

# ACT LAUNCH Project No 299662



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Lowering **A**bsorption process **U**ncertainty, risks and **C**osts by predicting and controlling amine degradation

**Task 5.3 substitute runs for original second-generation solvent tests at TERC (D5.3.1 & D5.3.2):**  
Comparison of NO<sub>2</sub> injection tests (15ppmv and 5ppmv) with the baseline.  
Comparison of N<sub>2</sub> stripping tests with baseline.

Dissemination level		
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## Executive summary

Task 5.3 was aimed at testing a 'second generation' solvent at the Translational Energy Research Centre (TERC) CO<sub>2</sub> capture plant. However, due to health and safety implications with alternative solvents and corrosion issues related to concentrated Monoethanolamine (MEA), it was decided to replace the deliverables D5.3.1 and D5.3.2 with extended tests to demonstrate the effectiveness of degradation countermeasures.

Four test campaigns were conducted for this purpose (one with NO<sub>2</sub> injection and three with N<sub>2</sub> stripping). The N<sub>2</sub> stripping tests were aimed at removing dissolved oxygen (DO) from the solvent while the NO<sub>2</sub> injection (5 ppmv) test was designed to mimic NO<sub>x</sub> removal to make a comparison with an earlier test campaign which was performed with 15 ppmv NO<sub>2</sub> injection.

The four test campaigns performed to replace task 5.3 are compared with a baseline test campaign. The following comparisons are made;

1. Comparison of NO<sub>2</sub> injection tests (15ppmv and 5ppmv) with the baseline
2. Comparison of N<sub>2</sub> stripping tests with the baseline

The three N<sub>2</sub> stripping campaigns were run with different N<sub>2</sub> flow rates. N<sub>2</sub> was injected into the second absorber in series in the solvent flow path (i.e. the 'lower' half of the virtual absorber). During the two test campaigns both absorber columns were used while during one of the test campaigns just one column was used for comparison purposes. Operational data from the plant was gathered. Solvent samples were collected for post analysis to determine degradation product concentrations. However, the samples are yet to be analysed (using new facilities not yet established at the University of Sheffield; there is no scope to analyse under the LAUNCH project) and therefore degradation products data is currently not available.

Comparison of the iron measurements have indicated that NO<sub>2</sub> injection may not have much impact on the solvent degradation under the condition tested while DO measurements have shown that N<sub>2</sub> stripping is very effective in removing DO. However, the detailed implications will not be known until the results of the solvent samples are available and published.



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## 1 Introduction

Translational Energy Research Centre (TERC) at University of Sheffield has a 1 tpd CO<sub>2</sub> capture plant. The plant is fully equipped with instrumentation for environmental and energy performance investigation of generic amines and proprietary solvents. The plant is equipped with a gas sampling system for gas analysis at six different locations and liquid sampling system for solvent analysis at three different locations.

WP5 (Task 5.3) of the LAUNCH project was aimed at testing a second generation solvent at TERC using a gas turbine flue gas composition. However, due to health and safety concerns it was decided to replace the testing of the second generation solvent with further tests on degradation countermeasures.

The plant was operated with 190 m<sup>3</sup>/h of flue gas and 300 kg/h of solvent flow. Flue gas was generated by mixing air with CO<sub>2</sub> from cryogenic storage. The CO<sub>2</sub> concentration in the flue gas was maintained at around 5%.

Four test campaigns were performed as replacements for the original task 5.3;

1. NO<sub>2</sub> injection at 5 ppmv
2. N<sub>2</sub> stripping at 10000 l/h
3. N<sub>2</sub> stripping at 10 l/h
4. N<sub>2</sub> stripping at 600 l/h (half column)

Prior to these test campaigns, earlier in the project a baseline test campaign was performed for comparison purposes. All the four test campaigns reported are compared with the baseline. Moreover, the NO<sub>2</sub> injection test campaign is also compared with an earlier test campaign with 15 ppmv NO<sub>2</sub> injection.

Iron (Fe) concentrations in the solvent over time and Dissolved Oxygen (DO) in the solvent are compared. But the solvent samples collected during the tests reported here cannot be analysed as part of the LAUNCH project as the required equipment is not yet set up at the University of Sheffield, so detailed analysis data for comparison of degradation product formation rates is not yet available for comparison purposes.

Iron measurements have indicated that impact of NO<sub>2</sub> on the solvent degradation under the conditions tested may not be considerable. On the other hand DO measurements have indicated N<sub>2</sub> stripping is an effective tool to remove dissolved oxygen and thus reduce solvent degradation. However, the real impact of removing DO on solvent degradation will be known only once the data of degradation products analysis is available.

## 2 Degradation countermeasure campaigns in TERC

### 2.1 Description of capture plant at TERC

The pilot scale CO<sub>2</sub> capture plant at TERC is capable of capturing 1tpd CO<sub>2</sub> based on 200 Nm<sup>3</sup>/h gas flow having 15% CO<sub>2</sub> i.e. the plant is designed for coal combustion flue gases. The plant is integrated with site combustion facilities including: Grate Boiler/Waste to Energy plant; Gasifier CHP; Biodiesel CHP, Gas Turbine CHP and a visiting/future rigs. It is designed to scrub 100-250 Nm<sup>3</sup>/h of flue/process gas with solvent flows of 300-1600 kg/h based on current packing. The plant can also be fed from a dedicated synthetic gas mixing skid comprising 3 bulk gas streams: CO<sub>2</sub>, N<sub>2</sub> and Air, each of 6-300Nm<sup>3</sup>/h flow range and a trace gas (NO<sub>2</sub>, SO<sub>2</sub>) injection capability; this enables the simulation or modulation of a range of combustion/process gases. A simplified flow diagram of the plant is shown in Figure 1. Equipment specifications are given in Table 1. The plant has a full absorption and desorption cycle and is equipped with two absorber vessels that can be connected in series, a stripper, a reboiler, a cross exchanger, a carbon filter and a water wash. The plant also has a gas pre-treatment section which can be used either as a Flue Gas Desulphurisation (FGD) unit or a Direct Contact Cooler (DCC). The plant has recently been upgraded to include gas humidification control in the DCC. However, during these tests the FG/DCC was bypassed.

