21GRD06 MetCCUS

# A3.3.6. Report on new methods for the chemical testing of capture solvents including purity, stability and $CO_2$ uptake to support European industries in understanding the degradation and purity of $CO_2$ and key impurities and ensuring efficient operation and utilisation of $CO_2$

## D6 – A3.3.6

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**Due date of the deliverable:** June 2025

**Actual submission date of the deliverable:** September 2025

**Confidentiality Status:** PU - Public, fully open (remember to deposit public deliverables in a trusted repository)

The project has received funding from the European Partnership on Metrology, co-financed from the European Union's Horizon Europe Research and Innovation Programme and by the Participating States.

**Note:**

**This report will describe a method to quantify the $CO_2$ capture efficiency of solid sorbents with CaO as a benchmark sorbent under post combustion capture conditions. Traceable primary reference gas mixtures were used in these experiments to ensure that the quantification was accurate. The effect of impurities and amount of $CO_2$ in the flue gas was assessed by varying the composition of the PRMs used for the tests. An uncertainty budget was developed to include all uncertainties involved in the measured using GUM. The initial objective of the activity to test solvents was not tested as the rig available to do the testing could not handle any solvents. Therefore, a change request was placed and approved to test solid sorbents for quantifying carbonation efficiency. The deliverable was successfully achieved for testing carbonation efficiency of solid sorbents.**

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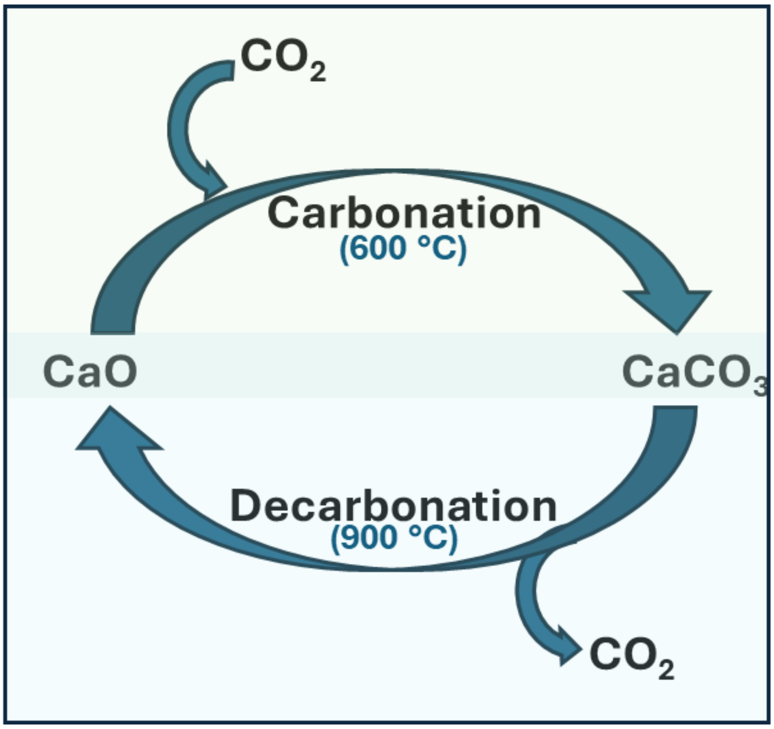
## Introduction

Carbon capture utilization and storage is a key driver to facilitate decarbonization strategies and for meeting 'Net-Zero' objectives by 2050 [1-2]. To provide a safe, sustainable cost-effective method of capturing carbon dioxide from various industrial processes, both solid and liquid capture agents are used [2]. These capture materials have their pros and cons relative to one another. However, it is important to assess the performance of these materials to select suitable candidates for $CO_2$ capture at industrial scale. There is a need for getting better insights into these materials including accurate capture capacity, homogeneity, stability, activity decay, repeatability and emissions/degradation products. Performance testing/evaluation of carbonation efficiency of various $CO_2$ capture materials and understanding the degradation products under real process conditions has a significant impact on the development of next generation capture technologies. However, there are no standardized and traceable methods to access these capture materials under relevant capture conditions. Therefore, there is a need to develop a metrology infrastructure using traceable Primary Reference Materials (PRMs) to validate various capture materials that are being developed simultaneously to tackle climate change. Under activity - A3.3.5 of the MetCCUS project, NPL's Energy Gas Metrology Group has been working towards developing a traceable capture efficiency testing protocol for $CO_2$ capture materials by using CaO as a benchmark material. NPL's traceable PRMs were used to assess the capture efficiency of CaO at this stage and the measurement methods developed will be extended to assessing carbon capture capacity of different $CO_2$ capture materials evolving in the market against CaO benchmark. The NPL's materials testing platform (MTP) consists of a micro-reactor coupled with an online monitoring system that records real-time data from flue gas (NPL PRMs) interaction with the capture materials. The micro-rector mimics a fixed bed reactor that can hold solid sorbents starting from few milligrams to 100 g scale. Liquids $CO_2$ capture solvents can also be evaluated by swapping the solid sorbent reactor with a reactor vessel that can handle liquids.

