

Article

The challenge of monitoring impurity content of CO₂ streams

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Abstract: Carbon capture and storage has gotten increased attention during the last decade, and several full-scale projects are currently being planned. This requires transportation of large quantities of CO₂ from the capturing plant to the end point. From economic and public acceptance point of view it is important to ensure that the transportation system is operated in a safe manner. Thus, avoiding threats like corrosion or formation of particles are important. It is therefore required to monitor that the transported CO₂ fulfils the required specifications, and in practice this means that the impurity content of the CO₂ must be analysed.

CO₂ will in most cases be transported in the liquid or supercritical state (high pressure), which makes the practicalities around chemical analysis more difficult. Phase transition from liquid or supercritical state to gaseous state may also introduce several physiochemical effects that may affect the analysis.

This paper discusses technical and practical challenges with such types of analysis. Most of this work is based on experience that was gained during development of analytical system for dense phase CO₂ in a joint industry project that studied corrosion and chemical reactions in a simulated CO₂ transport system.

Keywords: CO₂ impurities; monitoring; chemical analysis

1. Introduction

Carbon capture and storage (CCS) has gotten increased attention as a method to reduce emissions of anthropogenic CO₂ to the atmosphere. The CO₂ from the capture plants will contain various impurities. The types and concentrations depend on the CO₂ source, the capturing technique, and the liquefaction process. In most cases, the CO₂ streams will need further conditioning/purification before transport and injection.

It has been shown experimentally that certain combinations of impurities react if they are present above a critical concentration [1-3]. It was demonstrated that H₂O, H₂S, SO₂, NO₂ and O₂ at concentrations much less than 100 ppmv reacted and created separate aqueous phases containing high concentrations (several mol/kg) of sulfuric and nitric acid. Formation of acid was also observed by Yevtushenko et al. [4] in experiments containing SO₂, NO₂ and O₂ at water saturation. The acidic aqueous phase may introduce corrosion problems in the CO₂ transportation system, a system which most likely will be constructed of carbon steel due to cost and availability. Elemental sulphur is another possible reaction product [5,6]. If the sulphur remains dissolved in the bulk CO₂ phase it will probably not cause any problems, but it may precipitate as solids and create particulate problems in the transportation system and reduced injectivity in the reservoir.

Several CO₂ specifications has been developed to ensure safe operation of the CO₂ transportation chain. As the mechanisms for formation of corrosive species and particulate matter has become better understood, the CO₂ specifications have become more and more strict (lower maximum impurity content. The actual quality of a CO₂ stream (or

bulk carrier) needs to be documented by chemical analyses. There are, however, currently no standards practice for such type of analysis.

In most cases, CO₂ will be transported in pipelines or by ship (bulk transport). Pipelines will be operated at high pressures (> 74 bar) and ambient temperature. Bulk transport will be carried out with CO₂ cooled to the liquid state with a small gas cap, for which the pressure will be in the range of 6 - 20 bar depending on the temperature [7-9].

Measurements of impurities in dense phase CO₂ (liquid or supercritical state) is challenging, particularly since some of the impurities are present at low concentrations and in addition some of them may react before analysis can be carried out.

Most of the present work is based on the experience gained during the process of building a corrosion test system that can be operated under CCS conditions in our lab. Analysers were used to compare the impurity concentration of inlet and outlet CO₂ from the autoclave (reaction chamber). Work with the test system(s) has been going on for more than 10 years and the development is still in progress. Even if the practicalities around such analysis may vary significantly from the lab to the field, many of the general problems and challenges are still the same and will be discussed in the paper.

2. Analysing the impurity content of CO₂ streams

2.1. The pressure challenge

Highly accurate gas analysers have been available for a long time and there are numerous instruments and techniques available. However, none of the instruments available on the open market are able to carry out analysis directly in the CO₂ transportation system due to the high pressure.

In practice, the pressure must be reduced to near atmospheric before analysis can be made, and therefore the CO₂ must be transformed from the supercritical or liquid state to the gaseous state. A pressure regulator is needed for this, and usually it will also require a mass flow controller to maintain a stable (but adjustable) feed of analyte.