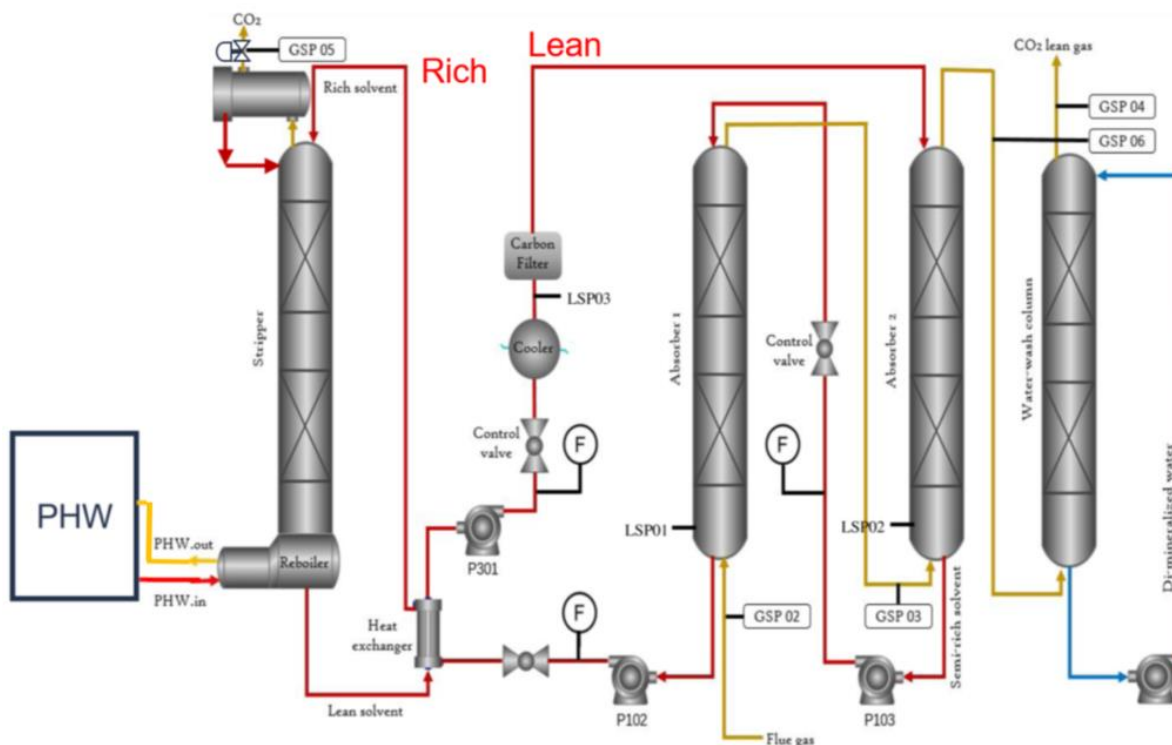


Figure 1: TERC CO<sub>2</sub> capture plant

Two absorber vessels are installed in series to increase residence time and contact between liquid and gas. Each of the absorbers is equipped with two beds of Flexipac 350X structured packing, 3m each. Total packed height, therefore, is 4 beds of 3 m each, so totalling 12 m, with liquid re-distribution at each bed. The stripper is packed with 7 m of IMTP25 random packing. The absorbers have 12 temperature measurement points each for temperature profiling.

Stripping is performed in the stripper and reboiler. The stripper is a 0.3 m diameter column packed with IMTP25 random packing. The reboiler is a shell and tube heat exchanger. Pressurized hot water (PHW)

generated by electrical heating is supplied on the tube side of the reboiler while solvent stays on the shell side.

The PHW has a bypass to control the flow rate through the reboiler or to bypass it. A pneumatically driven 3-way valve is used for this purpose. The energy used for stripping is calculated by measuring the inlet and outlet temperatures and the flow rate of the PHW. Stripper pressure is controlled automatically to a user defined set point by a pneumatic control valve installed at the exit of condenser.

The CO<sub>2</sub> product stream leaving the top of the stripper is passed through a condenser to remove steam and solvent vapours. The condensed liquid is separated from the gas in a reflux drum and is sent back to the stripper through a U-seal mechanism, while CO<sub>2</sub> is exhausted to atmosphere after analysis.

A blower is used to drive the gas through the plant. For this test campaign, air with CO<sub>2</sub> injection, rather than real flue gas, was used, to give enhanced O<sub>2</sub> levels. The tests were performed under general gas turbine conditions so the CO<sub>2</sub> concentration in the absorber entry gas was kept close to 5%.

CO<sub>2</sub> flow was measured by thermal mass flow meters, while the flow rate of gas into the absorber was measured by a pitot type flow meter. Gas composition for mass balance calculations was measured at the inlet and outlet of the absorber, along with temperature and pressure.

Table 1; Absorber and stripper specifications

Specifications	Absorber	Stripper	Water wash
Diameter (mm)	250	300	300
Packing name	Flexipak 350X	IMTP25	IMTP25
Packing type	Structured	Random	Random
Packing height (m)	12	7.5	7.5
Packed beds	4	1	1
Temperature measurements	24	9	-

Table 2 Process and analytical measurements

Analysis	Detail
Main Process parameters	<ul style="list-style-type: none"> <li>• Gas inlet flow, temperature and pressure</li> <li>• Interstage gas temperatures and pressures</li> <li>• Absorber 1 &amp;2 and desorbed temperature profiles and pressure drops</li> <li>• Stripper pressure (reflux condenser) and CO<sub>2</sub> product flow</li> <li>• Liquid flows, temperatures pressures and densities</li> <li>• Reboiler hot water flow; inlet, outlet, core temperature; supply pressure</li> </ul>
Gas analysis	Multipoint sampling and analysis by Gasmeter FTIR: <ol style="list-style-type: none"> <li>1. Absorber 1 column inlet,</li> <li>2. Absorber 2 column inlet,</li> <li>3. Water wash column inlet,</li> <li>4. Water wash outlet;</li> <li>5. Stripper outlet after reflux condenser</li> </ol>
Liquid titrations	Mettler Toledo auto titrator <ol style="list-style-type: none"> <li>1. Fast loop sampling from Absorber 1 (Rich), Absorber 2 (Semi-rich) and Stripper (Lean)</li> <li>2. MEA solvent concentration</li> <li>3. CO<sub>2</sub> concentration and loading</li> </ol>
Dissolved oxygen analysis	Jumo online oxygen analysis <ol style="list-style-type: none"> <li>1. Lean (stripper outlet)</li> <li>2. Semi-rich (absorber 2 outlet)</li> <li>3. Rich (absorber 1 outlet)</li> </ol>

Iron analysis	Analysis on <ul style="list-style-type: none"> <li>Lean (stripper outlet)</li> </ul>
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Process and analytical measurements are described in Table 2. Gas analysis can be performed at 6 different locations in the plant. Sampling lines are located at the FGD inlet, Absorber 1 inlet, Absorber 2 inlet, Water wash inlet and outlet, and Stripper outlet.

