Calibration of a GC System for the Quantification of CO_2 Capture in Aqueous Solutions of Alcanolamine

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http://www.mines-paristech.eu/Researcher/Fields-of-Research/Energy-and-process-engineering/CEP/

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 process engineering; Mechanical engineering and materials; Mathematics and systems; Economics, management and society.

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

The TEP laboratory specializes in the measurement of thermodynamic properties http:/ www.fcep.mines-paristech.fr/Accueil_Eng.htm. This laboratory, "Thermodynamique des équilibres entre phases", provides industry with specialists skilled in both experimental and theoretical aspects of handling fundamental and applied works successfully.

DESCRIPTION

A key area of study for the TEP lab 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 TCD detector. 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 CO_2 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 was developed to improve the accuracy, precision and consistency of syringe use by different operators. Even with the new tool the calibration process remains time consuming and operator dependent.

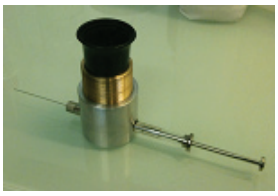

Figure 1. Magnifying tool developed to improve the accuracy, precision and consistency of syringe use.

To address these methodology issues, CEP/TEP lab have 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® XCHANGE® 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 detector.

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.

RESULTS

Calibration using eVol® 50 µL syringe

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

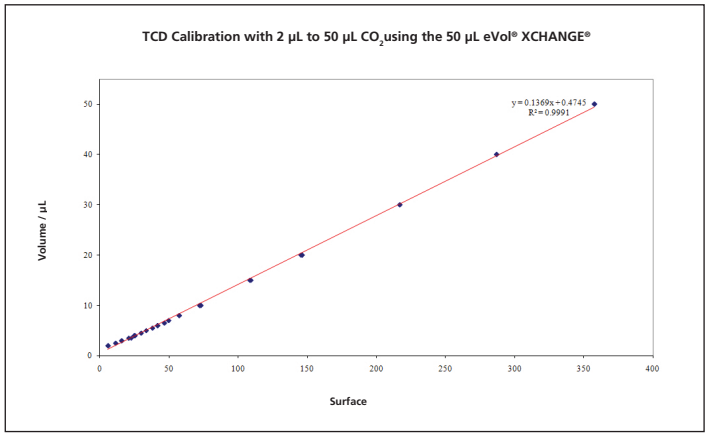

Figure 2. Linear regression of the calibration curve, R^2= 0.9991

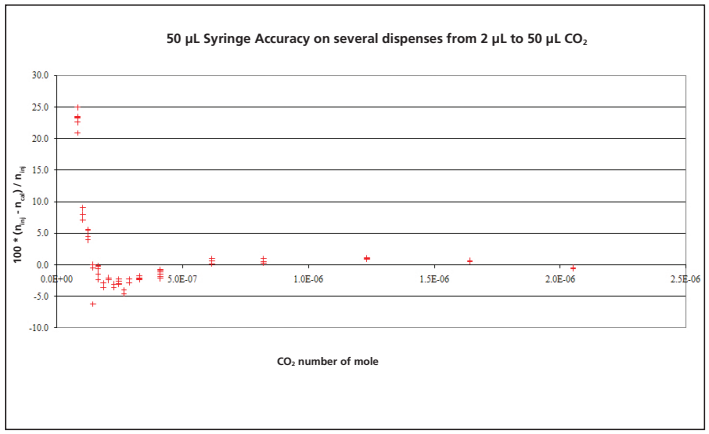

Figure 3. Accuracy of several injected volumes from 2 µL to 50 µL.

Calibration using the 500 µL syringe

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

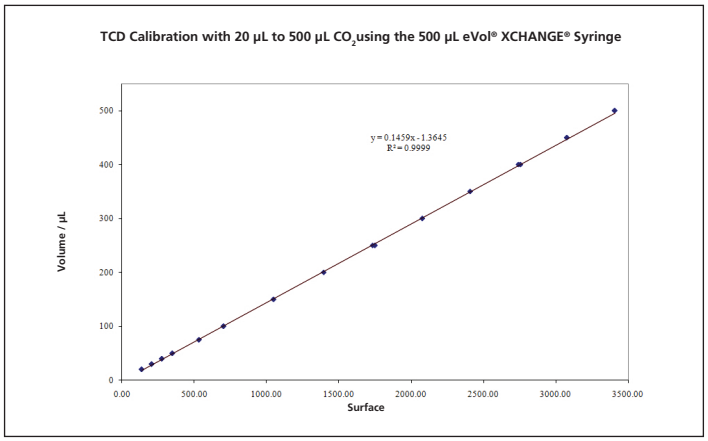

Figure 4. Linear regression of the calibration curve.

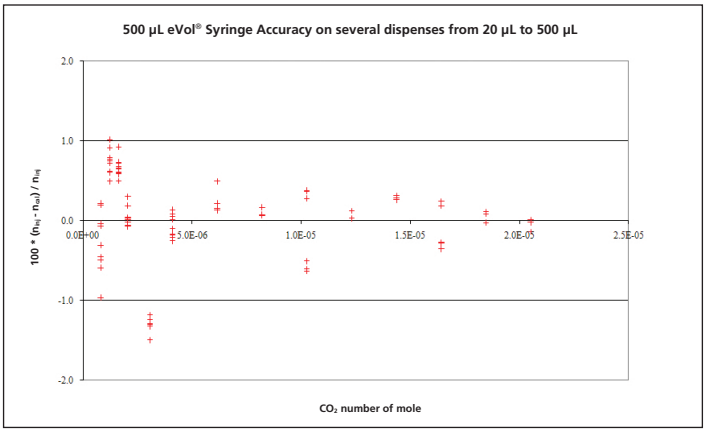

Figure 5. Accuracy of several injected volumes from 20 µL to 500 µL.

DISCUSSION

The accuracy of low volumes (2.00 µL and 2.5 µL) using the eVol® 50 µL syringe is affected by Helium back pressure, which causes a dilution of CO_2 during the injection process.

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

CONCLUSIONS

Using eVol® for injection of different volumes of CO_2, CEP/TEP 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 – similarly to the manual syringes used previously.

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

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