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## Update of DNV recommended practice RP-J202 with focus on CO<sub>2</sub> Corrosion with Impurities

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### Abstract

This paper presents results from experimental testing program designed to fill knowledge gaps identified during the process of making a recommended practice for design and operation of CO<sub>2</sub> pipelines. This testing program has been executed as part of the second phase of the CO<sub>2</sub>PIPETRANS Joint Industry Project (JIP).

The objective of the corrosion work package was to determine the mechanism and corrosion rate in dense phase CO<sub>2</sub> for various impurities including O<sub>2</sub>, SO<sub>x</sub>, NO<sub>x</sub>, and H<sub>2</sub>S with and without free water. The experimental work was carried out using a specialised rotating autoclave for carrying out CO<sub>2</sub> corrosion experiments, which gives a good mixing and disturbed flow around the test specimen. The test specimens used in the set-up were selected grades of carbon steel and stainless steel, typically used as pipeline materials.

The experiments were carried out at two different temperatures, that is 4°C and 50°C, at a pressure of approximately 100 bar. Different combinations and concentrations of the impurities O<sub>2</sub>, SO<sub>2</sub>, H<sub>2</sub>S, NO, NO<sub>2</sub> were added to the pure CO<sub>2</sub> with water concentrations of 50 and 500 ppmv. Published quality recommendations from the DYNAMIS were used as a starting point to decide the concentrations of the impurities to be tested. After the corrosion testing the metal loss was determined and the corrosion rates were calculated.

The effect of the impurities on the solubility limits of water, which results in increased corrosion rates, are presented. Further, how to use the results in order to define a safe operating window for dense phase CO<sub>2</sub> containing various impurities is discussed. Based on the experimental results, there are still uncertainties related to water solubility and the corrosion mechanisms in the presence of impurities. Further work is required to narrow the uncertainty, however, the uncertainty can be overcome with an appropriate design philosophy.

The results are a step in the refinement of the operational limits for different impurities found in pipelines carrying anthropogenic CO<sub>2</sub> and the moisture content. The results serves as input for updating DNV-RP-J202 “Design and Operation of CO<sub>2</sub> pipelines” [1] to provide improved guidance on safe and reliable design, construction and

operation of pipelines intended for large scale transportation of CO<sub>2</sub>. Increased understanding of corrosion mechanisms furthermore leads to more cost effective pipeline design and design of CO<sub>2</sub> conditioning processes.

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## 1. Introduction

CO<sub>2</sub>PIPETRANS is a JIP with three main technical work packages focusing on dense phase CO<sub>2</sub> release model validation data, fracture arrest and corrosion, respectively. The JIP consists of 15 partner organisations, who are: Arcelor Mittal, BP, DNV GL, Endesa, ENI, E.on Ruhrgas, Gassco, Gassnova, Health and Safety Executive (HSE) UK, Maersk Oil, Petrobras, Petroleum Safety Authority (PSA) Norway, Shell, V&M Tubes, and Vattenfall. During phase 1 of CO<sub>2</sub>PIPETRANS studies were undertaken on ten key topics related to design and operation of pipelines carrying dense phase CO<sub>2</sub> which were used as a basis for writing DNV-RP-J202 [1]. During this process six knowledge gaps were identified, namely: dispersion model validation, fracture arrest, corrosion, materials compatibility, hydrate formation, and the effect of impurities on thermodynamic properties of CO<sub>2</sub>. CO<sub>2</sub>PIPETRANS Phase 2 scope covers the former three knowledge gaps.

Given the current status of knowledge DNV-RP-J202 recommends “the primary strategy for corrosion control should be sufficient dewatering of the CO<sub>2</sub> at the inlet of the pipeline” to avoid the formation of a corrosive phase. There is not currently a consensus in industry, as to what the maximum water concentration should be for CO<sub>2</sub> pipelines.

The minimum water content required to form a corrosive aqueous phase strongly depends on the presence of other impurities and can be more than an order of magnitude less than the water solubility in pure CO<sub>2</sub> [2].

A number of tentative CO<sub>2</sub> specifications have been suggested [3,4,5,6,7]. It has frequently been referred to the DYNAMIS specification shown in Table 1 and the specifications for dried CO<sub>2</sub> issued by the Intergovernmental Panel on Climate Change-IPCC (Table 2).