The gas samples are extracted from the plant using isokinetic sampling probes and routed to the FTIR through heated filters, heated sampling lines and a heated cabinet housing solenoid for sample switching. The entire sampling system is heated up to 180°C to avoid condensation.

A Gaset DX4000 FTIR is used for gas analysis, which sequentially tests samples from each of the locations. The sequence and sampling time is user defined and can be changed in the FTIR software as and when required. For these tests, gas compositions at Absorber 1 inlet (GSP02) and Absorber 2 outlet (GSP06) were used for overall capture efficiency calculations.

Solvent analysis are performed by an in-line and offline measurements. For online analysis, the Mettler Toledo auto-titrator shown in Figure 2 is used. The apparatus was acquired to be run over the weekends remotely and keep weekend samples for analysis. The apparatus collects three solvent samples (rich, lean and semi-rich). The fast sampling closed loop keeps a small bleed stream of solvent in circulation in respective stream and peristaltic pumps are used to acquire samples when needed. The auto-titrator performs titrations on the three samples for solvent concentration and CO<sub>2</sub> loading analysis. Off line measurements were performed for Fe analysis. Lean samples collected by the auto-titrator were used for Fe analysis using colorimetric method using the apparatus shown in Figure 3.



Figure 2: Mettler Toledo auto-titrator



Figure 3: Fe measurement apparatus



Two optical sensors shown in Figure 4 are installed on lean and rich solvent streams to measure dissolved oxygen. The apparatus was acquired from JUMO. The data is sent to PLC as 4-20mA signal for logging real time DO levels.



Figure 4: DO measurement apparatus



### 3 Data collection

Four different sources of data are collected for post analysis.

1. PLC logs operation parameters of the plant (flows, temperatures, pressures and dissolved oxygen)
2. FTIR analysis and logs flue gas analysis at different location of the plant
3. Auto-titrator analysis and logs data for solvent analysis for rich, semi-rich and lean solvent streams
4. Iron measurement (manual)

PLC, FTIR and auto-titrator data are automatically logged into respective files however Iron measurements data is logged manually. Table 3 provides plant data and important operational parameters logged by PLC, FTIR, titrator for baseline case (See section 3.1).

Table 3 - Data collection for the most important parameters

Parameter	Unit	Mean value
<b>Absorber</b>		
Temperature Gas inlet	°C	37,10
Pressure Gas inlet	mbarg	30,00
Gas inlet flowrate	Nm3/h	190
Gas inlet composition, CO <sub>2</sub>	vol(%) wet	5,2
Gas inlet composition, H <sub>2</sub> O	vol(%)	0,6
Lean solvent inlet temperature	°C	40,0
Lean solvent inlet flowrate	kg/h	300
Solvent inlet composition, MEA	g/100g	39,40
Solvent inlet composition, CO <sub>2</sub>	mol/mol	0,27
Gas outlet to water wash	°C	35,50
Rich solvent outlet temperature	°C	20,0
Rich solvent outlet flowrate	kg/h	300,0
Solvent outlet composition, CO <sub>2</sub>	mol/mol	0,5
Absorber packing height	m	12
Absorber packing type	[-]	350X
Water outlet flowrate	kg/h	2,400
Water wash packing height	m	7
Water wash packing type	[-]	IMTP25
<b>Stripper</b>		
Rich solvent inlet temperature	°C	83,400
Pressure outlet to condenser	barg	0,50
Lean solvent outlet temperature	°C	111,000
Stripper packing height	m	7m
Stripper packing type	[-]	IMTP25
Temperature in reboiler liquid	°C	117
Pressure in reboiler	barg	0,5
Temp CO <sub>2</sub> product from condenser	°C	14,00
<b>Reboiler</b>		
Pressurised Hot Water flow	m3/h	14.5
Pressurised Hot Water pressure	bar g	4 barg
<b>X-changer</b>		

Cold lean outlet temperature	°C	48
Hot rich outlet temperature	°C	84

### 3.1 Approach

A total of 11 test campaigns conducted for the LAUNCH project, details below.

1. Normal operation
2. Higher stripper temperature
3. Higher solvent concentration
4. NO<sub>2</sub> injection (15 ppm)
5. Normal operation repeat
6. Low oxygen (8% O<sub>2</sub>)
7. Higher stripper temperature and pressure
8. N<sub>2</sub> stripping (10000 l/h)
9. NO<sub>2</sub> injection (5 ppm)
10. N<sub>2</sub> stripping (10 l/h)
11. N<sub>2</sub> stripping (600 l/h; single bed)

The plant was operated at the same baseline condition for all the tests. Following table 4 shows the parameters varied during the tests.