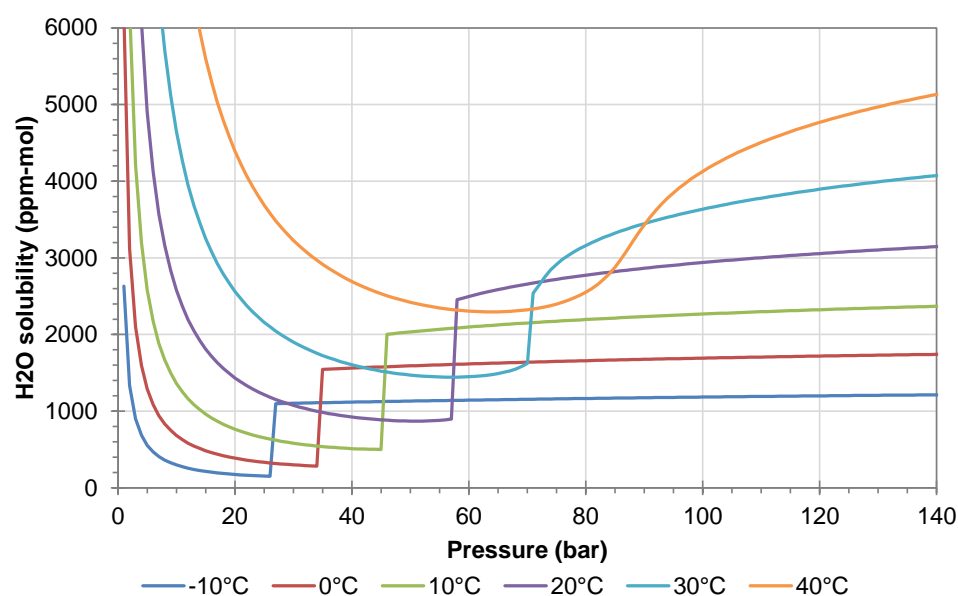


Figure 1: Water solubility (ppmv) in CO₂ as a function of pressure and temperature. The data has been calculated with the OLI software.

With pressure reduction and phase transformation there are several physiochemical factors that may affect the chemical analysis. Figure 1 shows the solubility of water in pure CO₂. When CO₂ is transferred from the liquid to the gaseous state there is a sharp drop in the water solubility, and hence there is a risk of water precipitation. The problem is enhanced by the Joule-Thomson effect, which reduces the temperature in the gas re-

duction valve. The reduction valves should therefore be operated with electric heating, as this prevents precipitation of liquid water. Heating will also prevent/reduce the risk of hydrate formation. In the gaseous state, the water solubility increases with decreasing pressure (left side of Figure 1).

A practical example that shows the effect of water precipitation in the gas regulator is given in Figure 2. The initial water analysis was very stable (500 ppmv), but it started to fluctuate somewhat when the water content was increased to 1200 ppmv. Shortly after injection of fully water saturated CO₂ (at 54 hours), the water signal started to fluctuate significantly. This is believed to be the result of the water precipitation/dissolution dynamics in the heated gas regulator. This type of observation is so common that it is used by the authors as an indication of water saturation

Figure 3 shows the predicted water solubility at 25 °C assuming full equilibrium and no temperature changes. If the water content is low, precipitation of water is not expected when the pressure is reduced from 100 to 1 bar (example with 250 ppmv shown by the green line/circles). The black lines/circles show the same process for a higher water content of 2000 ppmv. The water solubility is clearly exceeded during the phase transition from liquid to gaseous CO₂, where about 800 ppmv of water will precipitate as liquid water. It will gradually dissolve again when the pressure is reduced below 50 bar and it will be fully dissolved when the pressure reaches 20 bar.

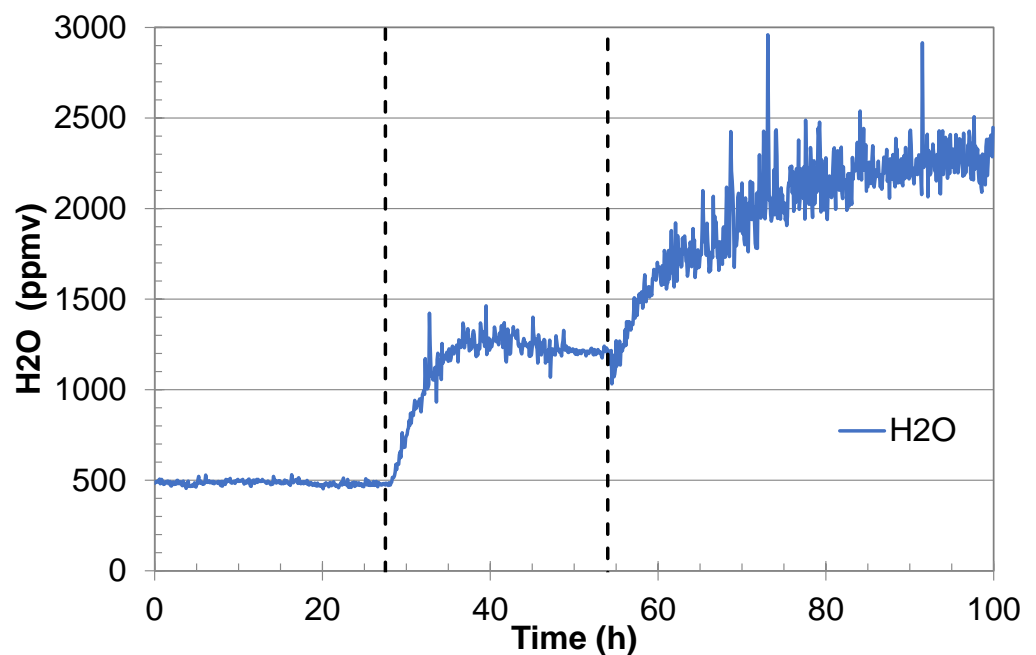


Figure 2: Water analysis from an experiment where the water injection was increased from 500 to 1250 ppmv (28 hours) and from 1250 to 2200 ppmv (54 hours).