CaO is a solid sorbent capable of $CO_2$ uptake and is often used as a benchmark sorbent for $CO_2$ capture materials. The chemistry of CaO sorbents during carbonation and decarbonation reactions is well known. CaO chemically combines with $CO_2$ at around 600 °C to form $CaCO_3$ stoichiometrically. The CaO sorbent is regenerated by thermal decomposition of $CaCO_3$ at around 900 °C to give back CaO and $CO_2$ as shown in schematic 1 below. According to a recent report, at high temperatures (~ 600 °C), a uniform layer of growth of $CaCO_3$ occurs over CaO due to chemical reaction between CaO and $CO_2$ and was found to be effected by the concentration of $CO_2$ [3]. Therefore, different $CO_2$ concentration - controlled study would shed more light on parameters such as capture efficiency / stability of CaO sorbent under different $CO_2$ stream. On repeated cycling, CaO undergoes agglomeration which leads to loss in carbonation efficiency. Various efforts are made in the recent past to improve the stability and efficiency of CaO through chemical and structural modifications. Carbon-supported and isomorphous substituted CaO based hybrid sorbent reported recently showed an increased carbonation efficiency and stability of the sorbent in comparision to pure CaO. This hybrid solid sorbent had around 65 % of its initial carbonation efficiency retained after 100 carbonation – regeneration cycles in contrast to the pristine CaO that lost 80 % of its initial carbonation efficiency after 10 carbonation – regeneration cycles [4].



Schematic 1. Schematic of the carbonation and decarbonation reactions of CaO sorbent

## Experimental

## Assessing HSE impact of the work

A detailed risk assessment was carried out for the proposed work and developed appropriate risk and COSHH documents and was approved from appropriate HSE authority at NPL. These are followed throughout various experiments done under this activity.

## Preparation and validation of PRMs/Calibration gas mixtures

PRMs needed to test the CaO sorbent was prepared in accordance with ISO6142-1:2015. Within this activity PRMs containing $CO_2$ (400 µmol/mol, 5 cmol/mol, 14 cmol/mol and 20 cmol/mol) in air or $N_2$ balance gas was prepared and validated. These PRMs were used for calibration, method development and testing carbonation efficiency of $CO_2$ sorbents. Effect of $CO_2$ concentration on carbonation efficiency of CaO was tested by using NPL PRMs containing 5 cmol/mol, 14 cmol/mol and 20 cmol/mol $CO_2$ in air or $N_2$ matrix.

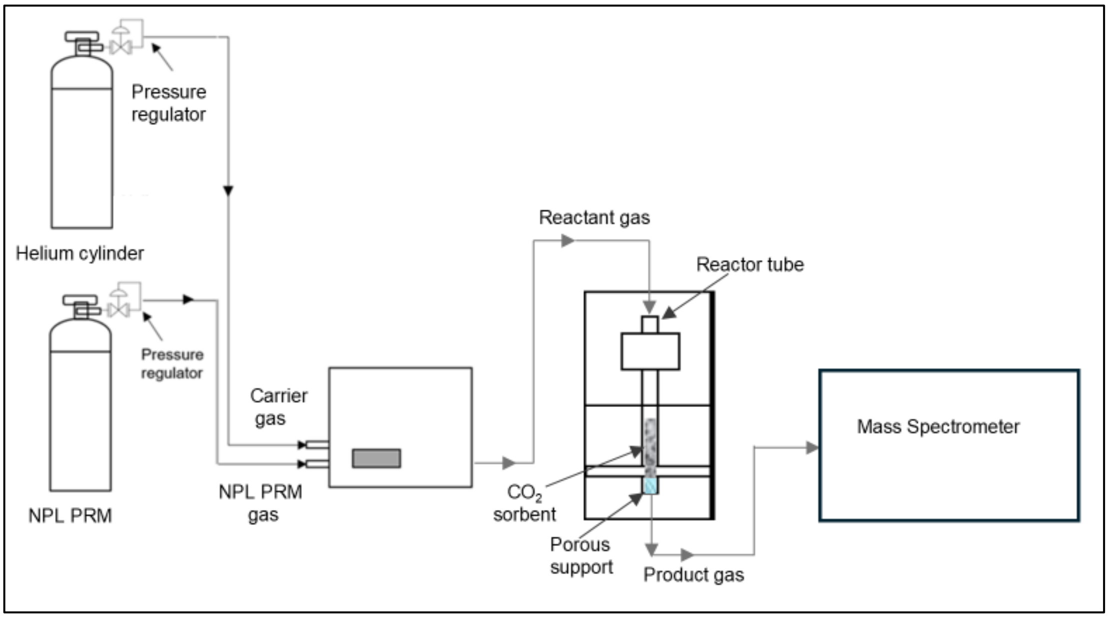


Figure 1. Schematic of NPL's MTP facility used to quantify carbonation efficiency of CaO using NPL PRMs