The motivation for the experimental program that was carried out in 2011 and 2012 was to confirm whether these CO<sub>2</sub> specifications represent a safe operation window with respect to the impurity contents in the CO<sub>2</sub> and if not suggest necessary adjustments.

Two experimental series were performed. The water content was kept at 500 ppmv in the first series while various combinations of NO<sub>2</sub>, SO<sub>2</sub>, O<sub>2</sub> and H<sub>2</sub>S were added at concentrations slightly above and below the concentration given in Table 1 and Table 2. Corrosion was observed in most of these experiments and it was therefore not possible to conclude on the maximum impurity levels for safe operation. The water content was reduced to 50 ppmv in the second series.

Table 1. DYNAMIS CO<sub>2</sub> quality recommendation [4]

Component	Concentration	Limitation
H <sub>2</sub> O	500 ppmv	Technical
H <sub>2</sub> S	200 ppmv	Health & safety
CO	2000 ppmv	Health & safety
O <sub>2</sub>	Aquifer < 4 vol%, EOR 100 – 1000 ppmv	Technical
CH <sub>4</sub>	Aquifer < 4 vol%, EOR < 2 vol%	
N <sub>2</sub> , Ar, H <sub>2</sub>	< 4 vol % (all non-condensable gases)	

SO <sub>x</sub>	100 ppmv	Health & safety
NO <sub>x</sub>	100 ppmv	Health & safety
CO <sub>2</sub>	>95.5 vol %	

Table 2. Concentrations of impurities in dried CO<sub>2</sub> suggested by IPCC [5]

	SO <sub>2</sub> ppmv	NO <sub>x</sub> ppmv	H <sub>2</sub> S ppmv	CO ppmv	N <sub>2</sub> /Ar/O <sub>2</sub> ppmv
<b>COAL FIRED PLANTS</b>					
Post-combustion capture	<100	<100	0	0	100
Pre-combustion capture	0	0	100-6000	300-4000	300-6000
Oxy-fuel	5 000	100	0	0	37 000
<b>GAS FIRED PLANTS</b>					
Post-combustion capture	<100	<100	0	0	100
Pre-combustion capture	0	0	<100	400	13000
Oxy-fuel	<100	<100	0	0	41000

## 2. Experimental

There are no recognized standards for corrosion testing in dense phase CO<sub>2</sub> with impurities. The data published in the literature are based on autoclave experiments performed under stagnant conditions or with rotating cages [8]. The main experimental challenge is impurity control. The volume of the corrosive phases that might form in a system with a few hundred ppmv of impurities is very small. The volume fraction is  $< 10^{-4}$  (< one droplet per litre) and key issues are the consumption of the impurities during the exposure and to which degree the corrosive phase actually reaches the exposed steel specimens in the autoclave experiments, particularly in stagnant experiments.

IFE uses an experimental set up for dense phase CO<sub>2</sub> testing where slim (ID 20-30 mm) autoclaves are rotated on a shaft inside a temperature controlled chamber (see Figure 1). The temperature can be varied from 0-50 °C. The rotating device can accommodate 5 long (2 m) and 4 short (0.6 m) autoclaves. The test specimens are mounted on small cylindrical racks that slide from one end to the other when the autoclave rotates. The cylindrical steel specimens (10 mm long, OD 10 mm) used in the present experiments were machined from ferritic-pearlitic X65 pipeline steel with the composition shown in Table 3. The sliding rack gives good mixing and disturbed flow around the test specimen. The rack weight determines the maximum flow velocity.

Table 3. Element analysis (wt-%) of the exposed steel specimens

Steel	C	Si	Mn	S	P	Cr	Ni	V	Mo	Cu	Al	Sn	Nb
API 5L X65	0.08	0.25	1.54	0.001	0.019	0.04	0.05	0.095	0.01	0.02	0.038	0.001	0.043

The autoclaves can be rotated continuously at various speeds or in steps according to a programmed sequence. The rotation speed was 3 revolutions per minute in the present experiments, corresponding to an average flow velocity of 0.2 m/s and a peak flow velocity around 1 m/s.



Figure 1. Left: Slim autoclaves rotated in a thermal cabinet. Right: Carbon steel specimens (OD 10 mm, length 10mm) mounted in a 8-10 cm long sliding specimen rack.