Table 4: LAUNCH test campaigns

Campaign	Test	MEA conc	Stripper Temp	Stripper pressure	Oxygen	NO <sub>2</sub> injection	N <sub>2</sub> stripping
		%	C	Barg	%	ppm	l/h
1	Normal operation	35	120	0.5	19	0	0
2	Higher stripper temperature	35	128	0.5	19	0	0
3	Higher solvent concentration	40	120	0.5	19	0	0
4	NO <sub>2</sub> injection 15	35	120	0.5	19	15	0
5	Normal operation repeat	35	120	0.5	19	0	0
6	Low oxygen	35	120	0.5	8	0	0
7	Higher stripper T&P	35	124	0.8	19	0	0
8	N <sub>2</sub> stripping 10000	35	120	0.5	19	0	1000
9	NO <sub>2</sub> injection 5	35	120	0.5	19	5	0
10	N <sub>2</sub> stripping 10	35	120	0.5	19	0	10
11	N <sub>2</sub> stripping 600	35	120	0.5	19	0	600

**Deliverables D5.3.1 and D5.3.2 were replaced with further tests on degradation countermeasures. Last four test campaigns (8-11) in the above table were performed for this purpose.**

For the baseline test campaign (1) the plant was operated for 500 hours with air and CO<sub>2</sub> mixture. The concentration of CO<sub>2</sub> was maintained at around 5% to mimic gas turbine flue gas conditions. Solvent (35% MEA) flow rate was 300 kg/h. Samples were collected for analysis by titration methods for MEA concentration and CO<sub>2</sub> loadings. Samples collected during the earlier six test campaigns (1-6) were sent to SINTEF for analysis of degradation products. In SINTEF, analysis for the degradation products using Liquid chromatography–Mass spectrometry (LC-MS) without derivatization was performed, while Total Inorganic

Carbon-Total Organic Carbon (TIC-TOC) method was used for the measurement of CO<sub>2</sub> in a selection of samples for comparison purposes. A list of the main components and degradation products analysed in this work and the method used are given in Table 5. Samples for the later test campaigns (7-11) have not been analysed yet therefore data for degradation products is not available at the moment for comparison purposes and thus is not included in this report.

Table 5: Details of solvent analysis

Component	CAS	Name	Formula	Analytical method
MEA	141-43-5	Monoethanolamine	C <sub>2</sub> H <sub>7</sub> NO	Titration, LC-MS
CO <sub>2</sub>	124-38-9	Carbon dioxide	CO <sub>2</sub>	Titration
HEEDA/AEEA	111-41-1	2-[(2-aminoethyl) amino]-ethanol	C <sub>4</sub> H <sub>12</sub> N <sub>2</sub> O	LC-MS
HEHEAA	144236-39-5	N-(2-hydroxyethyl)-2-[(2-hydroxyethyl) amino]-acetamide	C <sub>6</sub> H <sub>14</sub> N <sub>2</sub> O <sub>3</sub>	LC-MS
MEA urea	15438-70-7	N,N'-bis(2-hydroxyethyl)-urea	C <sub>5</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub>	LC-MS
HEI	1615-14-1	1H-imidazole-1-ethanol	C <sub>5</sub> H <sub>8</sub> N <sub>2</sub> O	LC-MS
HEF	693-06-1	N-(2-hydroxyethyl)-formamide	C <sub>3</sub> H <sub>7</sub> NO <sub>2</sub>	LC-MS
OZD	497-25-6	2-oxazolidinone	C <sub>3</sub> H <sub>5</sub> NO <sub>2</sub>	LC-MS
HEPO	23936-04-1	4-(2-hydroxyethyl)-2-piperazinone	C <sub>2</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub>	LC-MS
HeGly	5835-28-9	N-(2-hydroxyethyl)-glycine	C <sub>4</sub> H <sub>9</sub> NO <sub>3</sub>	LC-MS
BHEOX	1871-89-2	N1,N2-bis(2-hydroxyethyl)-ethanediamide	C <sub>6</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub>	LC-MS
HEA	142-26-7	N-(2-hydroxyethyl)-acetamide	C <sub>4</sub> H <sub>9</sub> NO <sub>2</sub>	LC-MS
HEIA	3699-54-5	1-(2-hydroxyethyl)-2-imidazolidinone	C <sub>5</sub> H <sub>10</sub> N <sub>2</sub> O <sub>2</sub>	LC-MS
Fe	7439-89-6	Iron	Fe	Colorimetry

### 3.1.1 NO<sub>2</sub> injection:

In order to assess the impact of NO<sub>x</sub> on the solvent degradation, NO<sub>2</sub> was injected at rate of 15 ppmv (Campaign 4). NO<sub>2</sub> was bought from BOC in 200 bar cylinders at 3000 ppm in air. A cost comparison was made for different NO<sub>2</sub> concentration and pressures and the above concentration and pressure was found to be the most suitable in terms of economics and personal safety. NO<sub>2</sub> detectors were used during the tests to detect leaks in the system (around the cylinder) and associated pipework.

For a gas flow rate of 190 m<sup>3</sup>/h into the absorber, ~ 20 cylinders were required to maintain a NO<sub>2</sub> concentration of 15 ppmv over the duration of the test campaign (150 hrs).

A follow-on test campaign was performed at a NO<sub>2</sub> injection rate of 5 ppmv to mimic NO<sub>x</sub> removal (countermeasure). For this test campaign ~7 cylinders were required to maintain 5 ppmv NO<sub>2</sub> in 190 m<sup>3</sup>/h gas flow over ~150 hrs test duration.

As mentioned previously, a mixture of air and CO<sub>2</sub> was used as flue gas for these tests. The air was sucked in by a blower. CO<sub>2</sub> from cryogenic storage tank, metered by gas mixing skid was injected into the air stream at the inlet side of the blower. NO<sub>2</sub> stream was injected into the mixed stream (air + CO<sub>2</sub>) upstream of the blower. The flow rate of the NO<sub>2</sub> stream was not measured but total flow rate of the flue gas stream was controlled to 190 m<sup>3</sup>/h via the PLC program using VSD control. NO<sub>2</sub> concentration was measured in the flue gas using FTIR at the inlet of the absorber. Figure 5 shows schematic of the set up used for NO<sub>2</sub> injection. Three cylinders connected in series were used to run the plant overnight. N<sub>2</sub> purge was provided to purge the lines before disconnected and connecting cylinders. Needle valves, shown as blue in the diagram, were used to control the NO<sub>2</sub> stream to adjust the NO<sub>2</sub> concentration in the flue gas stream to the desired levels (15/5 ppm).