## Preparation of Primary reference materials and calibration gas

The PRMs were prepared in 10L, aluminium cylinder with spectraseal passivation (BOC, United Kingdom) and valve outlet, BS14. The preparation was done using the gravimetric method in accordance with ISO 6142-1:2015 [5] using high purity $CO_2$. ISO 6142-1:2015 is applicable to mixtures of gaseous or totally vaporized components added gravimetrically for the preparation of reference gas mixtures in cylinders with traceable values for the amount fraction of one or more components. Highest purity components that are commercially available were used, and purity analysis was performed to identify and quantify any impurities present. The compounds were added to the cylinder by direct transfer via a 1/16-inch external diameter stainless steel tubing and an NPL-designed outlet diaphragm valve (Rotarex Ceodeux, Luxembourg) that includes an internal screw thread to minimise dead volume. Where direct transfer was not possible, suitable transfer loops were used to transfer fixed amounts of gas into the cylinders via the NPL's diaphragm valve. The cylinders were weighed after each component addition was complete and the amounts recalculated to arrive at the target composition of the mixtures. The mixtures were heated and rolled in the horizontal position for two hours to achieve homogeneity of the PRM. The compositions of the mixtures (amount fraction and associated uncertainty of each compound) were calculated using the software package 'GravCalc2'. The PRMs were validated by using standard NPL methods before being used for the carbonation experiments. A set of calibration gases of the composition, 400 µmol/mol, 5 cmol/mol, 14 cmol/mol and 20 cmol/mol $CO_2$ in air or $N_2$ matrix were also prepared and validated by matching standard method by comparing with independent NPL standards of similar composition.

## Online monitoring of reaction

The interactions of $CO_2$ by CaO sorbent is generally measured by different ways, such as by monitoring mass change of the sorbent, concentration change in initial gas mixture using various techniques . Here, a residual gas analyser [6-7] combined with mass spectrometers procured from Hiden Analytical Ltd., UK has been used here for measuring the concentration of the product gas coming out of the reactor after the interaction of the NPL PRM with a known weight of CaO sorbent. Figure 1 illustrates a schematic of MTP facility at NPL used to quantify carbonation efficiency of CaO using NPL PRMs with different $CO_2$ amount fractions and different matrix gases (nitrogen and air).

Hiden Analytical HPR-20 quadrupole mass spectrometer was used for online, continuous gas analysis to monitor the composition of the gas during the carbonation experiments of CaO sorbent. The gaseous analyte is ionised via electron impact (EI) through thermionic emission of the filament. The ionised samples travel through a mass filter to select species for detection through a secondary electron multiplier (SEM) [8-9]. Calibration was carried out using NPL PRMs (400 µmol/mol, 5 cmol/mol, 14 cmol/mol and 20 cmol/mol $CO_2$ in $N_2$ matrix) and the calibrated response of the analyser for the PRMs was within a correlation coefficient of greater than 0.95. The data was recorded using the CATLAB / MASsoft software provided by Hiden Analytical Ltd., Warrington, UK. The instrument was used to monitor concentration variation of the product gas produced after interaction of NPL PRMs with CaO sorbent during entire course of the experiments.

## Experimental setup used for Carbonation Experiments

CaO procured from (Sigma-Aldrich, Gillingham, UK) and was used without further purification for all the experiments. 0.5 g of CaO sorbent was accurately weighed and transferred on to a silica wool bed (of known weight). The silica wool bed holds the solid sorbent in place and allows only the product gas to pass through the reactor tube. Figure 1 shows a schematic of the MTP facility used for the carbonation experiments in which the reactor is interfaced with an online monitoring system that records data from a mass spectrometer instrument (Figure 1). The microreactor was used as a chamber to facilitate the interaction of CaO sorbents with different concentration of $CO_2$ gas PRMs containing impurities as shown in the schematic (Figure 1). The product gas after interaction with the CaO exits the reactor to enter the mass spectrometer for analysis. The data was recorded on the CATLAB / MASsoft software. The direction of the flow of gases are indicated by the arrows in Figure 1.