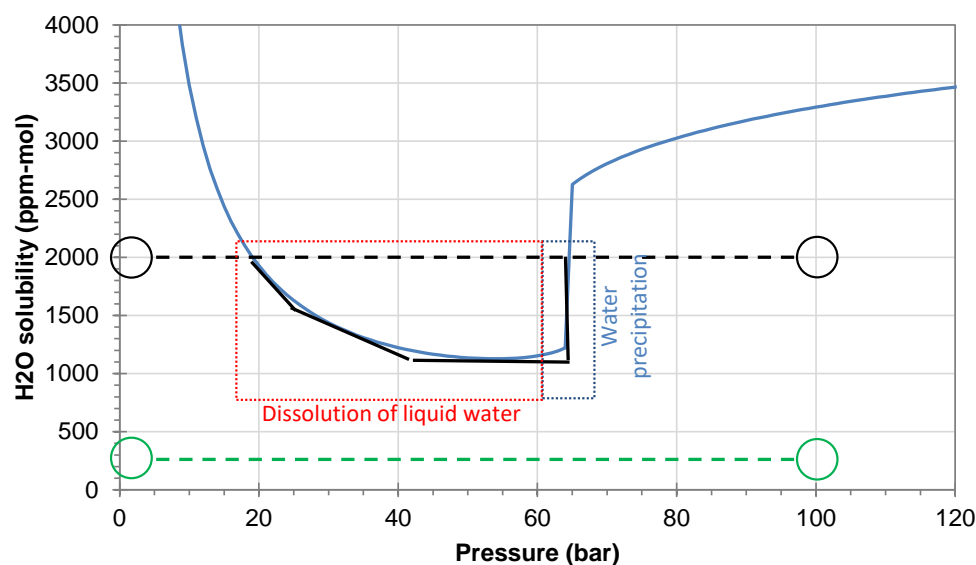


Figure 3: The blue line shows the water solubility (ppmv) in CO_2 as at 25°C (calculated with the OLI software). Green and black lines illustrate the (average) water solubility for depressurisation from 100 to 1 bara.

A sharp drop in solubility with decreasing pressure is also observed for other species like nitric acid, sulfuric acid, and elemental sulphur. If the precipitation is fast, species may accumulate in the heated gas regulator. This will of course result in erroneous chemical analysis, but it may also introduce clogging of the regulator, the mass flow controller, and the analysers (see example in Figure 6). Furthermore, if certain species precipitate, they could introduce chemical reactions in the feeding lines from the gas regulator to the analysers. If an aqueous phase with acids precipitates inside the heated gas regulator, the water measurements may also fluctuate even in the low ppmv range as shown in Figure 7.

2.2. The calibration challenge

Some instruments, like gas chromatographs, UV and IR photometers, need regular calibration. Even if this process is automated, it can be a somewhat tedious process, particularly in the field. It also means that bottles with calibration gas (one for each concentration) must be available at the site and be refilled in due time. Thus, there is a technological and logistic issue that has to be dealt with. This should be manageable at a CCS facility, which anyway will require a certain amount of qualified work force. The situation may be different on a remote site.

2.3. Saturating the sampling system with analyte

Several impurities may adsorb on internal surfaces of the sampling loop, like phase transition regulators, flow meters and analysing lines. Normally, the volume in the sampling system should be fully replaced with CO_2 feed (analyte) 3 to 5 times before a representative sample can be collected. This is not a problem for a continuous analysing system, but the lag time could be long when considering both the volume exchange and adsorption/desorption effects. This is commonly referred to as “saturating” the analysing system. If the analyte composition changes, these impurities will adsorb or desorb according to the surface equilibrium. It has been observed that H_2O , H_2S , and NH_3 requires long saturation times, while SO_2 , O_2 , and CO are much faster. The actual response time depend on the length of the analysis line and the volume and surface area of additional equipment's.

One of the setups in our lab consist of 20 meters 1/16" stainless steel tubing, 3 valves, 2 filters, a heated gas regulator and a 300 ml autoclave. It takes about 16 hours to saturate

this setup when changing the water content from 5 to 1500 ppmv with 500 ml/min total gas flow. If the water content is reduced from 1500 to 1000 ppmv, stable measurements are achieved after about 2 hours. The dry-up time for such a system is very long. The experience is that it takes *about 2 weeks* to reduce the water content from 1000 to 1 ppmv, and the last 50 ppmv are the most time consuming. It is possible to purchase tubing that has a special surface treatment ("inert tubing") that is claimed to reduce the problem. This effect has not yet been tested by the authors.