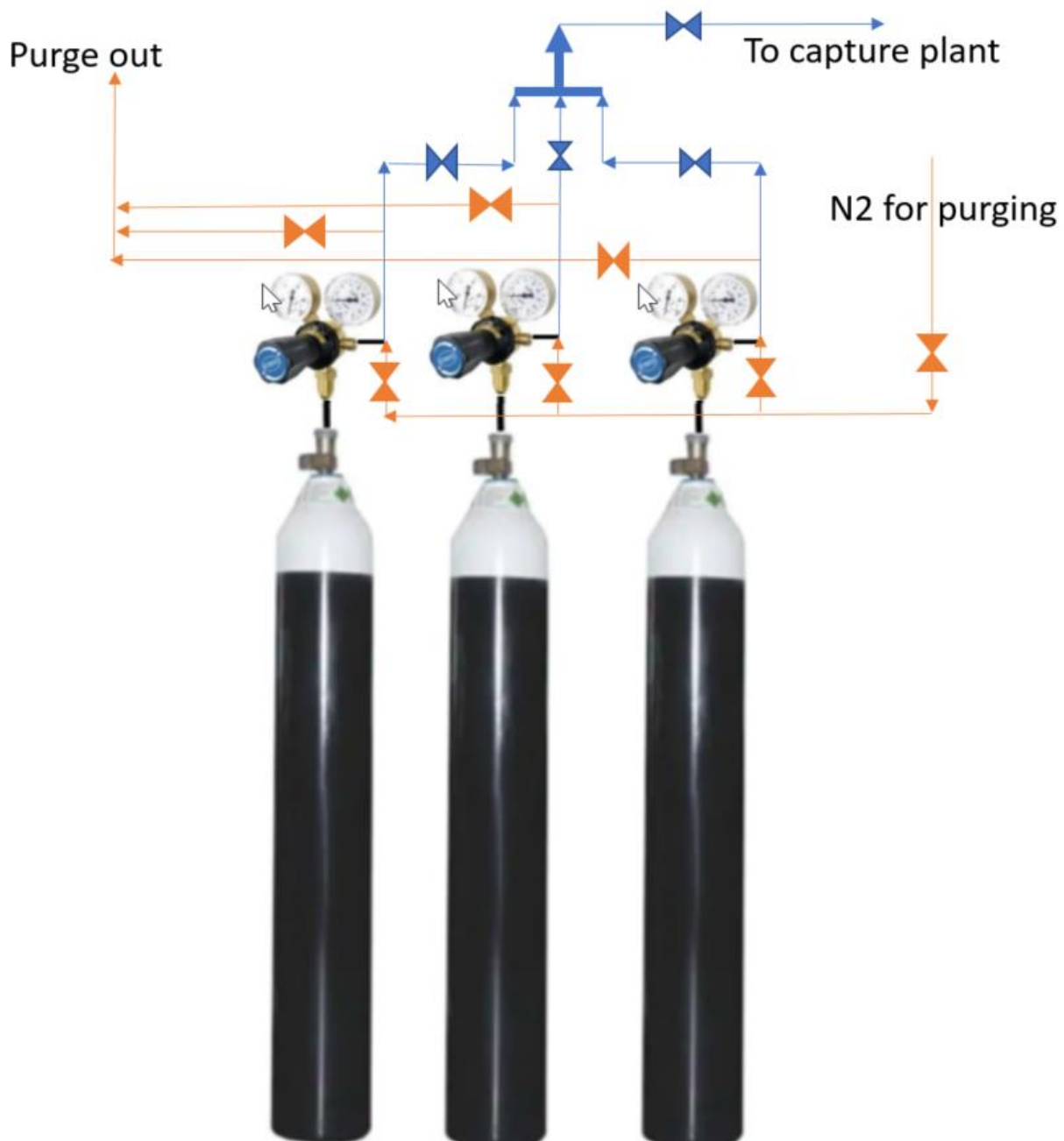


Figure 5: NO<sub>2</sub> injection set up

### 3.1.2 N<sub>2</sub> stripping:

The capture plant has two absorber columns. Under standard operation, solvent flows from the Stripper to Absorber 2, then to Absorber 1 and back to the Stripper while flue gas first enters Absorber 1 then flows to Absorber 2 and then to the Water Wash. For these tests, flue gas was fed to Absorber 2 and then to the Water Wash while N<sub>2</sub> was injected at the base of Absorber 1 where rich solvent from Absorber 2 (first in solvent flow sequence) was contacted with N<sub>2</sub> (Figure 6). The N<sub>2</sub> stream mixed with dissolved oxygen removed from the solvent joins the flue gas stream it enters the Water Wash.

