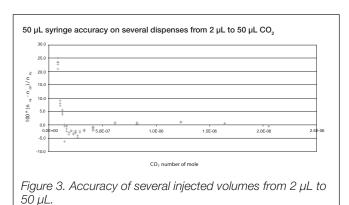
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Results

Calibration using 50 µL eVol syringe

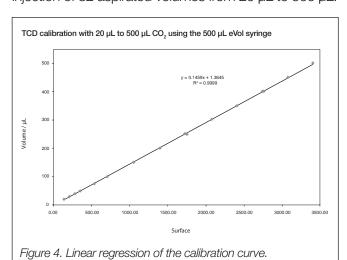
Injection of 64 aspirated volumes from 2 µL to 50 µL.





Calibration using 500 µL eVol syringe

Injection of 82 aspirated volumes from 20 µL to 500 µL.



 $500~\mu L$ eVol syringe accuracy on several dispenses from 20 μL to $500~\mu L$

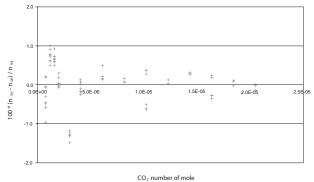


Figure 5. Accuracy of several injected volumes from 20 μ L to 500 μ L.

Discussion

The accuracy of low volumes (2 μ L and 2.5 μ L) using the eVol 50 μ L syringe is affected by Helium back pressure, which causes a dilution of CO₂ during the injection process.

The dispersion observed for low volumes using the 50 μ L syringe and reported in figure 2 is not observed in figure 4 when the 500 μ L syringe is used.

Conclusions

Using eVol for injection of different volumes of CO₂, the CEP TEP laboratory was able to achieve calibration of their instrument with levels of accuracy and precision not previously obtained using manual syringes.

In addition, the needles on the eVol syringes allow for sample collection directly from the gas bottle – similar to the manual syringes used previously.

Using the eVol, the CEP TEP laboratory increased the accuracy of their calibration while reducing the handling time involved in this process.

Information and support

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Specifications are subject to change without notice.