The benefit of the rotating autoclave approach compared to fixed autoclaves and closed loops are the simple geometry that gives few dead ends where impurities forming a minute separate water rich phase can be trapped. A shortcoming can be that the flow velocity and the flow direction are alternating. Renewal and control of test liquid during the exposure is also more difficult than in stationary equipment, but this problem has been resolved in a new set up that is presently used [8].

When steel corrodes, the reaction consumes water and other impurities and generates  $H_2$  as a reaction product. In order to maintain a reasonably stable impurity concentration in a closed system with a small amount of impurities, it is necessary to either replenish the consumed impurities or use very small specimens giving a low surface area to liquid volume ratio. No replenishment was done during the exposure.

### 3. Results

Test conditions and results are summarised in Table 5 and Table 4. The reported corrosion rates were determined from weight loss after removal of the corrosion film in an inhibited acidic stripping solution. The accuracy of the weighing is about 0.1 mg and this corresponds to a corrosion rate of 0.002 mm/y in a 1 week exposure. An error (inaccuracy) is introduced when the corrosion film is stripped as a tiny amount of steel is removed at the same time. This gives a practical detection limit of 0.01 and 0.005 mm/y respectively for exposures shorter and longer than 1 week. When the corrosion rates are lower they are reported as < 0.01 and < 0.005 mm/y, respectively. In case no corrosion or visible attack is seen, this is reported as NVA (No visible attack).

A known amount of impurities were added to the autoclaves when it was filled with  $CO_2$  and the reported concentrations in Table 5 and Table 4 are the nominal start concentrations. The consumption rate due to corrosion and the changes in the concentration of impurities due to cross chemical reactions in the bulk phase were not measured during the experiments.

Two experimental series with 500 ppmv and 50ppmv water respectively were performed. All experiments were run at 100 bar  $CO_2$ .

Table 4 Survey of test conditions and results obtained in experiments with 500 ppmv water. All experiments were run at about 100 bar.

Test No	Temp. °C	SO <sub>2</sub> ppmv	NO <sub>2</sub> ppmv	H <sub>2</sub> S ppmv	NO ppmv	O <sub>2</sub> ppmv	Duration days	CR mm/y
DnV 1a	4	200				0	7	0.022
DnV 1b	4	200				0	7	0.022
DnV 3a	4	200				500	7	0.006
DnV 3a	4	200				500	7	0.009
DnV 12a	4	200				500	7	0.013
DnV 19	4		200			0	7	0.017
DnV 4.2a	4		200			500	3	0.2
DnV 4.1a	4		200			500	3	0.21
DnV 1_3 NO	4			200		500	7	<0.005
DnV 6a	4			200		500	7	<0.005
DnV 3_3 NO	4				200	0	7	<0.05
DnV 5a	4				200	500	7	0.082
DnV 5b	4					500	7	0.008
DnV 6b	4					500	7	<0.005
DnV 21	50	100				0	7	NVA
DnV 15	50	200				0	7	<0.005
DnV 30	50		50			0	1	0.127
DnV 29	50		50			500	1	0.116
DnV 22	50		100			0	7	0.02
DnV 27	50		100			500	1	0.182
DnV 20	50		200			0	1	0.205
DnV 16	50		200			0	3	0.088
DnV 20	50		200			0	7	0.025
DnV 14	50		200			500	1	0.275
DnV 28	50		200			500	7	0.09
DnV 2_3 NO	50			200		500	7	0.005
DnV 9b	50			200		500	7	0.006
DnV 9a	50				200	0	7	0.013
DnV 10b	50				200	500	7	0.03
DnV 4_3 NO	50					500	7	0.006
DnV 10a	50					500	7	<0.005

Table 5 Survey of test conditions and results obtained in experiments with C-steel exposed to the impurities: SO<sub>2</sub>, water and O<sub>2</sub>. All experiments were run at about 100 bar.

