

Executive summary

The goal of WP4 is to develop the LAUNCH solvent qualification program. The qualification program will be developed using the lab scale LAUNCH Rig#2 (25 kgCO₂/day) from TNO and the TERC pilot scale unit ($1tCO_2/day$).

Prior to the tests, a comparison of rig characteristics and instrumentation was done to ensure that similar trends could be expected, and comparable results collected. The operational parameters to be used were agreed between TNO and TERC. In these tests LAUNCH rig#2 (TNO) and TERC (UnivSheff) were operated with the same cMEA (30-35% w/w) solvent for a total of approximately 500 hours operating time. A synthetic flue gas representative of gas turbine flue gas was used. The use of a synthetic mixture enabled a direct comparison between rigs. The gas was composed of air, CO₂ and water. Impurities were out of the scope of this activity since they were investigated in other subtasks within WP4.

The first tests done at TNO using 30%wt MEA in LAUNCH rig#2 had a duration of 528 hours continuously. The operation of LAUNCH rig#2 at TNO was done continuously using artificial flue gas (air + CO₂) and samples of the solvent were taken every day for analysis. A Fourier-transform Infrared Spectrometer (FTIR) was constantly connected to the outlet of the absorber to monitor the composition of the outlet gas stream.

It was found that MEA concentration demonstrated a decreasing behaviour, which is an indication of solvent degradation. Indeed, formic acid and oxalic acid, which are typical oxidative degradation products, were formed and their concentration increased linearly with time. Formic acid was detected at higher concentrations than oxalic acid, while acetic acid was not detected. As far as metals' concentration is concerned, chromium, nickel and iron in the solvent were found to be increasing during the course of the campaign. Nickel and chromium values were similar at around 1-2 mg/kg, while iron concentration was significantly higher at around 20 mg/kg. Nitrite (NO⁻₂) and nitrate (NO⁻₃) concentrations were also measured as indications of degradation via ammonia oxidation as well as a comparison basis for the planned accelerated degradation campaign via NOx addition. Both anions were detected, and it was observed that they had opposing trends, i.e. the higher the nitrate, the lower the nitrite concentrations. The results were used for the comparison with the operation of the TERC facility to validate LAUNCH rig#2 as a relevant testing rig for degradation tests.

The TERC pilot plant was operated for 500 hrs with synthetic flue gas comprising of CO_2 and air with 35% MEA as solvent. Gas analysis were measured at different locations using FTIR while solvent analysis was performed using Mettler Toledo auto-titrator. The plant was cleaned using a comprehensive cleaning procedure due the reason that plant was moved from the old site as a result of flooding and some of the instrumentation and piping had to be replaced. The plant was passivated post cleaning with different weak concentrations of MEA before starting the test campaign with 35% MEA.

Iron content, as an indication of degradation, of the solvent was measured at least once a day except weekends using colorimetric method. No other metals were monitored. Samples were collected throughout the test campaign for post analysis. Some of the samples, almost every hundred hours, were analysed by LC-MS at SINTEF for commonly known degradation products such as HEA, HEI, HEF, HEPO, HeGIy etc.

The results of the analysis have indicated that degradation is a complex phenomenon. There are no clear trends to conclude regarding the differences between the small 25 kg/day TNO's LAUNCH #2 rig and relatively larger 1000 kg/day TERC pilot plant. It is concluded that more work needs to be done to better understand the degradation phenomenon and the factors driving them.



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1 Introduction

The goal of WP4 is to develop the LAUNCH solvent qualification program. The qualification program will be developed using the lab scale LAUNCH Rig#2 (25 kgCO₂/day) from TNO and the TERC pilot scale unit ($1tCO_2/day$).

The ability to control degradation is necessary for guaranteeing long-term stable operation of CO₂ capture plants. Many elements affect the solvent degradation rate, such as oxygen, NO₂, iron and temperature. Within WP4 a solvent qualification program will be developed by performing activities using two different LAUNCH rigs: LAUNCH rig#2 (up to 25 kgCO₂/day) and the TERC facility (1 tonCO₂/day). The main objective of the program is to qualify the LAUNCH rigs as tools for evaluating solvent degradation.

This qualification is performed within Task 4.1 Qualification of small scale LAUNCH rigs approach, by running a given set of experiments in TERC and the LAUNCH rig#2, at the same controlled operational conditions, with the objective to show that the degradation trend between the two units is comparable. These include operating at normal and accelerated degradation conditions. This report presents the results of the first campaigns for both LAUNCH rig#2 and TERC facility and their comparison. The accelerated conditions will be reported as part of the deliverable 4.2.1. The qualification will require that the solvent loss is comparable between both units (less than 15% difference), as well as the trends in formation of degradation products.

This report presents the operational parameters and degradation results in the campaigns performed with the two units, as well as a comparison of the trends observed between the LAUNCH Rig#2 and the much larger TERC pilot scale unit.

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2 Data collection

Preceding the operation of LAUNCH rig#2, TNO and TERC elaborated a data collection file in order to share and agree on the operational parameters that should be used in both campaigns. This way it can be ensured that both plants could operate using similar settings and, thus, enabling the final comparison between LAUNCH rig #2 and TERC plant. Since not all the parameters were available, Table 1 shows the compilation of the available parameters to be used for the comparison of the rigs and for the operation, while the complete data list can be found in Appendix A.