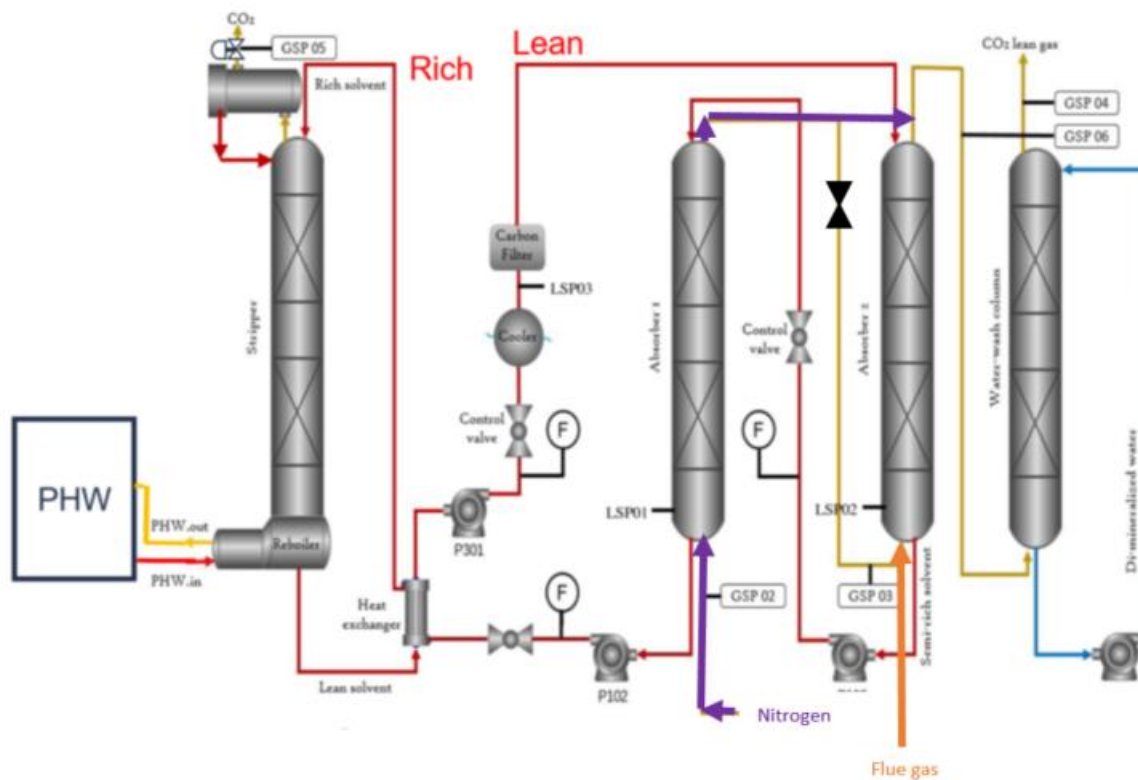


Figure 6: N<sub>2</sub> stripping set up

## 4 Results

This section covers the results obtained during the test campaigns. The first test campaign of LAUNCH (Work Package 4.1.1) was performed with 35% MEA solution. This test campaign was termed as 'normal operation'. After solvent analysis for test campaigns (1-4) it was decided to repeat the 'normal' test campaign (Campaign 5) for a limited number of hours to verify the results. Therefore, in the following sections the results from Campaign 5 as baseline are compared with the other test campaigns.

In order to make better understanding of the results and make a reasonable comparison, the following test campaigns are compared in the following sections.

**Section 4.1: NO<sub>2</sub> injection, comparison of Campaigns 9 and 4 with Campaign 5 (normal).**

**Section 4.2: N<sub>2</sub> stripping, comparison of Campaigns 8, 10 and 11 with Campaign 5 (normal).**

### 4.1 NO<sub>2</sub> injection:

#### 4.1.1 Iron (Fe)

The iron (Fe) content of the solvent was measured throughout the test campaigns. The plot of Fe content vs. operational hours is shown in Figure 7.

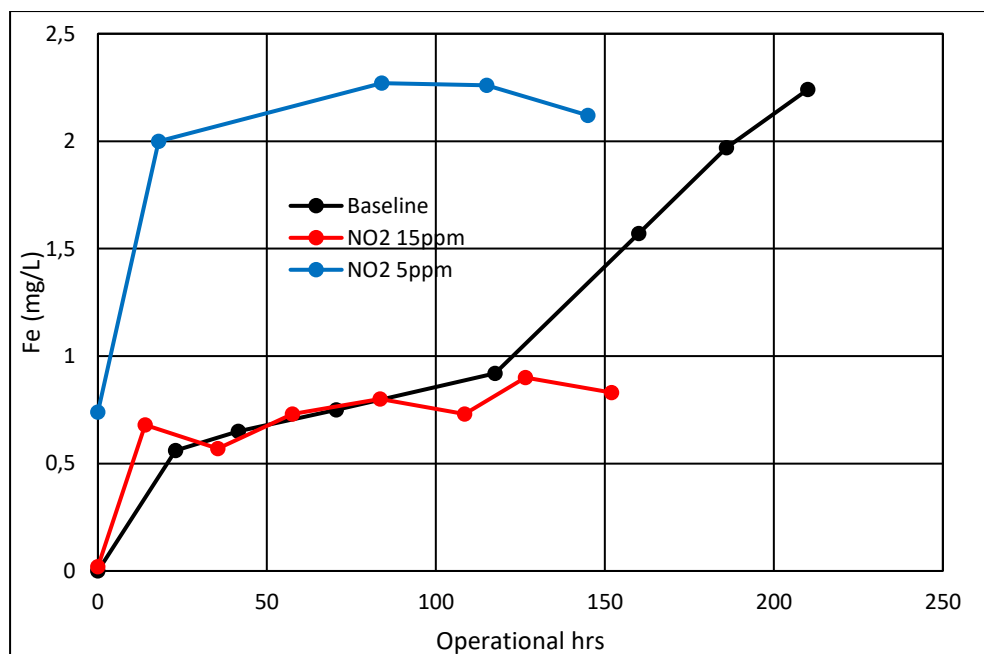


Figure 7: Iron measurements over the test period

The plot shows that iron concentration in the solvent does not change much during the tests. Iron concentration during the baseline and 15ppm campaigns was measured to be of a similar order and similar steady trend. However, Fe is measured higher for the 5ppm injection case as compared to the other two campaigns. The plot indicates that the iron concentration during the 5ppm campaign was measured higher at

the start. This could be due to the iron left in the plant from the previous test. The solvent was drained and replaced with fresh tests but extensive cleaning did not take place.

#### 4.1.2 Dissolved oxygen:

Dissolved oxygen was measured in rich and lean solvent streams using online measurement optical probes. The DO content in the lean stream was measured as zero so is not plotted. Figure 8 below shows DO concentration for the rich stream for the NO<sub>2</sub> injection campaigns in comparison with the baseline campaign for a typical 24 hr period. The data is only shown for a typical 24 hrs period as the data was very consistent throughout the test campaigns with only slight variation.

The plot shows that the DO content in the both cases of NO<sub>2</sub> injection tests was similar but was slightly higher than the baseline case.