Test No	Temp. °C	SO <sub>2</sub> ppmv	NO <sub>2</sub> ppmv	H <sub>2</sub> S ppmv	NO ppmv	O <sub>2</sub> ppmv	Duration days	CR mm/y
DnV 23a	4	100				0	7	NVA
DnV 31	4	100				0	7	NVA
DnV 41	4	100				200	7	NVA
DnV 37	4	100				10000	7	NVA
DnV 33	4		100			0	7	0.011
DnV 35	4		100			200	7	0.002
DnV 24	4		100			500	7	0.005
DnV 39	4		100			10 000	7	NVA
DnV 25	50	100				0	7	<0.005
DnV 32	50	100				0	7	NVA
DnV 42	50	100				200	7	NVA
DnV 38	50	100				10000	7	NVA
DnV 26	50		100			500	7	0.005
DnV 40	50		100			10 000	7	NVA

### 3.1. Experiments with 500 ppmv water

All the specimens were slightly attacked in the 500 ppmv water experiments (Table 4). Highest corrosion rate ( $>0.2$  mm/y) was measured in the experiments with  $\text{NO}_2$  and  $\text{NO}$ . Post examination of the specimens showed that the steel surfaces were covered with a thin brownish layer of corrosion products. Examples of the surface appearance are shown in Figure 2b. EDS analyses indicated Fe and O only, no N containing compounds were found. XRD (X-ray Diffraction) analyses gave poor signals and no positive identification of crystalline products.

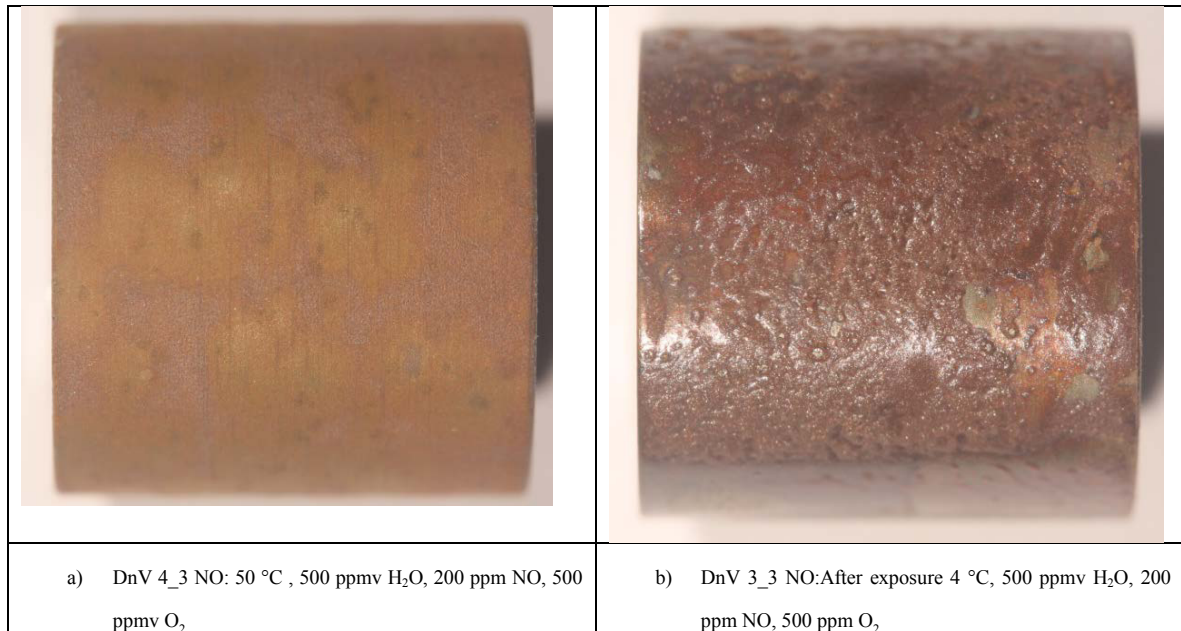


Figure 2 Surface appearance of exposed carbon steel in experiment

The weight loss corrosion rate in the  $\text{SO}_2$  experiments were lower than in the  $\text{NO}_x$  experiments and too low in many cases to be converted to an accurate corrosion rate. Most of the specimens experienced small local attacks or stained areas as shown in Figure 3. Clusters of corrosion products with corrosion underneath stuck to the surface and SEM and EDS analysis indicated the formation of  $\text{FeSO}_4$  or  $\text{FeSO}_3$  containing products. The local corrosion rate under the clusters were much higher than the reported weight loss corrosion rate as only a small fraction of the surface was attacked. It was not possible to determine these rates accurately.