Parameter	Unit	Mean value			
Absorber					
Temperature Gas inlet	۵°	37,10			
Pressure Gas inlet	mbarg	30,00			
Gas inlet flowrate	Nm3/h	189			
Gas inlet composition, CO2	vol(%) wet	5,19			
Gas inlet composition, H2O	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, CO2	g/100g	7,70			
Solvent inlet composition, CO2	mol/mol	0,27			
Gas outlet to water wash	°C	35,50			
CO2 outlet to water wash	vol(%) wet	0,57			
H2O outlet to water wash	vol(%)	5,7			
Rich solvent outlet temperature	°C	20,0			
Rich solvent outlet flowrate	kg/h	300,0			
Solvent outlet composition, MEA	g/100g	37,3			
Solvent outlet composition, water	g/100g	12,6			
Solvent outlet composition, CO2	mol/mol	0,5			
Absorber packing height	m	12			
Absorber packing type	[-]	350X			
Liquid volume in absorber sump	L	74			
Water w	ash				
Water inlet flowrate	kg/h	2400			
Gas outlet to vent	O°	31,00			
MEA outlet to vent	ppm	3,000			
NH3 outlet to vent	ppm	32,000			
Water outlet flowrate	kg/h	2,400			
Water wash packing height	m	7			
Water wash packing type	[-]	IMTP25			
Stripper					
Rich solvent inlet temperature	۵°C	83,400			
Pressure outlet to condenser	barg	0,50			
Lean solvent outlet temperature	٥C	111,000			
Stripper packing height	m	7m			
Stripper packing type	[-]	IMTP25			
Temperature in reboiler liquid	°C	117			
Pressure in reboiler	barg	0,5			

Table 1 - TERC data collection for the most important parameters



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Temp CO ₂ product from condenser	°C	14,00				
Reboiler						
Steam	kg/h	14.5 m3/h				
Steam	bar g	3.5 barg				
Cold lean outlet temperature	°C	48				
Hot rich outlet temperature	°C	84				
Hot lean inlet temperature	°C	111				
Addition of process water	kg/h	3,60				

In order to maintain the water balance, the same water input as at the TERC facility was used for LAUNCH rig#2 in order to calculate the CO₂ concentration in dry basis that should be used in the flue gas inlet. Along with that, during operation, the amount of water lost in the system was collected in a condensation vessel and pumped back to the plant frequently to avoid variations in the amine concentration.

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3 Degradation campaign with LAUNCH rig#2

3.1 Description of LAUNCH rig#2

TNO's LAUNCH rig#2 is a CO₂ capture plant (5 Nm³/h flue gas capacity) which allows for 24/7 continuous operation of the system. It enables tests of different solvents, multiple technologies for solvent management (oxygen removal, iron removal) and process quality control under realistic conditions at TRL5. The rig can be operated with artificial or real flue gas; however this campaign was done with artificial flue gas in a composition similar to that from a gas turbine. The gas inlet is controlled by mass flow controllers and an evaporator is connected to the lines to guarantee that the flue gas is saturated with water prior to entering the absorber column.

The gas outlet of the absorber column is connected to a flow meter and a Fourier-transform Infrared Spectrometer (FTIR) to allow for quantification of emissions and also enable the calculation of capture rate. On the stripper side, an electrical heater is used in the reboiler and the gas outlet is monitored with the use of a mass flow meter and a CO_2 analyser. The rig was not originally equipped with water washes, however, it is been revamped to include water washes in the absorber and stripper side and also a quench in the inlet. Figure 1 illustrates LAUNCH rig#2 at TNO.



Figure 1 - LAUNCH rig#2 at TNO

The main characteristics of the miniplant are presented in table 2.

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Specifications	Absorber	Stripper
Diameter (mm)	44.96	44.96
Packing name	Sulzer BX SS Sulzer BX	
Packing type	Structured	Structured
Packing height (m)	4.3	2.04
Temperature measurements	15	5

Table 2 - Absorber and stripper specifications

3.2 Approach

The main goal of the campaign was to operate the plant as close to the operating profile of TERC and maintain stability during its course.

Since the start of LAUNCH rig#2's operation, lean and rich loaded samples (~20mL each) were withdrawn every 2-3 days. These samples were analysed for amine, water and CO₂ concentration using FT-IR method by TNO. By analysing the time series of available samples, several of them were analysed for degradation products and metals in order to provide information on the level of degradation undergone as well as corrosion. All samples were stored in sealed vials and at low temperature, in order to ensure that any possible (thermal) degradation reactions taking place, would be terminated.

The analysis was conducted by both TNO and SINTEF. Since there is a plethora of degradation products making it impossible to analyse for every one of them, a selection was made based on the operating conditions and on which products would provide the most valuable information.

In TNO, five different analytical methods were used for the analysis: phosphoric acid titration (PA) and FTIR for CO₂, FTIR and Ion Chromatography (IC) for MEA, IC for degradation products (acids), Karl-Fischer titration (KF) and FT-IR for water and Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) for metals. Phosphoric acid and FTIR analysis were done in TNO laboratories in Delft while Karl-Fischer, IC and ICP-MS were performed at TNO laboratories in Petten. The degradation products analysed with IC were acetic acid, formic acid and oxalic acid while ammonium was also analysed. The metals analysed were Cr, Fe, Ni, Mn, Cu, Zn, Mo, Ba and Pb.

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 CO₂ in a selection of samples for comparison purposes. Alkylamine/ammonia/aldehyde were not prioritized since it was decided that the focus would be in the lean solvent samples. It was chosen to focus on the nitrosamine that has been shown to be present in large amount for MEA (NHEGly) to limit the number of components. This is also supported by available data from MEA campaign at TCM (Anne K. Morken et al., 2014). In Table 3, a list of the main components and degradation products analysed in this work and the method used are given.