## Experimental Setup and baseline tests

Baseline tests to check for adsorption of $CO_2$ in the absence of CaO sorbent was carried out using the PRMs used for capture tests. This amount of $CO_2$ adsorbed during the baseline studies was subtracted from the amount of $CO_2$ uptake from the CaO sorbent under similar conditions to arrive at the carbonation efficiency (capture capacity) of CaO. The partial pressure of $CO_2$ obtained from these measurements (p$CO_2$ background) were used to correct the amount fraction of $CO_2$ measured during the carbonation experiments (equation 1). Relative sensitivity factor, RS$CO_2$ is 1.38 (taken from the Hiden database) [10] due to the unique ionizability of $CO_2$ in the mass spectrometer. The amount fraction of $CO_2$ was determined through the partial pressure reading of $CO_2$ given by the mass spectrometer, with the equation for quantifying the amount fraction given in equation 1 [11].

$$Amount\ fraction\ \ of\ CO_2\ (cmol/mol) = \left( \frac{\left( \frac{p_{CO_2\ experimental} - p_{CO_2\ background}}{RS_{CO_2}} \right)}{p_{total}} \right) \times 100 \qquad (1)$$

$p_{CO2\ experimental}$ : partial pressure of $CO_2$ from the experiment.
$p_{CO2\ background}$ : partial pressure of $CO_2$ during the background experiment.
$RS_{CO2}$ : relative sensitivity of $CO_2$.
$p_{total}$ : partial pressure of all the gases in the analyte mixture.

The total uncertainty in the amount fraction of $CO_2$ (cmol/mol) was calculated by using the expression given in equation 2..

$$U_{Total} = \sqrt{\left(Up_{CO_2\ experimental}\right)^2 + (U\ p_{CO_2\ background})^2 + (Up_{total}\ )^2} \qquad (2)$$

$U_{Total}$ : total uncertainty of the amount fraction of $CO_2$.
$U_{pCO2\ experimental}$ : uncertainty in $p_{CO2\ experimental}$, *k*=2 (µmol/mol)
$U_{pCO2\ background}$ : uncertainty in $p_{CO2\ background}$, *k*=2 (µmol/mol)
$U_{p\ total}$ : uncertainty in $p_{total}$, *k*=2 (µmol/mol)

## Carbonation and decarbonation experiment design

Carbonation experiments were carried out using the setup shown in the schematic (Figure 1). Known amount of CaO (0.5 g) was weighed and used for the carbonation experiment as explained above. The sample was heated to 600 °C (20 °C/min ramp rate) under pure He gas flow (40 ml/min) before introducing $CO_2$ PRM for carbonation to remove any surface adsorbed species (water or $CO_2$) before the actual carbonation experiment. The following this, NPL PRM containing either 5, 14 or 20 cmol/mol of $CO_2$ was introduced (40 ml/min) and held at 600 °C for 30 minutes. Once the carbonation stage was over, gas flow was switched back to pure He and the temperature has raised to 900 °C (20 °C/min ramp rate) and holding it at that temperature for 30 min to regenerate/decarbonise the CaO sorbent. These temperatures were chosen for carbonation and decarbonation steps based of literature reports [12]. The holding time for both carbonation and regeneration step (30 min) has arrived by doing trial experiments to see when sample reaches complete carbonation/regeneration. The outlet of the micro reactor is connected mass spectrometer and gas coming off during both carbonation and decarbonation steps was continuously analysed and the data was recorded using the CATLAB /MASsoft software as described earlier. Since the interaction of gases with reactor, tubing and associated parts could give rise to errors in the observed values by interfering with the carbonation/decarbonation reactions, a bank test (baseline test) with empty reactor was undertaken to account for any variations in the measured values under each experimental condition prior to every experiment. Calibration of mass spectrometer was carried with at least 4 different PRMs having varying amount fractions of $CO_2$ every time prior to the experiment. The silica wool that was used to hold the CaO sorbent was kept close to 17 mg for every experiment and blank to ensure the interference due to silica wool is maintained across all trials. Each experiment was repeated two times to check for reproducibility within the experimental conditions summarised in Table 1.

Table 1. Summary of the different experimental conditions used to test carbonation and decarbonation reactions of CaO sorbent with NPL PRMs

| Sl. No | Concentration of NPL PRM in $N_2$ | Amount of CaO (g) | Carbonation Temperature (°C) | Decarbonation Temperature (°C) | Flow of NPL PRM (ml/min) | Number of repeats |
|---|---|---|---|---|---|---|
| 1 | 20 cmol/mol | 0.0 - Blank | 600 | 900 | 40 | 2 |
| 2 | 20 cmol/mol | 0.5 | 600 | 900 | 40 | 2 |
| 3 | 14 cmol/mol | 0.0 - Blank | 600 | 900 | 40 | 2 |
| 4 | 14 cmol/mol | 0.5 | 600 | 900 | 40 | 2 |
| 5 | 5 cmol/mol | 0.0 – Blank | 600 | 900 | 40 | 2 |
| 6 | 5 cmol/mol | 0.5 | 600 | 900 | 40 | 2 |

## 2.8 Calculation of carbonation efficiency/adsorption capacity by Calcium Oxide

The carbonation efficiency/absorption capacity expressed in grams of $CO_2$ per gram of CaO on interaction with NPL PRMs at 600 °C was calculated using equation 3 [13-14] and the total uncertainty involved was calculated using equation 4.