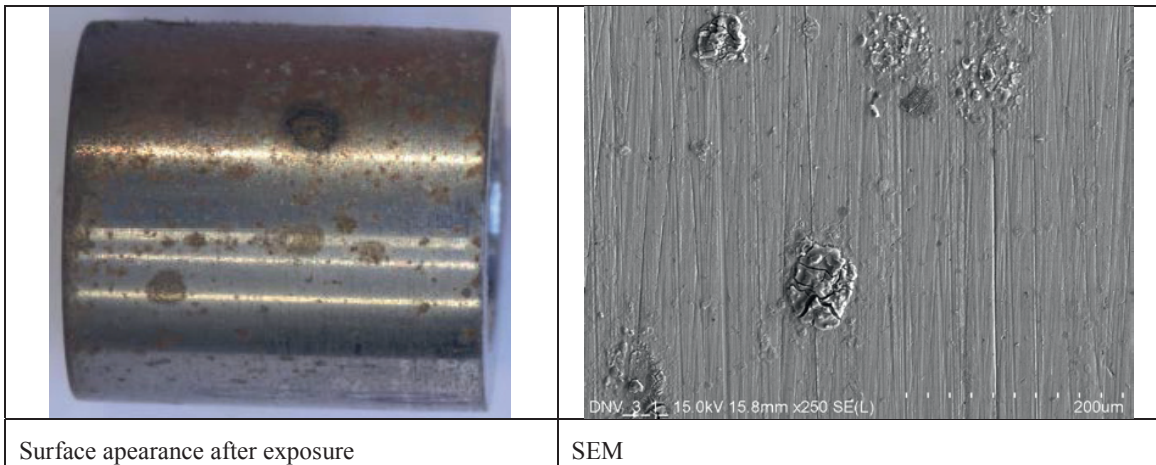


Figure 3 Experiment DnV-3. Temperature 4 °C, 500 ppmv H<sub>2</sub>O, 200 ppmv SO<sub>2</sub>, 500 ppmv O<sub>2</sub>, duration 7 days

The surface was stained, but little attack was observed on the specimens in the H<sub>2</sub>S experiments. Small elemental sulphur particles formed during the exposure as shown in Figure 4.

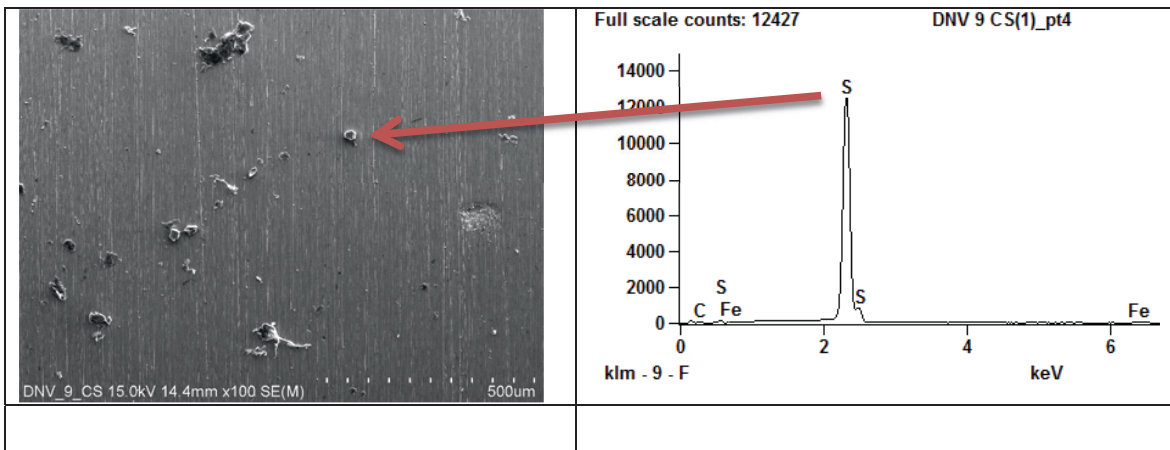


Figure 4 Experiment DnV-5. Temperature 4 °C, 500 ppmv H<sub>2</sub>O, 200 ppmv H<sub>2</sub>S, 500 ppmv O<sub>2</sub> duration 7 days.

Apart from a small area, the steel surfaces looked shiny and non-attacked after exposure to water and O<sub>2</sub> only .