Abbreviation	CAS	Name	Formula	Analysis method
MEA	141-43-5	Monoethanolamine	C2 H7 N O	FT-IR, IC, LC-MS
H ₂ O	-	Water	H2O	FT-IR, KF
CO ₂	124-38-9	Carbon dioxide	CO2	FT-IR, PA, TIC-TOC
Formic acid	64-18-6	Methanoic acid	C H2 O2	IC
Acetic acid	64-19-7	Ethanoic acid	C2 H4 O2	IC
Oxalic acid	144-62-7	Ethanedioic acid	C2 H2 O4	IC
Nitrate	14797-55-8	Nitrate	NO ₃ -	IC
Nitrite	14797-65-0	Nitrite	NO ₂ -	IC

Table 3 – Overview of components analysed in this work and corresponding analytical method



HEEDA/AEEA	111-41-1	2-[(2-aminoethyl)amino]- ethanol	C4 H12 N2 O	LC-MS
HEHEAA	144236-39-5	N-(2-hydroxyethyl)-2-[(2- hydroxyethyl)amino]- acetamide	C6 H14 N2 O3	LC-MS
MEA urea	15438-70-7	N,N'-bis(2-hydroxyethyl)- urea	C5 H12 N2 O3	LC-MS
HEI	1615-14-1	1H-imidazole-1-ethanol	C5 H8 N2 O	LC-MS
HEF	693-06-1	N-(2-hydroxyethyl)- formamide	C3 H7 N O2	LC-MS
OZD	497-25-6	2-oxazolidinone	C3 H5 N O2	LC-MS
HEPO	23936-04-1	4-(2-hydroxyethyl)-2- piperazinone	C6 H12 N2 O2	LC-MS
HeGly	5835-28-9	N-(2-hydroxyethyl)-glycine	C4 H9 N O3	LC-MS
BHEOX	1871-89-2	N1,N2-bis(2-hydroxyethyl)- ethanediamide	C6 H12 N2 O4	LC-MS
HEA	142-26-7	N-(2-hydroxyethyl)- acetamide	C4 H9 N O2	LC-MS
HEIA	3699-54-5	1-(2-hydroxyethyl)-2- imidazolidinone	C5 H10 N2 O2	LC-MS
NHEGly	80556-89-4	2-[(2-hydroxyethyl) nitrosoamino]-acetic acid	C4 H8 N2 O4	LC-MS
Cr	7440-47-3	Chromium	Cr	ICP-MS
Fe	7439-89-6	Iron	Fe	ICP-MS
Ni	7440-02-0	Nickel	Ni	ICP-MS
Mn	7439-96-5	Manganese	Mn	ICP-MS
Cu	7440-50-8	Copper	Cu	ICP-MS
Zn	7440-66-6	Zinc	Zn	ICP-MS
Мо	7439-98-7	Molybdenum	Мо	ICP-MS
Ва	7440-39-3	Barium	Ba	ICP-MS
Pb	7439-92-1	Lead	Pb	ICP-MS

3.2 Results

The results of the campaign are provided in this section. They are divided to two parts, one dedicated to the operational parameters and one focused on the analysis results.

3.2.1 Operation

The LAUNCH rig#2 was operated continuously for 528 hours in total (22 days), excluding preparations and leakage control. Overall, the operation was very stable with minor adjustments, mainly related to maintaining the water balance in the rig. As already noted, though, the rig was not equipped with water washes in either absorber or desorber during this campaign. Table 4 shows the operation parameters of the LAUNCH rig averaged for the duration of the campaign. The L/G ratio used in LAUNCH rig #2 was 1.5 kg/kg.

Parameter	Unit	Mean value
Absorber		
Temperature Gas inlet	С°	37,8
Pressure Gas inlet	barg	54,20
Air inlet flowrate	nL/h	4224
O2 inlet flowrate	nL/h	887
CO ₂ inlet flowrate	nL/h	247



Inlet flowrate dry total	nL/h	4471
H ₂ O inlet flowrate	g/h	235
H ₂ O inlet flowrate	nL/h	293
Inlet flowrate wet total	nL/h	4765
Gas inlet composition, CO2	vol(%) dry	5,53
Gas inlet composition, CO2	vol(%) wet	5,19
Gas inlet composition, O2	vol(%) dry	19,8
Gas inlet composition, O2	vol(%) wet	18,6
Gas inlet composition, H2O	vol(%)	6,2
Lean solvent inlet temperature	°C	39,7
Lean solvent inlet pressure	barg	524,000
Lean solvent inlet flowrate	kg/h	11,5
Lean solvent density	kg/m³	1080
Lean solvent outlet flowrate	m³/h	0,011
Gas outlet temperature	С°	43,4
Pressure outlet	barg	26,7
Rich solvent outlet temperature	°C	40,9
Rich solvent outlet pressure	barg	1180
Rich solvent outlet flowrate	kg/h	12,1
Rich solvent density	kg/m ³	1110,0
Rich solvent outlet flowrate	m³/h	0,011
abs T profile (top – 0,29 m packing depth)	°C	57,2
abs T profile (0,57 m packing depth)	°C	59,8
abs T profile (1,15 m packing depth)	°C	61,5
abs T profile (1,43 m packing depth)	°C	61,2
abs T profile (1,72 m packing depth)	°C	60,9
abs T profile (2,01 m packing depth)	°C	59,8
abs T profile (2,29 m packing depth)	°C	58,9
abs T profile (2,58 m packing depth)	С°	57,7
abs T profile (2,87 m packing depth)	°C	56,5
abs T profile (3,15 m packing depth)	°C	54,7
abs T profile (3,73 m packing depth)	С°	50,5
abs T profile (bottom – 4,30 m)	С°	47,8
Liquid level in the absorber sump	%	20,68
Liquid volume in absorber sump	m ³	0,000006
Temperature in the absorber sump	°C	Not available
Pressure outlet to vent	barg	26,7
MEA outlet to vent	mg/Nm ³	179,5
NH ₃ outlet to vent	mg/Nm ³	9,6
Stripper		
Rich solvent inlet temperature	°C	93,1
Rich solvent inlet pressure	barg	1060
Gas outlet to condenser	°C	88,80
Pressure outlet to condenser	barg	802,00
Lean solvent outlet temperature	°C	Not available
Lean solvent outlet pressure	barg	666
stripper T profile (top – 1,11 m packing depth)	°C	93,9
stripper T profile (1,96 m packing depth)	С°	95,2
stripper T profile (2,81 m packing depth)	°C	96,2