$$A_c = \frac{PM}{RTm} \int_{t_i}^{t_f} c_o Q_o - c_i Q_i \, dt \qquad (3)$$

Where;
$A_c$= absorption capacity of CaO,
$P$ = pressure of system ($P$ = 1 bar)
$M$ = molecular weight of $CO_2$ ($M$ = 44.009 g/mol)
$R$ = molar gas constant
$m$ = mass of CaO taken for reaction
$T$ = temperature of reaction

$t_f$ = time at the end of the experiment
$t_i$ = initial time
$c_o$ = outlet gas concentration (taken to be the concentration of gas analysed after the reaction with CaO)
$Q_o$ = outlet gas volumetric flow rate (taken to be the flow rate of gas after the reaction with CaO)
$c_i$ = inlet gas concentration (taken to be the concentration of gas from the water baseline)
$Q_i$ = inlet gas volumetric flow rate (taken to be the flow rate of gas from the water baseline)

The total uncertainty in is given by

$$U_{total} = A_c\sqrt{\left(\frac{U_P}{P}\right)^2 + \left(\frac{U_m}{m}\right)^2 + \left(\frac{U_T}{T}\right)^2 + \left(\frac{U_{c_o}}{c_o}\right)^2 + \left(\frac{U_{c_i}}{c_i}\right)^2 + \left(\frac{U_{Q_o}}{Q_o}\right)^2 + \left(\frac{U_{Q_i}}{Q_i}\right)^2} \qquad (4)$$

Where;
$U_P$ : uncertainty in pressure of system ($P$ = 1 bar)
$U_m$ : uncertainty in mass of CaO within reactor
$U_T$ : uncertainty in temperature of reaction
$U_{c_o}$ : uncertainty in outlet gas concentration (taken to be the concentration of gas analysed after the reaction with CaO)
$U_{Q_o}$ : uncertainty in outlet gas volumetric flow rate (taken to be the flow rate of gas after the reaction with CaO)
$U_{c_i}$ : uncertainty in inlet gas concentration (taken to be the concentration of gas from the empty reactor)
$U_{Q_i}$ : uncertainty in inlet gas volumetric flow rate (taken to be the flow rate of gas from the empty reactor baseline)

## Results and Discussion

## Preparation and validation of PRMs

PRMs with amount fractions of $CO_2$, 400 µmol/mol, 5 cmol/mol, 14 cmol/mol and 20 cmol/mol of $CO_2$ in $N_2$ and air matrix gases were prepared in accordance with ISO1642-1:2015 as described under the experimental section. The gravimetric amount fractions of the PRMs along with the uncertainties (%, *k*=2) were calculated using GravCalc2 software and are listed in Table 2. The concentrations of these PRMs were then validated by comparing with a matching NPL standards. 400 µmol/mol $CO_2$ in $N_2$ were analysed using Agilent GC fitted with a Pulsed Discharge Helium Ionization Detector (PDHID) while the 5 cmol/mol, 14 cmol/mol and 20 cmol/mol of $CO_2$ in air and $N_2$ matrix PRMs analysed using a GC fitted with Thermal Conductivity Detector (TCD) against traceable, matching NPL standards. The analytical amount fractions of the PRMs along with the analytical uncertainties (%, *k*=2) are also listed in Table 2. A calibration plot taking the MS response for $CO_2$ for each of the PRMs were plotted using XLGenline software as shown in Figure 2. The curve under first degree polynomial gave an $R^2$ value of >0.95.

Table 2. The gravimetric amount fractions and the analytical amount fractions with their relative uncertainties (%, *k*=2) of the $CO_2$ PRMs in $N_2$/air matrix gas.

| **Cylinder No** | **Gravimetric amount fraction** | **Gravimetric uncertainty (%), *k*=2** | **Analytical amount fraction** | **Analytical uncertainty (%), *k*=2** | **Matrix gas** |
|---|---|---|---|---|---|
| D180752 | 390.13 | 0.21 | 382.19 | 2.10 | Air |
| 232267SG | 50232.38 | 0.04 | 50271.21 | 0.24 | Nitrogen |
| D180566 | 138306.22 | 0.01 | 138575.19 | 1.25 | Nitrogen |
| D180755 | 198462.57 | 0.02 | 198561.20 | 0.84 | Air |