### 3.2. Experiments with 50 ppmv water

Experiments were performed with 50 ppmv water and either NO<sub>2</sub> or SO<sub>2</sub>, with and without O<sub>2</sub>. Corrosion was only observed in experiments with NO<sub>2</sub>. The attack was much less severe than in the experiments with 500 ppmv water. The steel surface was covered with a thin layer of brownish corrosion products.

## 4. Discussion

### 4.1. Worst case corrosion

The consumption rate of impurities due to corrosion depends on the corrosion rate, the steel surface to dense phase CO<sub>2</sub> volume ratio and the corrosion mechanism. The H<sub>2</sub>O, SO<sub>2</sub>, NO<sub>2</sub> consumption will in most cases be 1 mol per 1 mol corroded iron and the consumption rate in a closed system can be high as shown in Figure 5. The consumption might be so fast that it will either be necessary to add controlled amounts of water/SO<sub>2</sub>/NO<sub>2</sub> during exposures lasting more than one day or use a very small steel surface to liquid volume ratio. A steel surface to water volume ratio of 0.02 was used in the present experiments.

The actual consumption rate of impurities was studied in an in-house program at IFE where autoclave experiments carried out under similar experimental conditions as those in the present work. Typically less than 5% of the added impurities were consumed by corrosion in NO<sub>x</sub> and SO<sub>x</sub> experiments before the corrosion rate slowed down. The difference in the impurity concentration at start up and when the experiment is terminated is much larger than the consumption estimated from corrosion (Figure 5). The observation that a large part of the impurities become “non-active” during the exposure can have several reasons:

- Immobilization: The corrosive phase can be trapped in dead legs or wet the autoclave walls preferentially.
- Some of the impurities will react with other impurities and thus slowly change to other products that are less corrosive or more easily immobilised; e.g. formation of elemental sulphur from H<sub>2</sub>S ( $\text{H}_2\text{S} + \text{O}_2 = \text{S} + \text{H}_2\text{O}$ ), oxidation of H<sub>2</sub>SO<sub>3</sub> to H<sub>2</sub>SO<sub>4</sub>, formation of NO<sub>2</sub> from NO and O<sub>2</sub>.
- The corrosion rate slows down with time due to the formation of protective corrosion product layers on the surface. This might to a certain degree explain reduced corrosion with time, but not the reduced concentration of impurities measured when experiments are finished.

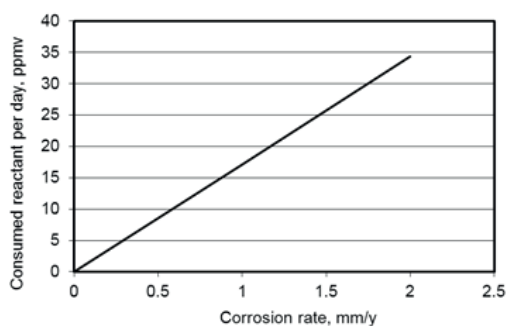


Figure 5. Consumption rate per day of water, SO<sub>2</sub>, and NO<sub>2</sub> when 10 cm<sup>2</sup> steel is exposed in 1 kg dense phase CO<sub>2</sub>. It is assumed that 1 mol Fe consumes 1 mol impurity

Since only a small part of the added impurities were consumed by corrosion, it can be questioned if the measured corrosion rates in the present experiments and in other reported lab experiments reflect the worst case conditions in a pipeline. The experimental observation so far supports the need for a dynamic test system with replenishment of impurities and instant (continuous) analyses of the dissolved impurity concentrations. Such dynamic tests are required in order to define acceptable CO<sub>2</sub> specifications with confidence.



#### 4.2. Solid formation

Small amounts of sulfur formed in the H<sub>2</sub>S experiments. Thermodynamically, the H<sub>2</sub>S-O<sub>2</sub> reaction has the potential to form sulfur at very low H<sub>2</sub>S and O<sub>2</sub> concentrations; i.e. in the ppbv range. The minimum required H<sub>2</sub>S and O<sub>2</sub> concentration, the reaction rates and the trigger mechanism are poorly understood and no literature data have been found for dense phase CO<sub>2</sub> systems. Conversion of 100 ppmv H<sub>2</sub>S will give more than 100 tons of sulfur per year in a 20" pipeline with a flow velocity of 1.5 m/s. It has to be confirmed that massive sulfur formation does not take place under pipeline conditions and the mechanisms that catalyses the sulfur formation therefore needs to be understood and quantified before the safe limit for H<sub>2</sub>S and O<sub>2</sub> can be defined with high confidence.