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stripper T profile (3,66 m packing depth)	С°	101,0			
stripper T profile (bottom – 4,25 m)	С°	114,0			
Reboiler duty	kW	0,88			
Liquid level in the reboiler	%	44,83			
Liquid volume in reboiler	m ³	0,005			
Temperature in reboiler liquid	С°	120,09			
Pressure in stripper	barg	0,75			
Temp CO ₂ product from condenser	С°	14,76			
Flow CO ₂ product from condenser	L/h	227,93			
	nL/h	216,24			
Cross heat exchanger					
Cold rich inlet temperature	С°	39,9			
Cold lean outlet temperature	С°	42,5			
Hot rich outlet temperature	О°	99,9			
Hot lean inlet temperature	°C	114,4			

As far as stability of operation is concerned, it can be evaluated following the progress of the operational parameters along the campaign. For example, by following the temperature profile along the absorber, as shown in Figure 2, one can see that the LAUNCH rig #2 was operated stably. With respect to the apparent lack of data between 15th and 16th of January 2021, this was the result of a software issue and does not affect the mean values reported here, since no data were logged in that period.



Figure 2 – Temperature stability along the absorber during the campaign

During the campaign, the average lean loading was 0.30 and the rich loading 0.45 mol CO₂/mol amine, leading to a cyclic capacity of the solvent of 0.16 mol CO₂/mol amine. The aforementioned stability of operation can be also seen in the loading data presented in Table 5. These values are calculated based on the FTIR measurements as shown in Table 5, since this is the method that was used for the checks and the campaign and it is the only method that was used for the analysis of both lean and rich samples. The FTIR measurement reports the CO₂ amount in mol/L. Therefore, the density of the solution is required for expressing all solvent components in the same units (mass-based). The density was calculated according to the model proposed by Han et al. (Han et al., 2012). Since the analysis takes place at ambient conditions, a temperature of 25°C was assumed. The model was also validated against density measurements in the LAUNCH rig#2. Density is measured and recorded continuously in the rich line after the absorber sump and the lean line before the solvent introduction to the absorber. A relative deviation of maximum 1.2% and average relative deviation of 0.7% was found between the estimated values and the online measurements of the rig, which is considered acceptable.

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Data	Uouro	Lean	Rich	Cyclic capacity
Dale	Hours	mol CO ₂ /mol MEA	mol CO ₂ /mol MEA	mol CO ₂ /mol MEA
14-1-2021	96	0.27	0.44	0.17
19-1-2021	216	0.30	0.45	0.15
22-1-2021	288	0.30	0.44	0.14
26-1-2021	384	0.29	0.45	0.16
29-1-2021	456	0.29	0.45	0.16
1-2-2021	528	0.32	0.46	0.14

Table 5 – Lean and rich loadings measured by FTIR-Delft.

The CO_2 capture rate in the campaign was ~87%. The capture rate can be calculated either using the measurements in the gas side or the measurements in the liquid side. For the calculation of the capture rate in the gas side, the inlet CO_2 flow as set in a mass flow controller and the average value of the flow of CO_2 in the outlet of the condenser after the stripper were used, leading to a capture rate of 87%. There is no flowmeter in the outlet of the absorber therefore the capture rate cannot be calculated from the absorption part. For the calculation of the capture rate in the liquid side, the inlet CO_2 flow as set in a mass flow controller, the average values of lean and rich loadings as well as the lean solvent flow entering the absorber were used, leading to a capture rate of 88%. It can be seen that the capture rates calculated in two different ways are in excellent agreement.

As far as the emissions are concerned, they are measured and registered with the aid of the FTIR connected on the top of the absorber. MEA emissions varied from 100 to 250 mg/Nm³ (105 mg/Nm³ in average) and ammonia emissions were measured in the range of 4 to 14 mg/Nm³ (5 mg/Nm³ in average). Although 105 mg/Nm³ of MEA emissions are considered high, the reader should keep in mind that this is the result of the lack of water washes in the LAUNCH rig #2. The water wash is a proven emission mitigation technology and integral part of a capture plant. This is the reason why the LAUNCH rig#2 was upgraded after the campaign with two water washes, one after the absorber and one after the desorber.

Moreover, an important parameter affecting the degradation profile of a solvent is the residence time, specifically in the absorber sump (more relevant for oxidative degradation), in the reboiler and the hot rich line (more relevant to thermal degradation). By knowing the volume of the different parts of the rig, the level of the liquid in the sumps and the liquid flowrate, the residence time can be calculated. It was found that the mean residence time in the absorber sump was 3.5 min, the residence time of the solvent in the reboiler was 30.9 min and the residence time in the hot line connecting the cross heat exchanger with the stripper was 1.3 min.