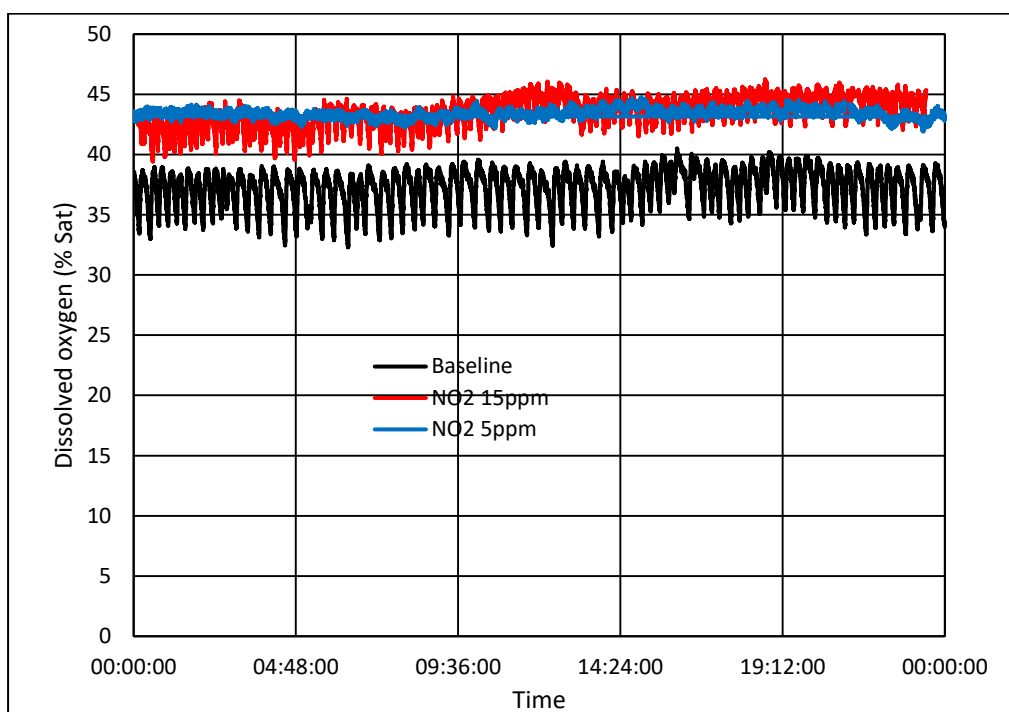
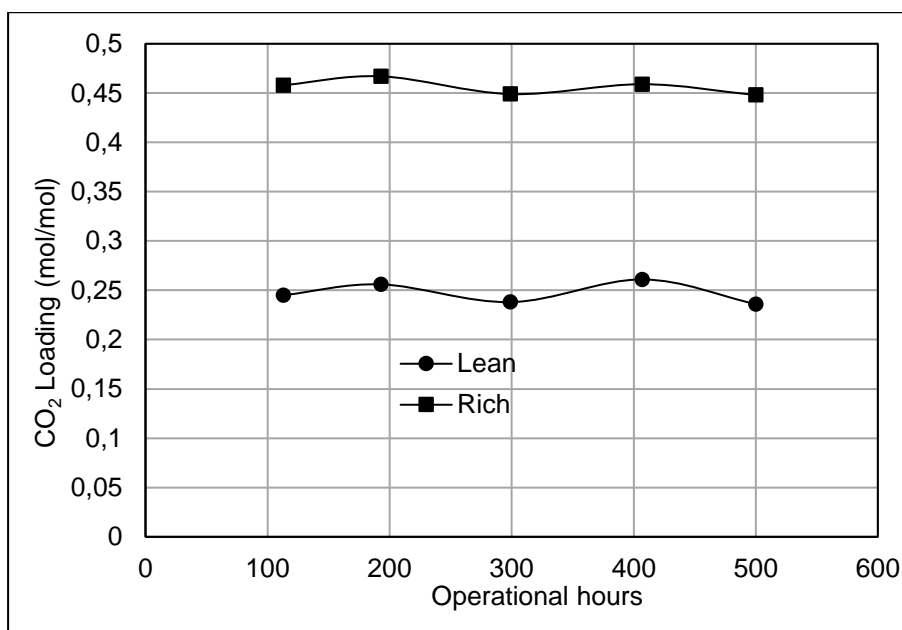


Figure 8: Dissolved oxygen comparison for NO<sub>2</sub> injection tests with baseline

#### 4.1.3 Solvent analysis:

Samples were collected regularly throughout the test campaigns. CO<sub>2</sub> loadings data for the samples analysed for degradation products for the baseline test campaign (1) is presented in Figure 9. The figure indicates that rich and lean loadings were measured in the range of 0.45-0.46 mol/mol and 0.24-0.26 mol/mol, respectively. Similar data was obtained for the NO<sub>2</sub> injection test campaigns as plant operational parameters were similar.





**Figure 9: CO<sub>2</sub> loadings data for the samples analysed**

Samples collected for degradation product analysis are yet to be analysed so comparative data is not available and therefore not presented in this report.

## 4.2 N<sub>2</sub> stripping:

Three test campaigns were conducted with N<sub>2</sub> stripping.

Campaign 8: 10,000 l/h

Campaign 10: 10 l/h

Campaign 11: 600 l/h

Campaign 8 (10,000 l/h N<sub>2</sub> flow) was designed to feed to ~ 4.5 % N<sub>2</sub> of the total gas flow. Campaign 10 was designed based on calculations to achieve 21% O<sub>2</sub> in the N<sub>2</sub> stream. However, the flow rate of the N<sub>2</sub> stream in this case was too low for FTIR sampling therefore a third campaign (11) was designed to feed N<sub>2</sub> at rate just enough for FTIR measurements.

### 4.2.1 Iron (Fe)

Figure 10 shows that iron concentration during the N<sub>2</sub> stripping tests were slightly higher than the baseline case. However, there is common trend in the N<sub>2</sub> stripping cases that there was sharp increase in iron concentration during the first 50 hrs of testing which then stayed reasonably steady during the course of the test. This suggests that there may be residual iron in the plant from previous tests which dissolved in the solvent at the start. Beyond this, however, dissolved Fe did not increase considerably during the tests due to potentially limited solvent degradation, and hence a reduced increase in solvent corrosivity, as a result of oxygen removal.

