Experimental report

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Proposal:	**CRG-2657**	**Council:**	4/2019
Title:	In situ monitoring of the stabilization of crystalline vaterite (and an amorphous phase) by arsenic		
Research area:			

This proposal is a new proposal

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Samples: Calcium carbonate with As(V)

Instrument	Requested days	Allocated days	From	To
D1B	2	1	14/10/2019	15/10/2019

Abstract:

Experimental report CRG-2675

In situ monitoring of the stabilization of crystalline vaterite (and an amorphous phase) by arsenic

During the Beamtime granted, *in situ* monitoring of the crystallization of calcium carbonate polymorphs in the presence of arsenic was attempted. For this purpose, several solutions containing different amounts of arsenic were prepared at the chemistry lab (ILL) just before the mixture, trying to repeat the same experiments previously conducted at our laboratory. Solutions were called A, B and C. Solution A consisted of 0.2M of Na_2CO_3; solution B consisted of 0.2M of $CaCl_2$ and solution C consisted of an ICP Standard Solution containing 1000 mg/L of arsenic (Sigma Aldrich). As a first attempt, solutions A and C were mixed (in order to obtain a final concentration of arsenic equal to 100 mg/L) prior the addition of the solution B. Immediately after the addition of the $CaCl_2$ (solution B), solution became whitish and cloudy, indicating that the precipitation of $CaCO_3$ phase(s) was taking place.

In order to proceed the monitoring of the crystallization of $CaCO_3$ phase(s), seconds after the addition of solution C, neutrons beam was opened and recorded. Unfortunately, signal recorded did not show any signal from the precipitating sample. On the contrary, signal from the glass vial was collected. The signal collected did not show any information related to calcium carbonate phases, although the precipitation was taking place. This situation could be related to a low amount of diffracting phase(s). Hence, the experiment was left under the beam in order to check how long we would need for the next trial(s). After 1 hour, the signal did not enhance any evidence of the calcium carbonates and we decided to stop the *in situ* experiment.

As only 24 hours were awarded and the experimental setup and tests took several hours, we decided to get information from some *ex situ* samples brought from Salamanca, in order to get information concerning the structural positioning of the arsenic. For that purpose, solid samples were collected from experiments conducted under the same conditions, except two variables: concentration of arsenic (ranged from 0, 10, 50 and 100 mg/L) and experimental times (5 minutes and 96 hours). Samples collected were also characterized by means of in-house XRD. Additionally, we could conduct an experiment in the Chemistry Lab of ILL, in order to check the precipitating phase under extremely high As concentration (1000 mg/L).

For a better understanding of the incorporation of arsenic within the structures of the different calcium carbonates polymorphs, two different wavelengths of neutrons beam were used in order to enhance the spectral resolution. From these measurements, we have collected characterized 10 samples which need to be analysed in a near future.

Despite the unsuccessfulness of the in situ experiments we were able, notably owing to the extensive experience of Dr. Puente and Beamline technician, to obtain some information of the *ex situ* samples. If an additional beamtime (requested in the proposal) would be granted, conditions of the *in situ* experiments would be modified to rise up the supersaturation with respect the carbonate phase(s) and therefore the amount of diffracting solids, but this could be another proposal.