3.2.2 Analytical measurements

An overview of the measured data, as they were reported after the laboratory analysis, is given in Appendix C. Since more than one analysis methods were used for the measurement of MEA, water and CO₂, the method used and corresponding result is also included.

The data processing has been conducted as follows: For the components that have been measured with more than one technique, the measurements were compared, and one method analysis was chosen based on agreement with the other methods and uncertainties reported in the measurement. These components are MEA, water and CO₂ and the selected analysis methods are IC for MEA, KF for water and FTIR for CO₂. Their concentrations together with the concentration of the rest of the components shown in Table 3 were collected and were converted to be expressed in mass-basis (i.e. mg/kg) using the model from by Han et al. (Han et al., 2012), as explained earlier. Due to the uncertainties in the measurement, the sum of the components was not 10⁶ mg/kg. Therefore, the values were normalized. The data were then expressed in CO₂-free basis.

It was found that looking at the data as obtained from the analytical laboratories, or even at CO₂-free basis, can lead to erroneous conclusions regarding the degree of degradation. This is the result of the water

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concentration changes in the system, since during normal operations of the rig, the operator often needs to pump water in the system, thus diluting the solvent. For this reason, the following values depicted in the graphs are both expressed in CO_2 -free basis and they are corrected for the water content, assuming the amount of water in the start of the campaign remained constant, i.e. 65 wt%. Here, it is worth mentioning that the lean loadings calculated based on the content of MEA by IC, of water by KF and of CO_2 by FTIR are very similar as the ones in Table 5, where all three components were analysed with FTIR. The average value is 0.31 compared to 0.30 mol CO_2 /mol MEA, whose difference is considered negligible.

Figure 3 shows the MEA concentration along the campaign. It can be seen that it remains relatively stable and no clear trend of decreasing amine, that could indicate degradation, is demonstrated.



Figure 3 – MEA concentration, expressed in CO₂-free basis and water-corrected.

The composition of the treated flue gas on the outlet of the absorber is followed using an FTIR. Among the components followed, ammonia is also included since it is a main degradation component. Figure 4 shows the ammonia concentration in the gas coming out of the absorber outlet during the duration of the campaign. It is seen that the concentration is highest in the start of the campaign, stabilizes around 13 mg/kg after 3 days of operation, then drops again until day 15 when it increases and stabilizes again around 8 mg/kg.



Figure 4 – Ammonia concentration in treated flue gas stream in the absorber outlet

From the components that were analysed, acetic acid, HEEDA as well as lead were not detected over the lower limit of quantification of the analysis method. Figure 5 shows that the formation of formic and oxalic acid

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increases along the campaign, with formic acid detected at higher concentration than oxalic acid. As far as the organic compounds measured by LC-MS are concerned (Figure 6), it is seen that their concentration remains relatively constant along the campaign except for HEPO and MEA-Urea. Specifically, HEPO concentration increases sharply and reaches 10759 mg/kg on the last day of the campaign, MEA-Urea concentration reaches 2946 mg/kg and HEGly concentration reaches 1307 mg/kg. Dividing them into concentration areas; HEI and HEF are found between 500-1000 mg/kg, HEHEAA & HEA between 100-500 mg/kg and BHEOX, HEIA, OZD and Nitroso-HEGly lower than 100 mg/kg. Interestingly, both HEI and HEF concentrations start to decrease after day 12. Nitroso-HeGly is, as expected, low and close to the detection limits.



Figure 5 – Formic and oxalic acid, expressed in CO₂-free basis and water-corrected. Acetic acid was not detected (detection limit <10 mg/kg).



Figure 6 – Organic compounds which are representative for MEA degradation, expressed in CO₂-free basis and water-corrected. HEEDA not detected over the lower limit of quantification (LLQ < 1 mg/kg).

The samples were analysed for metal content to be used as a corrosion indicator. The analysis results are presented in Figure 7. What stands out from this graph, is the high starting concentration of iron of >5 mg/kg. This is believed to be either due to the storage material of MEA solution, or due to the fact that the solvent was loaded with CO_2 and let circulating for a few days before the official commencement of the campaign, or

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both. Generally, literature related to solvent management agrees that the metals' content in the solvent is a key determinant aspect of the degradation rate (Chi & Rochelle, 2002; Léonard et al., 2014). Therefore, it is recommended to keep the metals' level below a certain threshold (Anne Kolstad Morken et al., 2019). However, other investigations found no correlation between metal concentration and solvent degradation in long-term tests with 30% MEA and CESAR1 (Moser et al., 2020, 2021), the latter within LAUNCH WP5.



Figure 7 – Metals' concentration in CO₂-free basis and water-corrected.

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4 Degradation campaigns in TERC

4.1 Description of TERC

The pilot scale CO₂ capture plant at TERC is capable of capturing 1tpd CO₂ based on 200 Nm³/h gas flow having 15% CO₂ 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 Nm³/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: CO₂, N₂ and Air, each of 6-300Nm³/h flow rage and a trace gas (NO₂, SO₂) 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 8. Equipment specifications are given in Table 6. 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 including gas humidification control in the DCC. However, during these tests the FG/DCC was bypassed.



Figure 8: TERC CO₂ 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.

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The PHW has a bypass to control the flow rate through the reboiler or to bypass it. A pneumatically driven 3way 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.

The CO_2 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_2 is exhausted to atmosphere after analysis.