The problem can be reduced by designing the sampling system with a high-volume exchange rate. In practice this means low diameter tubing or a large flow of CO₂. With smaller diameter tubing there is an increased risk of clogging by solids and therefore some optimisation has to be made.

The phase transition regulator should be made of inert material. Usually, a fine tuning of the heat supply would also be necessary. More heat would at least give shorter adsorption/desorption time, but it could increase the risk of reaction between the impurities. Thus, there is a trade-off between response time and accuracy.

2.4 Sampling location

Corrosion and impurity reactions can form solids and liquid phases [2,3,10]. These products (solids, liquids) may separate from the CO₂ bulk phase due to density differences and either stick to the wall or follow the CO₂ stream. Depending on the pressure and temperature (density of CO₂), the products may be lighter or heavier than the bulk CO₂ phase. Flow and turbulence may also have an effect, particularly in pipelines. Products that are expected to be separated by gravity could be kept in the bulk phase as emulsions or small particles, but they may also accumulate either at the top or bottom of the transportation system. The practical sampling locations will have to be evaluated for each system, but one approach could be to sample from the top position (light components), middle position (bulk phase) and bottom position (heavy components), as indicated in Figure 4. Applying a large-diameter flange at the bottom would allow for accumulation of heavy components if the turbulence is not too high.

Partitioning of impurities in two-phase CO₂ is the case for a number of impurities, and sampling from the gas or liquid phase will therefore give different results [11-13]. Experiments showed that H₂O, SO₂, H₂S partition preferentially into liquid CO₂ phase while O₂ partition preferentially into the gas phase. This means that it is important to carry out sampling in a manner that prevents a two-phase system, e.g., by introducing a large pressure drop by fast sampling. A piston cylinder with back pressure could be used to avoid two phase formation during analysis of a batch CO₂ sample.

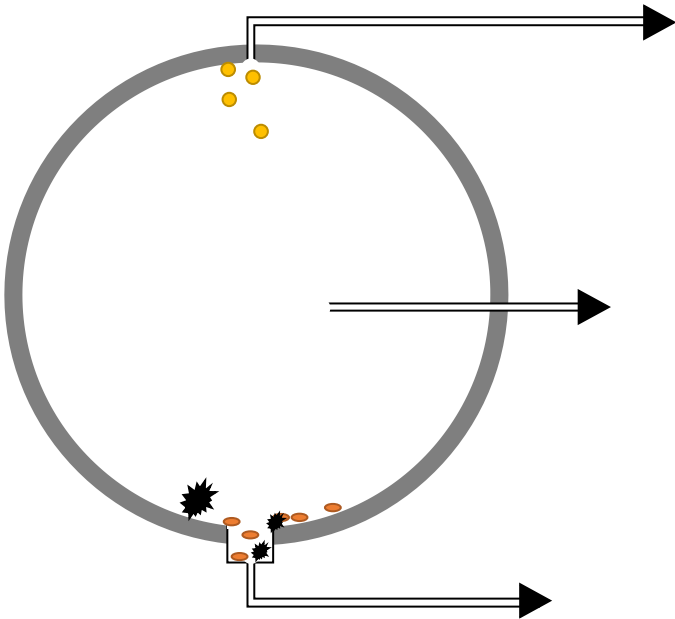


Figure 4: Schematic drawing indicating possible sample locations in a pipeline or CO₂ bulk carrier.

2.5. Impurity range

The impurity levels are expected to be low (typically <10 ppmv) during normal operation, and consequently low detection limits are required. Upset conditions, on the other hand, may introduce relatively high impurity levels. Thus, there is a trade-off between wide range and accuracy of the lowest concentrations.

3. Analysis and the need for phase separation

The CO₂ stream can in principle contain both gaseous, liquid and solid products. Each of these components will usually require different types of analysing techniques and will therefore have to be separated. With gaseous components, it is here meant components that are in gaseous state around room temperature and at pressure near 1 atm (after pressure reduction). The analysing approach will probably vary from site to site, but one example of a possible solution is shown in Figure 5.