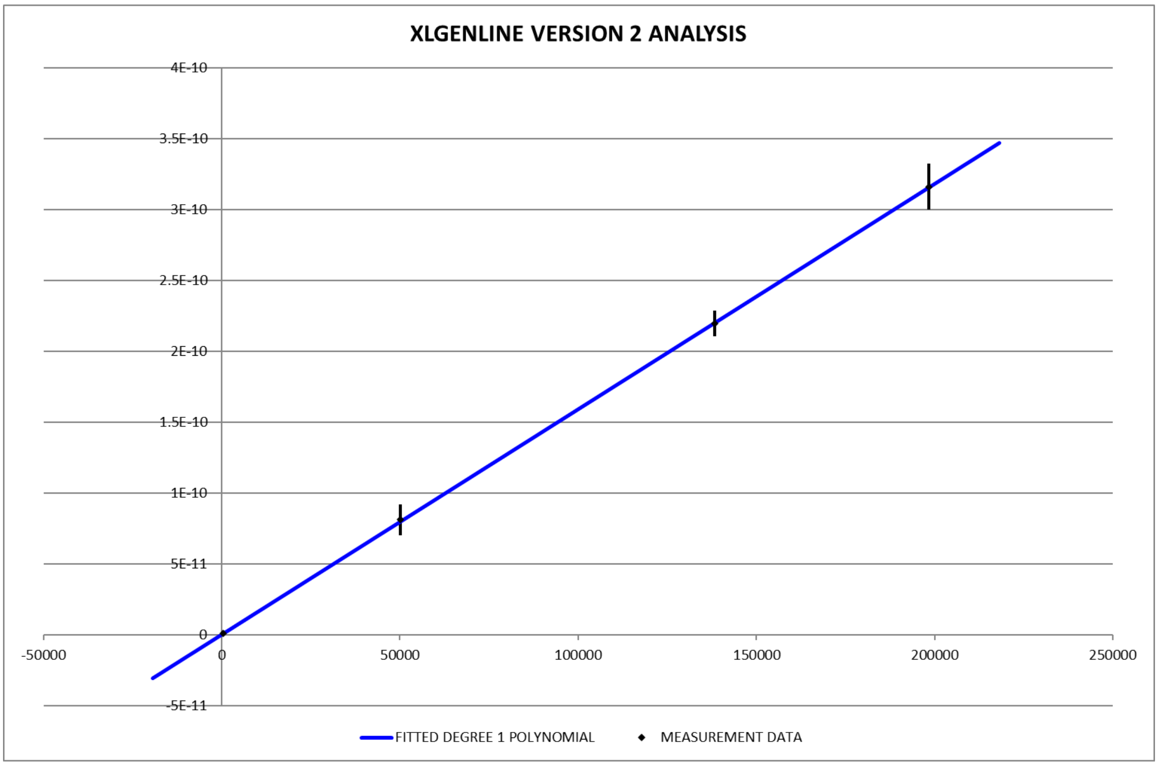


Figure 2. Calibration curve for the MS response for the four PRMs listed in Table 2.

## Carbonation and decarbonation experiments

Carbonation and decarbonation experiments were carried out as explained in the experimental section. Blank experiments were carried out in the absence of any CaO sorbent to get a baseline value to account for the contribution due to the setup and any residual adsorption by the tubing, connections and reactor. In a typical experiment, pure He gas (at 40 ml/min) was flowed through the reactor containing the accurately weighed amount of CaO. The temperature was ramped at 20 °C/min from room temperature to 600 °C. The gas was switched over to either of the NPL PRMs (containing 5, 14 or 20 cmol/mol $CO_2$ in air or $N_2$) and the temperature was held at 600 °C for 30 minutes to complete carbonation reaction of the CaO sorbent. The gas was then switched back to pure He (40 ml/min) and the temperature was raised to 900 °C at 20 °C/min and the sample was held at that temperature for 30 minutes to facilitate desorption of the earlier captured $CO_2$ gas. Following the desorption step the temperature was cooled to room temperature under the He gas flow (40 ml/min). Identical experimental conditions were provided for the blank experiment is the absence of CaO sorbent. As an example, Figure 3 shows the amount of $CO_2$ recorded on the MS during an experiment carried out by interaction of 14 cmol/mol $CO_2$ PRM with 0.5 g CaO sorbent in contrast to the empty reactor as an example. The temperature profile in Figure 3 also shows the temperature variation during the experiment. All the $CO_2$ in the NPL PRM was absorbed by the CaO sample in comparison with the empty reactor up to the end of the isothermal carbonation step at 600 °C. At this stage, as the gas was switched over to pure He gas and temperature was raised to 900 °C, the adsorbed $CO_2$ was given off completely by the end of the isothermal decarbonation step (at 900 °C). For the three trials of 14 cmol/mol $CO_2$ PRM interaction with CaO, 0.70 to 0.77 g of $CO_2$ was captured by 1 g of CaO. This value is quite close to the theoretical value which is 0.78 g of $CO_2$/g of CaO.