A blower is used to drive the gas through the plant. For this test campaign, air with CO_2 injection, rather than real flue gas, was used, to give enhanced O_2 levels. The tests were performed under general gas turbine conditions so the CO_2 concentration in the absorber entry gas was kept close to 5%.

CO₂ 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.

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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 6; Absorber and stripper specifications

Table 7 Process and analytical measurements

Analysis	Detail
Main Process parameters	 Gas inlet flow, temperature and pressure Interstage gas temperatures and pressures Absorber 1 &2 and desorbed temperature profiles and pressure drops Desorber pressure (reflux condenser) and CO2 product flow Liquid flows, temperatures pressures and densities Reboiler hot water flow; inlet, outlet, core temperature; supply pressure
Gas analysis	 Multipoint sampling and analysis by Gasmet FTIR: 1. Absorber 1 column inlet, 2. Absorber 2 column inlet, 3. Water wash column inlet, 4. Water wash outlet; 5. Desorber outlet after reflux condenser
Liquid titrations	 Mettler Toledo auto titrator 1. Fast loop sampling from Abs 1 (Rich), Abs 2 (Semi-rich) and Desorber (Lean) 2. MEA solvent concentration 3. CO2 concentration and loading
Dissolved oxygen analysis	Jumo online oxygen analysis 1. Lean (desorber outlet) 2. Semi-rich (absorber 2 outlet) 3. Rich (absorber 1 outlet)
Iron analysis	Analysis on Lean (desorber outlet)



Process and analytical measurements are described in Table 7. 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 Gasmet 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, Mettler Toledo auto-titrator shown in Figure 9 is used. The apparatus was acquired to be run over the weekends remotely and keep weekend samples for analysis. However, the apparatus did not work as intended and some of the samples over the weekend could not be collected. 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 peristatic pumps are used to acquire samples when needed. The auto-titrator performs titrations on the three samples for solvent concentration and CO2 loading analysis. Offline 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 10.



Figure 9: Mettler Toledo auto-titrator



Figure 10: Fe measurement apparatus

4.2 Approach



For the test campaign the plant was operated for 500 hours with air and CO_2 mixture. The concentration of CO_2 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 CO₂ loadings and some of the samples (six samples including zero sample) 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 CO₂ 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 is given in Table 8.

Component	CAS	Name	Formula	Analytical method
MEA	141-43-5	Monoethanolamine	C2H7NO	Titration, LC-MS
CO2	124-38-9	Carbon dioxide	CO2	Titration
HEEDA/AEEA	111-41-1	2-[(2-aminoethyl) amino]-ethanol	C4H12N2O	LC-MS
HEHEAA	144236-39- 5	N-(2-hydroxyethyl) -2-[(2- hydroxyethyl) amino]-acetamide	C6H14N2O3	LC-MS
MEA urea	15438-70-7	N,N'-bis(2- hydroxyethyl)-urea	C5H12N2O3	LC-MS
HEI	1615-14-1	1H-imidazole-1- ethanol	C5H8N2O	LC-MS
HEF	693-06-1	N-(2-hydroxyethyl) -formamide	C3H7NO2	LC-MS
OZD	497-25-6	2-oxazolidinone	C3H5NO2	LC-MS
HEPO	23936-04-1	4-(2-hydroxyethyl)- 2-piperazinone	C6H12N2O2	LC-MS
HeGly	5835-28-9	N-(2-hydroxyethyl) -glycine	C4H9NO3	LC-MS
BHEOX	1871-89-2	N1,N2-bis(2- hydroxyethyl)- ethanediamide	C6H12N2O4	LC-MS
HEA	142-26-7	N-(2-hydroxyethyl) -acetamide	C4H9NO2	LC-MS
HEIA	3699-54-5	1-(2-hydroxyethyl)- 2-imidazolidinone	C5H10N2O2	LC-MS
Fe	7439-89-6	Iron	Fe	Colorimetry

Table 8: Details of solvent analysis

4.3 Results

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This section covers the results obtained during the test campaign and the post processing of the collected samples. The section is divided into two sub-sections; (1) operational data (2) degradation analysis

4.3.1. Operational data:

The plant was operated for 500 hours with 35 wt% MEA. Operational parameters of the plant are summarized in Table 9 below.

	mai parameter	3
Parameter	Unit	Mean value
Temperature Gas inlet	С°	27.9
Pressure Gas inlet	mbar	30
Air inlet flowrate	m³/h	189
Gas inlet composition, CO2	vol(%) wet	5.1
Gas inlet composition, H2O	vol(%)	0.8
Lean solvent inlet temperature	°C	40
Lean solvent inlet flowrate	kg/h	300
Lean solvent density	kg/m ³	1029
Lean solvent outlet flowrate	kg/h	300
Gas outlet temperature	°C	35.5
Pressure outlet	mbar	26
Rich solvent outlet temperature	°C	31
Rich solvent outlet flowrate	kg/h	300
Rich solvent density	kg/m ³	1045
Absorber 2 (Top)	°C	39.7
Absorber 2	°C	39.4
Absorber 2	°C	38.6
Absorber 2	°C	37.6
Absorber 2	°C	42.2
Absorber 2	°C	35.2
Absorber 2	O°	34.5
Absorber 2	°C	34.9
Absorber 2	°C	34.2
Absorber 2	°C	33
Absorber 2 (bottom)	С°	30.7
Absorber 1 (Top)	С°	30
Absorber 1	С°	32.3
Absorber 1	С°	44.8
Absorber 1	С°	54.1
Absorber 1	С°	59.6
Absorber 1	С°	61.4
Absorber 1	С°	60.4
Absorber 1	С°	57.9
Absorber 1 (bottom)	C°	53.3
Liquid volume in absorber sump	litres	70
Rich solvent inlet temperature	С°	90
Pressure outlet to condenser	barg	0.5
Lean solvent outlet temperature	С°	112
stripper T profile (top)	°C	107.4
stripper T profile	O°	104
stripper T profile	O°	111
stripper T profile	°C	112.2
stripper T profile (bottom)	°C	112.9