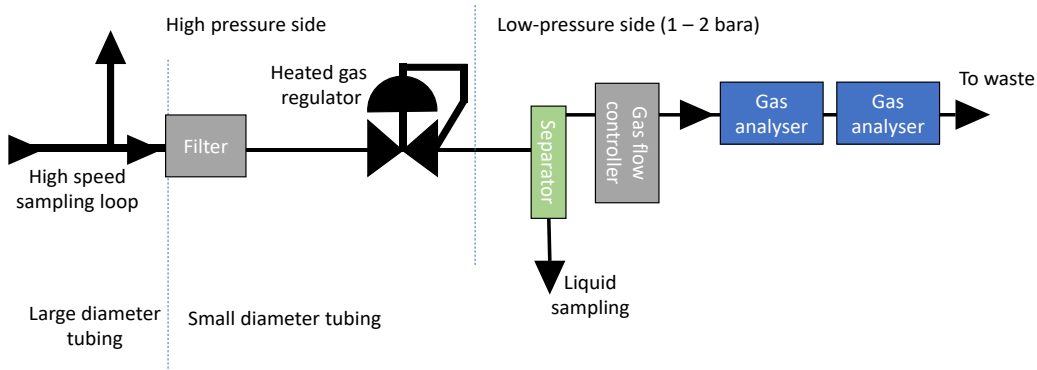


Figure 5: Schematic flow chart for analysis system.

3.1 Gaseous species

After the heated gas regulator, the gas can be analysed directly. The choice of analysing method is usually based on a number of factors, of which economy, number of analysed species, detection limit, reproducibility, need for calibration, ease of re-

pair/maintenance and equipment lifetime are the most important. Analysers based on laser absorption spectroscopy was selected in the authors' lab as the best choice due to high analysis frequency and long calibration intervals. One disadvantage with the current setup is that the number of possible analysed species is fixed. Furthermore, not all species can be analysed for (there are several reasons for this). Although this probably can be improved in the future, it means that these laser-based techniques must be combined with other analytic methods. Several laser-based analysers may also be applied in series.

Gas chromatography (GC) needs regular calibration and is therefore not used for long term analysis by the authors (with dense phase CO₂). It is sometimes used for infrequent analysis. However, GC has the advantage that it relatively easy can be modified to analyse for new species.

Regardless of the analysis technique, it is difficult to find one technique and instrument that can handle all species, and several analysers have to be used in combination.

3.2 Solids

Solids can be separated from the bulk CO₂ phase with filters. Since the solubility of certain components vary with pressure, it is best to apply the filter before the phase transition regulator. This will also prevent clogging problems of the gas regulator. Nevertheless, the filter housing must be able to handle the CO₂ pressure, and it must withstand frequent depressurization in a controlled manner (particularly soft materials like rubber gaskets or Teflon-coated diaphragm can be damaged due to rapid depressurization). The filters could be analysed by standard methods, e.g., by X-ray powder diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). Dissolving the products in a suitable solvent could be another solution.

A filter can also be installed on the low-pressure side for protection of the analysers and the mass flow controllers etc. Comparison of filters from up and downstream the reduction valve may give useful information, since the pressure drop over the heated gas regulator may lead to precipitation of certain species, see Figure 6.



Figure 6: Pictures showing products that have precipitated inside the heated gas regulator body (left) and on the regulator's diaphragm (right).

3.3 Liquids

Liquids can be difficult to separate from dense phase CO₂ since they may have almost the same density. The density differences are much higher downstream the pressure regulator and gaseous and liquid species can then be separated by top and bottom

streams in a small separator. The separator could even be equipped with a small window for in-situ observation of liquids. The liquid could be analysed using conventional method, like ion chromatography, liquid chromatography, etc.

As shown in Figure 6, liquids may condense inside the heated regulator due to the pressure drop. A drain on the regulator would decrease the need for maintenance. Typical signs of liquid condensation in the regulator or downstream are fluctuations in the water analysis, as shown in the example in Figure 7.

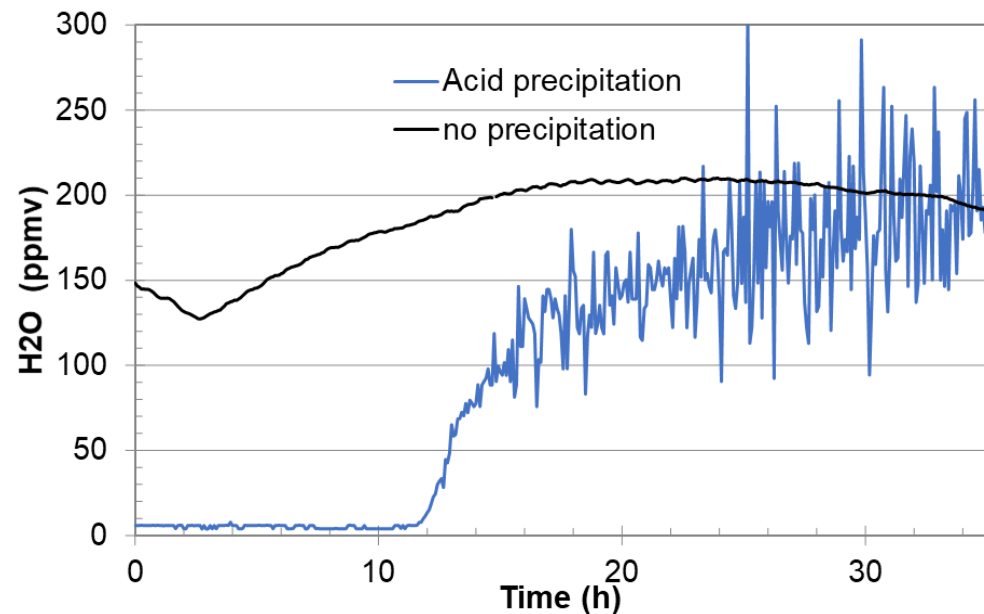


Figure 7: Water analysis from two experiments. The blue line is from an experiment where the exhaust CO₂ was saturated with sulfuric acid (after 11 hours), which precipitated in the heated gas regulator and introduced significant fluctuations in the water content. The black line is from another experiment without sulfuric acid.