The amount of solids that forms when e.g., 1 ppmv H<sub>2</sub>O reacts with the pipe wall and form FeCO<sub>3</sub> amounts to 25 t/year and a 0.1 mm corrosion product thick layer on the pipe wall corresponds to about 50 tons solids in a 100 km long 20" pipeline transporting dense phase CO<sub>2</sub> at 1.5 m/s. A key question will be to which degree the corrosion products stick to the wall or become mobilized. Some products might grow on the wall for a long time and eventually spall off. Such products might form larger flakes (mm scale) while other products are more dust-like and detach easily. Dust-like products have been observed particularly in experiments with NO<sub>2</sub>.

#### 4.3. Safe operation window

Defining a safe dense phase CO<sub>2</sub> specification with impurities is challenging due to lack of data and consensus regarding acceptable corrosion and solids formation rates. The experiments with 500 ppmv water show that the impurity concentrations given in the CO<sub>2</sub> specifications suggested by Dynamis and IPCC are too high to be classified as a safe operation window if safe is defined as no corrosion and no solid formation in the bulk phase. If some corrosion and solid formation are acceptable the worst case corrosion needs to be determined and the consequences of solid formation must be assessed.

When the water concentration was reduced from 500 to 50 ppmv, corrosion was observed in experiments with NO<sub>x</sub> only. These results indicate that the suggested impurity concentrations in Table 1 might be acceptable if no NO<sub>x</sub> is present and if the water concentration is reduced to 50 ppmv. It is however too early to conclude as:

- only a few combination of impurities were tested.
- the experiments were performed without replacement of consumed impurities.
- the test duration was short.

The presented results increase the awareness of the effect of impurities on the corrosion behavior, and will be included together with other recent research in the update of DNV-RP-J202 "Design and Operation of CO<sub>2</sub> pipelines" [1]. The objective of RP-J202 is to provide guidance on safe and reliable design, construction and operation of pipelines intended for large scale transportation of CO<sub>2</sub>.

### 5. Conclusion

Field experience and most lab experiments show that dry pure CO<sub>2</sub> and pure CO<sub>2</sub> that contains dissolved water well below the saturation limit in the pure CO<sub>2</sub>-H<sub>2</sub>O system is non-corrosive to carbon steel under transportation pipeline operation conditions.

Only a few percent of the added impurities were consumed for corrosion during the experiments. The impurities apparently became "non-active" during the exposure and it can be questioned if the measured corrosion rates in the present experiments and in other reported lab experiments reflect the worst case conditions in the pipeline for the mixtures tested. The experimental observations so far support the need to use dynamic test system with replenishment of impurities and instant (continuous) analyses of the dissolved impurity concentrations. Such dynamic tests are required in order to define acceptable CO<sub>2</sub> specifications with confidence.

The experiments show that 500 ppmv water gives corrosion and solid formation under most circumstances when NO<sub>2</sub>, SO<sub>2</sub>, H<sub>2</sub>S and O<sub>2</sub> are present in moderate amounts. Reducing the water content to 50 ppmv reduced the corrosion rate and a small amount of corrosion was observed only when NO<sub>2</sub> was present, supporting the philosophy of controlling water content to control corrosion. It is however too early to conclude on the safe operation limit as only a few combinations of impurities were tested, the experiments were performed without replacement of consumed impurities and the test duration was short. Therefore for designs that are likely to contain impurities even with water contents above 50ppmv it is recommended that further experimentation is carried out to ensure acceptable corrosion rates.

Corrosion products and other solid reaction products mobilized in the dense phase CO<sub>2</sub> stream could also potentially impact reservoir injectivity. Therefore where corrosion is expected it is recommended that corrosion products are quantified in terms of the amount that will remain on the steel wall and identify the type and amount of solids that will be mobilized and follow the CO<sub>2</sub> stream.

There is a need to better understand the relation between the water content with carrying levels of the impurities and the impact on corrosion rate. At present, there is a lack of data and therefore it is not possible to define the limits for the various impurity mixtures.

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