Table 9: Plant operational parameters

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450
118
0.5
15
31
47
67
99

As a result of flooding at the PACT site in Nov 2019, most of the ground floor instrumentation including pumps and level transmitters were damaged. The control system of the plant was also flooded. The plant was moved from PACT site to a newly build TERC site in late 2020/early 2021. The control system was severely damaged so has to be rebuilt. Two different operational systems at the PACT site (National instruments and Allan Bradley) were merged into a single control system. The whole plant has a new wiring and distributed control architecture. Due to the whole new system there were significant complications in commissioning the control system which took a lot of time and resources. One of the main reasons for taking long time was that no single company took the hardware and software contracts. As a result of dependency on different contractors the commissioning issues took longer than normal to resolve.

After commissioning the control system, the plant was prepared for LAUNCH test campaign. As the plant was moved after flooding of the old site and most of the instrumentation and some pipe work was replaced, the plant was cleaned through a comprehensive cleaning process using sodium carbonate followed by passivation as described in Appendix E.





The first test campaign of LAUNCH (Work package 4.1.1) was started with 35% MEA solution. However, due to a bug in the control system the plant started tripping. It took some time to find and fix the bug. During this period after the plant tripping, fully loaded solvent stayed in the plant, sometimes, for few days resulting in potential degradation overtime without accumulating operational hours. Plant operational time line to indicate plant trips and restarts is shown in Figure 11. Note that the x-axis in the plot shown in Figure 10 does not correspond to operational hours as it also includes non-operational hours; however, it does not include all of the non-operational hrs.

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4.3.2 Analytical measurements

The iron (Fe) content of the solvent was measured on most days throughout the test campaign, excluding weekends. The plot of Fe content vs. operational hours is shown in Figure 12.

The plot indicates a steady build-up of Fe in the solvent over the test period, as might be expected.



Figure 12: Iron measurements over the test period

A total of 37 samples were collected over the period of 500 hrs. However, due to the fact that degradation is a relatively slow process at the start and to reserve the resources, it was decided to analyse the samples for degradation every 100 hrs. Table 10 shows the operational hours for the samples sent to SINTEF for analysis. Sample No. 1 is zero sample, collected after circulating the solvent in the plant for couple of hours without heating up and without any gas feed. As the test campaign is aimed at comparison of TERC and TNO rigs, SINTEF was requested to perform the same analysis using the same analytical techniques for TERC samples as was used for TNO samples. The results of LC-MS analysis on the samples as a function of operational hours are presented on CO₂ free basis in Figure 13. The same data is presented as a function of cumulative CO₂ capture in Figure 14. The results are plotted on CO₂ free basis for easy comparison with TNO data presented in Figure 6. Figure 13 indicates that HEPO concentration is the highest reaching around 6700 mg/kg after 500 hrs test. There is some discrepancy in the HEPO results at 300 and 400 hrs samples. However, a closer look at the graph highlights that HEPO is not the exception. Most of the degradation components show similar concentration at 300 and 400 hrs (only HEF, HEI and MEA-urea has a small increase here). The MEA concentration also goes down from 300 to 400 hours. The slope of the iron curve also seems to change after 300 hrs. The reason for this trend is not clear as there was no operational changes in the plant. MEA-Urea has the second highest concentration in the degradation products followed by HEI and HEA.

Comparison of the Figures 6 and 13 indicates that the concentration of the degradation products measured at the TERC plant are considerably lower than those measured at the TNO rig. This could be due to a number of factors such as differences in CO_2 capture capacity of the rigs, solvent flow rate, solvent loadings etc. The CO_2 capture capacity of the TNO and TERC rigs is 25 kg/d and 1 ton/d, respectively. Solvent flow rate at the TERC plant was 300 kg/h. The total solvent inventory of the TERC plant is around 550 kg. Therefore, the ratio of total solvent inventory to the solvent flow rate for these tests was around 110 mins.

Table 10: Samples for degradation analysis at SINTEF

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Sample no	Operational hrs
1	0
2	113
3	193
4	299
5	407
6	500









Figure 14: Results of LC-MS analysis vs CO₂ captured

Emissions of ammonia at the exit of water wash as measured by FTIR are plotted in Figure 15. Generally the emissions are below 50 ppm through out the test campaign. However, there are some peaks which are observed during start up of the plant after tripping. The peaks could be due to the loaded solvent being kept in the plant which may have undergone some degradation during the idle period, resulting in peaks in ammonia emissions at the start which after some time go down and become at normal levels. However, the ammonia emissions measured at the TERC plant are considerably higher than those at TNO's LAUNCH rig #2.



Figure 15: Ammonia emissions at water wash exit

 CO_2 loadings data for the samples analysed for degradation products is presented in Figure 16. 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.

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Figure 16: CO₂ loadings data for the samples analysed

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5 Rig Comparison

Table 11 below compares operational parameters for the TNO's LAUNCH rig #2 and the TERC pilot plant.