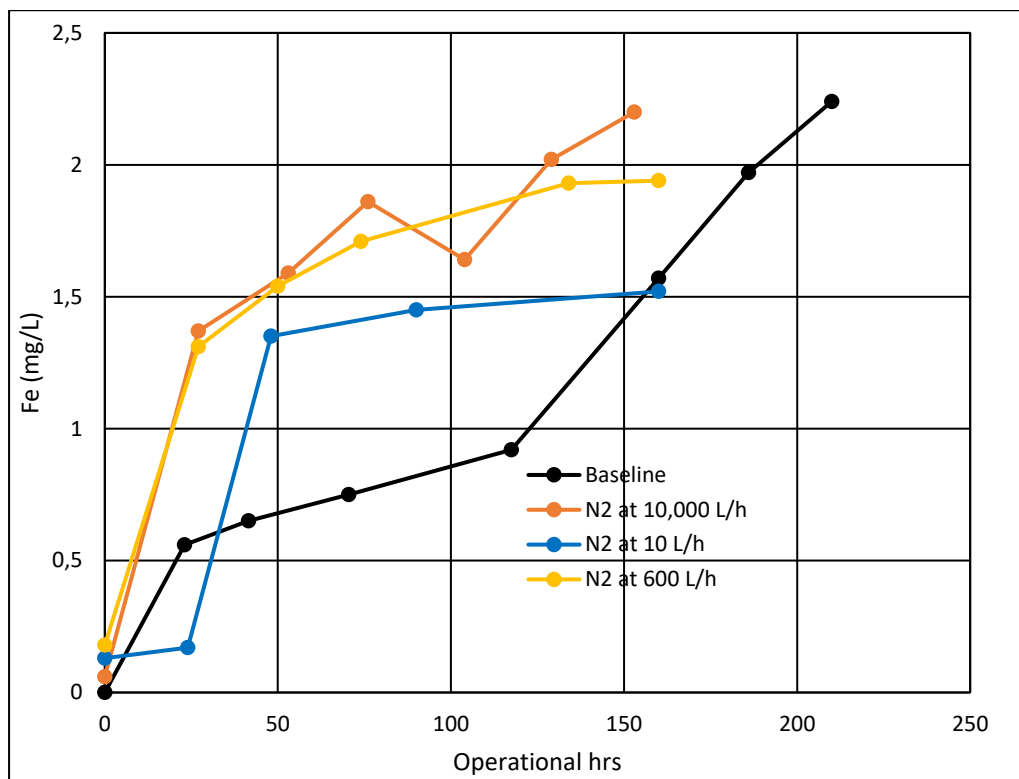


Figure 10: Iron measurement comparison of N<sub>2</sub> stripping tests with baseline

#### 4.2.2 Dissolved oxygen:

Figure 11 shows a plot of dissolved oxygen measurements at Absorber 1 exit (rich stream) during the N<sub>2</sub> stripping test campaigns in comparison with the baseline. It is to be noted that for the baseline case solvent came in contact with flue gas in both absorbers. However, in the case of N<sub>2</sub> stripping Absorber 1 used for N<sub>2</sub> injection, thus solvent only came in contact with flue gas in Absorber 2. For the 1000 l/h and 10 l/h tests the whole Absorber 1 packing (6m) was used (i.e. the solvent was fed from the top) while for the 600 l/h test only half of the Absorber 1 packing (3m) was used (i.e. the solvent was fed half way down).

The figure shows that N<sub>2</sub> stripping has significant impact in removing DO from the solvent in all the three tests. For the 10 l/h N<sub>2</sub> injection there was still some DO left in the solvent indicating that the N<sub>2</sub> flow was not enough but still around 90% DO was removed. For the other two cases the residual DO level in the solvent was very low.

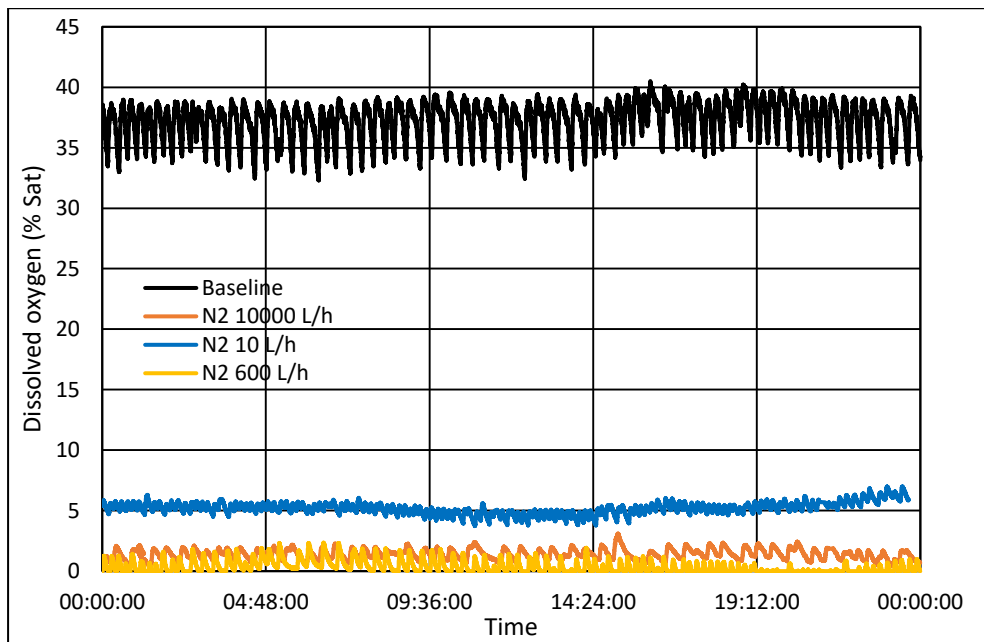


Figure 11: Comparison of DO for N<sub>2</sub> stripping cases with the baseline

#### 4.2.3 Solvent analysis

Solvent analysis were performed for the MEA concentration and CO<sub>2</sub> loadings. It was observed that rich loadings measured for the three N<sub>2</sub> stripping test campaigns were almost the same at the exit of the two absorbers indicating that N<sub>2</sub> stripping at the flow rates tested did not desorb much CO<sub>2</sub>.

The samples collected for post analysis are being still to be analysed thus comparison of degradation products is not provided here.



## 5 Conclusions

After internal discussions deliverables 5.3.1 and 5.3.2 related to testing a 'second generation' solvent at the TERC plant were replaced with tests on countermeasures for solvent degradation (removal of dissolved oxygen and NO<sub>x</sub>). Two test campaigns were conducted with NO<sub>2</sub> injection at 5ppm and 15 ppm, the former being designed to mimic NO<sub>x</sub> removal. Three test campaigns were performed with N<sub>2</sub> stripping to remove DO.

The data available (Fe measurements) indicates that there may not be much impact of NO<sub>2</sub> injection rate variation from 0 to 15 ppm on solvent degradation. However, firm conclusions cannot be drawn until samples are analysed for degradation product levels.

In addition, N<sub>2</sub> stripping through a relatively short (3m) packed column has been shown to be an effective tool to remove DO. However, its impact on solvent degradation has yet to be determined, again waiting until data from solvent analyses for degradation products is available.