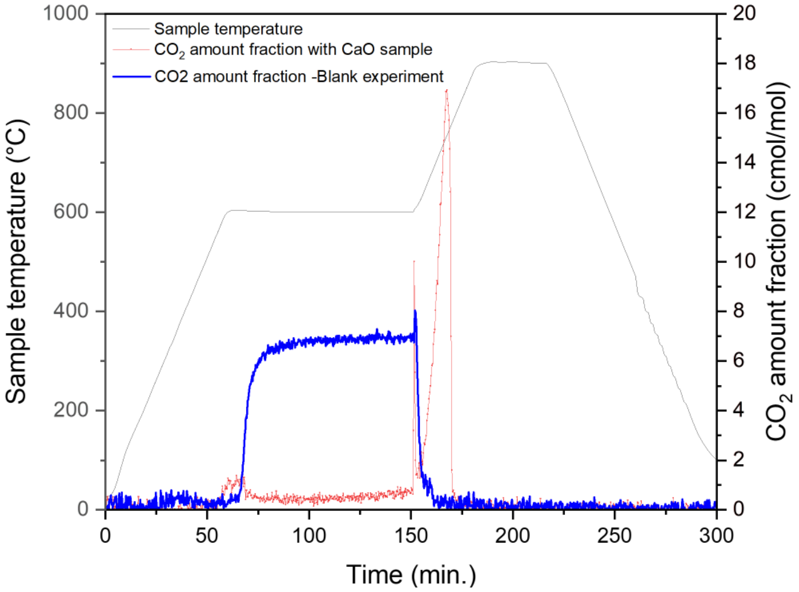


Figure 3. MS response for the adsorption of 14 % $CO_2$ PRM on an empty reactor (a) and CaO sorbent (b).

Since the sample could have absorbed moisture and $CO_2$ from the atmosphere (prior to the carbonation experiment), the sample was heated in a stream of He gas (40 ml/min) to 900 °C to liberate any surface absorbed species ($H_2O$ or $CO_2$) and regenerate the CaO sorbent. The weight of the sorbent was accurately measured before and after heating under pure He at 900 °C to quantify the effective amount of CaO that was actually present in 0.5 g of sorbent used for the experiments. Based on the effective amount of CaO that was present in 0.5 g of sorbent, the expected mass of $CO_2$ in g to be absorbed / g of CaO was found to be 0.71 g of $CO_2$. $CO_2$ absorption in grams by 1 g of CaO on interaction with different NPL PRMs and relative combined uncertainty ($k$=2) are listed in Table 3. Average amount for $CO_2$ absorbed per g of CaO for the 5, 14 and 20 cmol/mol of $CO_2$ PRMs was found to be 0.69 g, 0.74 g and 0.75 g respectively. Figure 4 also shows the amount of $CO_2$ sorbed by 1 g of CaO in the sample against the expected value with the combined uncertainty (cmol/mol, $k$=2) for carbonation experiments of CaO with all the 3 NPL PRMs. Most of the values showed good agreement with the expected value and more experiments are required to reduce uncertainty. There was increase in the average carbonation efficiency of 1 g of CaO observed with increase in $CO_2$ concentration in the NPL PRMs as reported in literature [3]. At this instance, no pronounced effect on carbonation efficiency was observed for $N_2$ and air matrix gas. However, more experiments with PRMs containing other impurities and amount fractions should be used to get more meaningful data.

Table 3. $CO_2$ absorption in grams by 1 g of CaO under the interaction with different NPL PRMs and combined uncertainty ($k$=2).

| $CO_2$ amount fraction (cmol/mol) | | 5, Combined uncertainty (g, $k$=2) | 14, Combined uncertainty (g, $k$=2) | 20, Combined uncertainty (g, $k$=2) |
|---|---|---|---|---|
| $CO_2$ absorbed/g of CaO (g) | Trial-1 | 0.75 (±0.05) | 0.77(±0.03) | 0.74(±0.04) |
| | Trial-2 | 0.65(±0.04) | 0.70(±0.03) | 0.69(±0.04) |
| | Trial-3 | 0.67(±0.04) | 0.74(±0.03) | 0.81(±0.05) |
| | Average | 0.69(±0.04) | 0.74(±0.03) | 0.75(±0.05) |

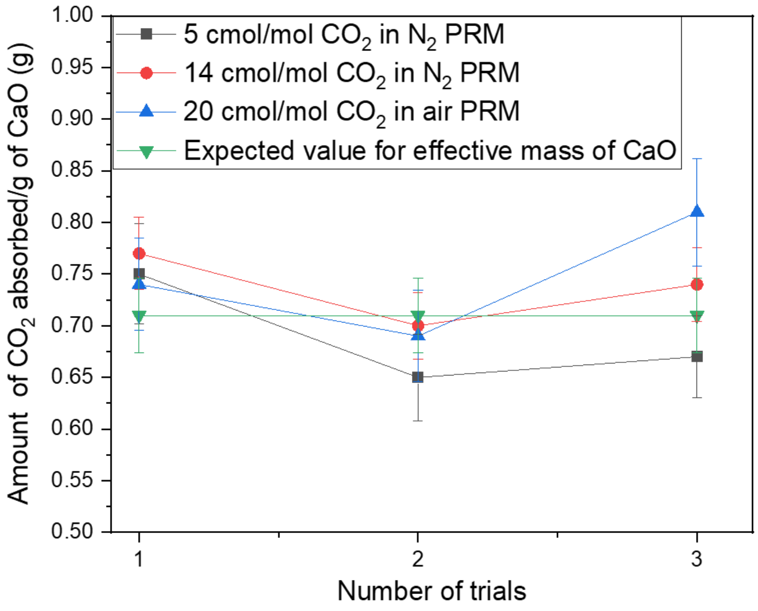


Figure 4. Amount of $CO_2$ sorbed for 1 g of CaO by different NPL PRMs.