4. Monitoring multiple CO₂ streams

For single CO₂ streams it should be relatively straight forward to document that the quality is within a given specification by using the earlier mention techniques. If no reaction or corrosion occurs, the content of impurities should be the same along the whole transport system. Changes in impurity concentration would indicate that chemical reactions or even corrosion is taking place. This will obviously require multiple analysing points.

For a large CO₂ transportation network there will be CO₂ streams from several CO₂ sources gathered in large transport lines or temporary stored for ship transport. See illustration in Figure 8. The different CO₂ streams might have different impurities in both type and concentration. Even if these streams are stable individually (no chemical reactions), chemical reactions could occur when the streams are mixed. Thus, there is a need to document the impurity content before and after mixing.

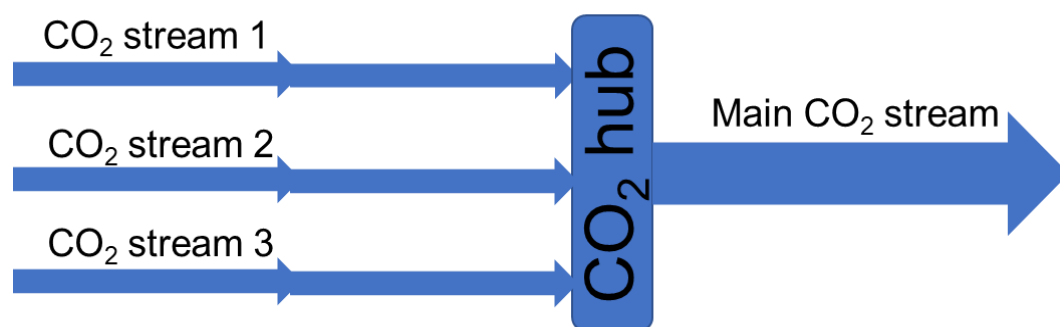
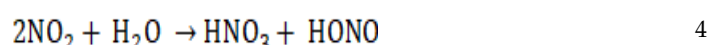
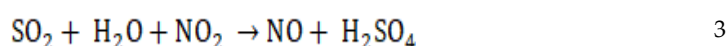
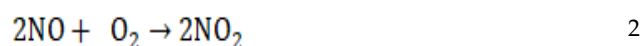
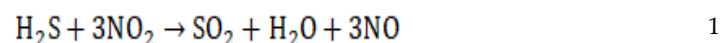


Figure 8: Illustration of several CO₂ streams mixed in a hub for then to follow one main flow to storage or utilization.

4.1 Reactions

Due to the large number of possible impurities, the number of possible chemical reactions is high. The most important reactions that were identified in the Kjeller Dense phase CO₂ project (KDC) [1,2,5,14-16] were:



Nitrogen dioxide is a strong oxidation agent and will readily react with hydrogen sulphide to sulphur dioxide and water (reaction 1). As long as oxygen is present, nitrogen dioxide will be regenerated according to reaction 2. It should be noted that due to this regeneration, only trace amount of NO₂ is required to oxidise H₂S as long as oxygen is present. The only NO₂-sink will be formation of nitric acid or corrosion.

Sulphuric acid is formed according to reaction 3, but to form liquid acid inside the transport system, it has been observed that the SO₂ content need to exceed 50 - 60 ppmv before H₂SO₄ forms and precipitates as a liquid phase (25 °C and 100 bar).

Reaction 1 to 4 could be used for guidance when interpreting the results from the analyses of the CO₂ streams. This will be discussed further in the following chapters.

4.2 False accordance with the specification

Monitoring the composition of a CO₂ stream where the impurities may react before the analysis introduce the need for special evaluations in addition to the chemical analysis. For example, monitoring parameters like flow, pressure and temperature could reveal possible risk of precipitation (see for example Figure 3). Comparison of analyses at different positions can also give valuable information. All this should be combined and compared with the known chemical reactions and limits (see Chapter 4.1) and also compared to thermodynamic models (if available).

Examples of how misleading the analyses can be are shown in Figure 9 and Figure 10. The injected impurity concentrations were the same in both experiments [5] but the injections of the impurities were started at different times. In Figure 9, it is quite clear that no chemical reactions took place when H₂O, SO₂, O₂ and H₂S were injected as they all reached stable target values. Once the NO₂ injection started, reactions took place, and an aqueous phase of H₂SO₄ and HNO₃ precipitated. In the other experiment (Figure 10), where all the impurities were injected simultaneously from start-up, the analysis showed practically zero content of H₂S, and SO₂ and O₂ were much lower than the injected content. The readings in **Error! Reference source not found.** could therefore erroneously

lead to the conclusion that the CO₂ stream is in accordance with the specification, even if more than 70 percent of the impurities are missing due to reactions. The reason for the missing impurities was reaction to acids (reactions 3 and 4) and solid formation. If all the sulphur species (SO₂ + H₂S) react to sulphuric acid (reaction 1 + reaction 3), about 500 gram would be produced per ton CO₂. A system transporting one megaton per year would in this case produce 500 tonnes acid per year. This large amount of acid would most likely threaten the integrity of the transportation system due to corrosion, and routines must be implemented to prevent (and detect) such a situation. This emphasises the importance of several measuring points to ensure sufficient control over the transport system and the design of the analysing system must take into consideration that new products from chemical reactions could appear in addition to those impurities included in the specification.

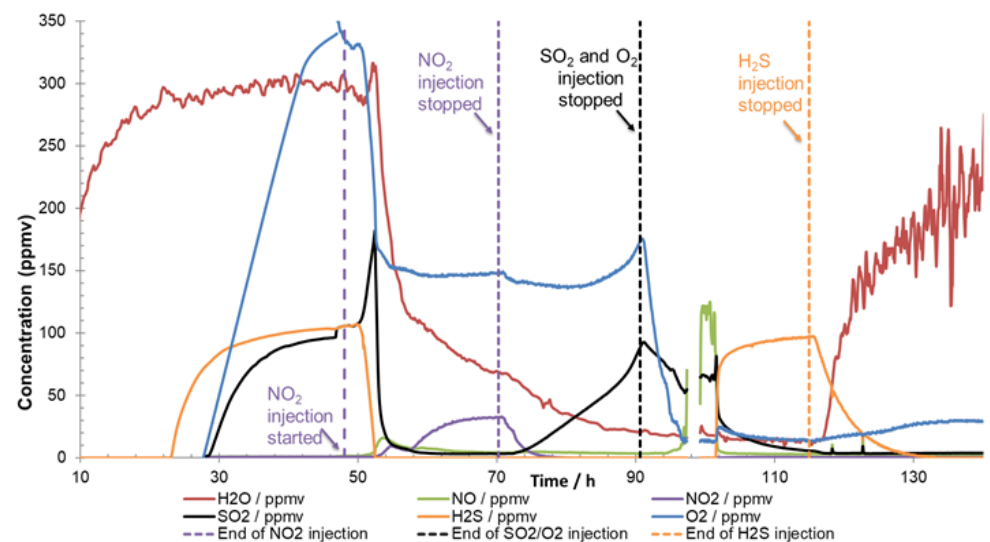


Figure 9: The result of first injecting of 300 ppmv H₂O, 350 ppmv O₂, 100 ppmv of SO₂ and H₂S until the concentration stabilized in 100 bar CO₂ and 25 °C. After about 50 hours the injection of 100 ppmv NO₂ was started and immediately a reaction about the impurities causes a deviation from the setpoints [5].

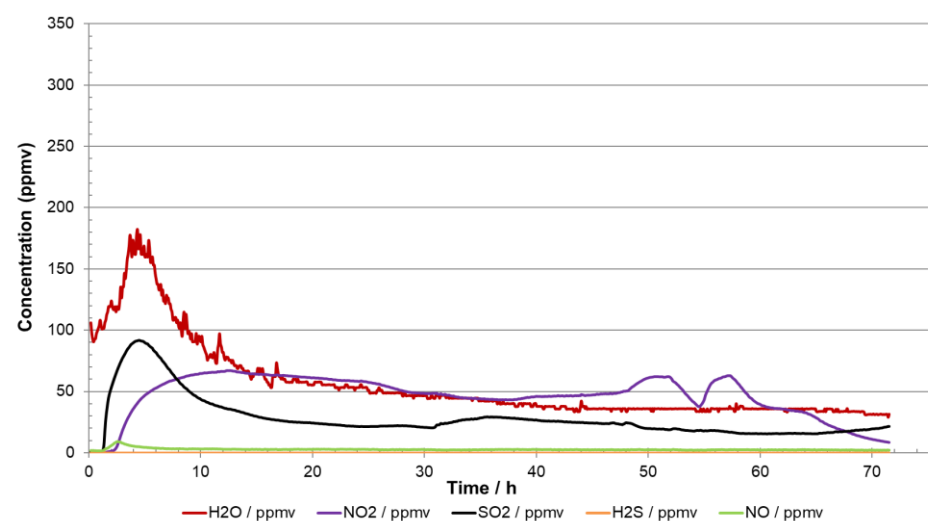


Figure 10: The result of simultaneously injection of 300 ppmv H₂O, 350 ppmv O₂, 100 ppmv of NO₂, SO₂, and H₂S in 100 bar CO₂ and 25 °C None of the impurities reaches their setpoint due to reactions [5]. The O₂ content was not measured due to experimental problems.