## Uncertainty budget for the measurements

An uncertainty budget for the measurements made were calculated using GUM guidelines using equation 4. The uncertainty budget calculation for one of the three trials for the carbonation efficiency experiment of 0.5 g CaO interacting with 14 cmol/mol of $CO_2$ in $N_2$ is shown in Table 3 as an example. The measurements showed an expanded relative uncertainty (%, $k$=2) of < 6.56 % for any trial with any of the 3 NPL PRMs.

Table 3. Uncertainty budget for interaction of 14 cmol/mol of $CO_2$ in $N_2$ with 0.5 g of CaO sorbent for trial-1

| **Source of uncertainty** | **Relative uncertainty value** | **Probability distribution** | **Divisor - used to produce the standard uncertainty from the uncertainty value** | **Relative Standard uncertainty, $u(x_i)$** | **Sensitivity coefficient, $c_i$** | **Contribution to combined standard uncertainty, $u_i(y)$** |
|---|---|---|---|---|---|---|
| Pressure | 3.55 | Rectangular | 1.73 | 2.05 | 1 | 2.05 |
| Temperature | 1.07 | Rectangular | 1.73 | 0.62 | 1 | 0.62 |
| Mass of CaO within tube | 1.00 | Rectangular | 1.73 | 0.58 | 1 | 0.58 |
| Concentration of inlet stream | 0.50 | Rectangular | 1.73 | 0.29 | 1 | 0.29 |
| Volumetric flow of inlet stream | 0.50 | Triangular | 2.45 | 0.20 | 1 | 0.20 |
| Concentration of | 0.50 | Rectangular | 1.73 | 0.29 | 1 | 0.29 |

| | | | | | | |
|---|---|---|---|---|---|---|
| outlet stream | | | | | | |
| Volumetric flow of outlet stream | 0.50 | Triangular | 2.45 | 0.20 | 1 | 0.20 |
| Time | Negligible - drift over 2 days is < 1 second | | | | | |
| Combined standard uncertainty, $u_c(y)$ - quadrature sum | | | | | | 2.27 |
| Expanded uncertainty (%, $k$=2) | | | | | | 4.54 |

## Conclusion

A new materials testing platform (MTP) was set up at NPL to test and quantify the carbonation efficiency of solid sorbents. Under this work package of the MetCCUS project, CaO was used as a benchmark sorbent to test and validate the new setup at NPL. The setup consisted of a microreactor that held the solid sorbent for the interaction with NPL PRMs with varying $CO_2$ amount fractions (5 – 20 cmol/mol) in air or nitrogen matrix gas. The unit also consisted of MFCs that accurately controlled the flow of reactant gases, CatLab software that that controls and executes the stepwise temperature, MFC and measurement controls required for the carbonation and decarbonation steps. The concentration of the product gas was analysed using a mass spectrometer and data was recorded by the CatLab software. The CaO sample was tested with varying compositions of NPL PRMs to see the effect of $CO_2$ concentration in the NPL PRMs on the carbonation efficiency of the CaO sorbent. A blank reaction was conducted to remove any influence of the empty reactor system on the measured values.

The results from these preliminary experiments validates the experimental setup for the estimation of carbonation efficiency of solid sorbents application. The carbonation efficiency for interaction of 5, 14 and 20 cmol/mol $CO_2$ was found to vary between 0.65 to 0.81 g of $CO_2$ / g of CaO which was close to the theoretical value. The average carbonation efficiency was observed to increase from 0.69 to 0.75 g of $CO_2$ / g of CaO with the amount fraction of $CO_2$ in the NPL PRMs indicating the effect of $CO_2$ concentration on carbonation efficiency as per literature reports. An uncertainty budget was constructed using GUM by taking into account various factors that can contribute to overall uncertainty of the measurements. However, at this stage a strong effect of different matrix gases could not be established with certainty and more experiments are required to check for the effect of other impurities in the $CO_2$ PRMs, reduce uncertainties and check for reproducibility of these experiments. Other sorbents can be tested under similar conditions by benchmarking against CaO sorbents. The setup can also be used for testing liquid $CO_2$ sorbents by modifying the reactor setup and the MASsoft software can be tuned to identify and quantify specific degradation products that are coming out of specific $CO_2$ capture technology.

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