4.3 Simulation and predicting

By using reaction 1 to 4, it is possible to make simple predictions of the results of mixing CO₂ streams. If all side streams and the main CO₂ stream (**Error! Reference source not found.**) is monitored, it is possible to identify upsets. If no reactions take place, a simple mass balance of the small streams should give the composition of the mixed stream.

However, even if most of the chemical reactions are known, a full simulation of the mixing of multiple CO₂ streams with occurring reactions is a quite complex process. Parameters like chemical kinetics and competition between reactions, surface adsorption, and catalysing effects are not fully understood at present. Another aspect that would add complexity to the simulation occurs if a separate aqueous phase forms and accumulate in the system. The presence of an aqueous phase might not only change the kinetical parameters but could also change the ongoing reactions or introduce new reactions, including corrosion reactions, that would further complicate the simulation.

The results from the KDC-project are currently being implemented in a thermodynamic model developed by OLI Systems [17,18]. The model is still in the development stage for use in CCS-systems.

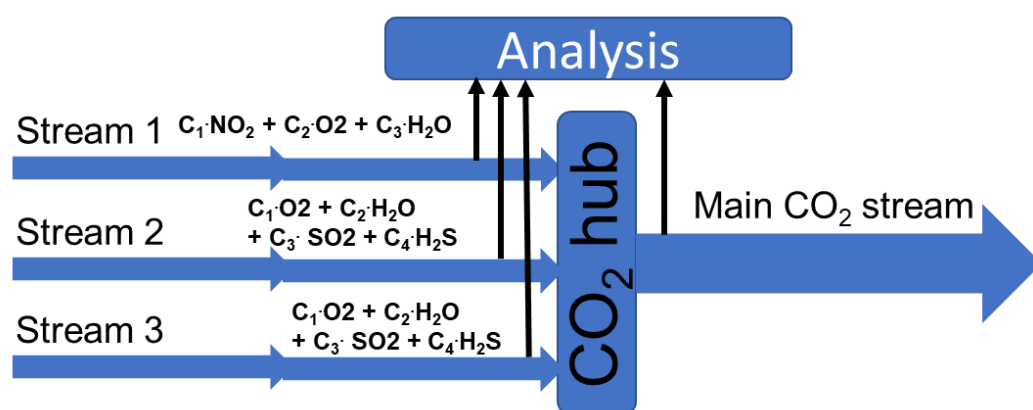


Figure 11: The mixing of different CO₂ streams with known impurities and concentration in a hub.

Even though an accurate simulation might be complex, the prediction from the reaction alone would give valuable input to the outcome of mixing streams or building specifications. Table 1 shows an example of such prediction, where no aqueous phase will form since the sum of H₂S and SO₂ is well below the threshold value for reaction 3 (see Chapter 4.1). An interesting outcome of the exercises is that reaction 1 leads to higher concentration of SO₂ and H₂O, which in certain situations could exceed the threshold for reaction 3, even if the original SO₂ content was well below this level. Further could this increase also lead to the limits of the accepted specification are exceed. Yet, research has shown that the concentrations in Table 1 are safe [1].

The understanding of ongoing processes in the transported CO₂ might ease the simulations but it might also untangle the complex analysis after mixing several CO₂ streams loaded with even more types of impurities..

Table 1. Prediction based only on the reactions; the input impurities are the sum of all streams while the output concentration are the results of the reactions.

| Impurity | Concentration before reaction (ppmv) | Concentration after reaction (ppmv) |
|------------------|--|---|
| NO ₂ | 10 | 3 |
| O ₂ | 10 | 0 |
| H ₂ O | 30 | 39 |
| SO ₂ | 10 | 19 |
| H ₂ S | 9 | 0 |
| NO | 0 | 7 |

5. Conclusion

Analysis of impurities in dense phase CO₂ require pressure reduction and phase transformation to gaseous CO₂ and is therefore more complicated than analysis of gaseous CO₂ alone. Possible reaction of impurities makes such analysis even more challenging. Even 99.95 percent clean CO₂ (food grade) has the potential to produce acids and solids, which may have negative effects on the analysis system.

Multiple analysing points along the transportation system (e.g., inlet and outlet) will increase the possibility to reveal ongoing processes like chemical reactions or corrosion. If multiple CO₂ side streams are being merged in a network, it is necessary to analyse all CO₂ streams before and after mixing, to ensure that the specification is fulfilled. Such analysis could also be assisted by predictions based on identified chemical reactions.

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