Parameter	LR2 Unit Mean Parameter		Unit	TERC Mean value	
ABSORBER		value			500 m
Temperature Gas inlet	°C	37,8	Temperature Gas inlet	°C	27.9
Pressure Gas inlet	mbarg	54,20	Pressure Gas inlet	mbarg	30
Air inlet flowrate	nL/h	4224	Air inlet flowrate	nL/h	189000
O2 inlet flowrate	nL/h	887			
CO ₂ inlet flowrate	nL/h	247			
Inlet flowrate dry total	nL/h	4471			
H ₂ O inlet flowrate	g/h	235			
H ₂ O inlet flowrate	nL/h	293			
Inlet flowrate wet total	nL/h	4765			
Gas inlet composition, CO2	vol(%) dry	5,53	Gas inlet composition, CO ₂	vol(%) dry	5.14
Gas inlet composition, CO ₂	vol(%) wet	5,19	Gas inlet composition, CO2	vol(%) wet	5.1
Gas inlet composition, O2	vol(%) dry	19,8	Gas inlet composition, O2	vol(%) dry	
Gas inlet composition, O2	vol(%) wet	18,6			
Gas inlet composition, H2O	vol(%)	6,2	Gas inlet composition, H2O	vol(%)	0.8
Lean solvent inlet temperature	°C	39,7	Lean solvent inlet temperature	°C	40
Lean solvent inlet pressure	mbarg	524,00 0			
Lean solvent inlet flowrate	kg/h	11,5	Lean solvent inlet flowrate	kg/h	300
Lean solvent density	kg/m3	1080	Lean solvent density	kg/m³	1029
Lean solvent outlet flowrate	m3/h	0,011	Lean solvent outlet flowrate	m³/h	300
Gas outlet temperature	°C	43,4	Gas outlet temperature	°C	35.5
L/G (dry gas/lean solvent)	kg/kg	1.94	L/G (dry gas/lean solvent)	kg/kg	1.2
Pressure outlet	mbarg	26,7	Pressure outlet	mbar	26
Rich solvent outlet temperature	°C	40,9	Rich solvent outlet temperature	°C	31
Rich solvent outlet pressure	mbarg	1180			
Rich solvent outlet flowrate	kg/h	12,1	Rich solvent outlet flowrate	kg/h	300
Rich solvent density	kg/m3	1110,0	Rich solvent density	kg/m³	1045
Rich solvent outlet flowrate	m3/h	0,011	Absorber 2 (Top)	°C	39.7
abs T profile (top)	°C	57,20	Absorber 2	°C	39.4
abs T profile	°C	61,50	Absorber 2	°C	38.6

Table 11: Comparison of operational parameters for TNO's LAUNCH rig#2 and TERC pilot plant



abs T profile	°C	59,80	Absorber 2	°C	37.6
abs T profile	°C	56,5	Absorber 2	°C	42.2
abs T profile (bottom)	°C	50,5	Absorber 2	°C	35.2
			Absorber 2	°C	34.5
			Absorber 2	°C	34.9
			Absorber 2	°C	34.2
			Absorber 2	°C	33
			Absorber 2	۰C	20.7
			(bottom)	C	50.7
			Absorber 1 (Top)	°C	30
			Absorber 1	°C	32.3
			Absorber 1	°C	44.8
			Absorber 1	°C	54.1
			Absorber 1	°C	59.6
			Absorber 1	°C	61.4
			Absorber 1	°C	60.4
			Absorber 1	°C	57.9
			Absorber 1	°C	53.35
			Absorber 1	°C	
			(bottom)	Ū.	
Liquid level in the absorber	%	20,68	Liquid level in the		
Liquid volume in absorber		0,0000			
sump	m3	006	absorber sump	m3	0.070
		Not			
I emperature in the	°C	availabl			
		е			
Residence time in absorber	min	3.5	Residence time in	min	14
sump		00.7	absorber sump		
Pressure outlet to vent	mbarg	26,7			
MEA outlet to vent	mg/Nm3	105			
	mg/Nm3	5,31			
STRIPPER			Dish and a stricted		
temperature	°C	93,100	temperature	°C	90
Rich solvent inlet pressure	mbarg	1060			
Rich loading range	mol/mol	0.45	Rich loading range	mol/mol	0.45 – 0.46
Gas outlet to condenser	°C	88,80			
Pressure outlet to condenser	barg	802,00	Pressure outlet to condenser	barg	0.5
Lean solvent outlet temperature	°C	Not availabl e	Lean solvent outlet temperature	°C	112
Lean loading range	mol/mol	0.3	Lean loading range	mol/mol	0.24 – 0.26
Lean solvent outlet pressure	mbarg	666			
stripper T profile (top)	°C	93,9	stripper T profile (top)	°C	107.4
stripper T profile	°C	95,2	stripper T profile	°C	104
stripper T profile	°C	96,2	stripper T profile	°C	111
stripper T profile	°C	101,0	stripper T profile	°C	112.2



stripper T profile (bottom)	°C	114,0	stripper T profile (bottom)	°C	112.9
Reboiler duty	kW	0,88	Reboiler duty	kW	
Liquid level in the reboiler	%	44,83			
Liquid volume in reboiler	m3	0,005	Liquid volume in reboiler	litres	450
Temperature in reboiler liquid	°C	120,09	Temperature in reboiler liquid	°C	118
Residence time in reboiler	min	30.9	Residence time in reboiler	min	90
Pressure in stripper	barg	0,75	Pressure in stripper	barg	0.5
Temp CO ₂ product from condenser	°C	14,76	Temp CO ₂ product from condenser	°C	15
Flow CO ₂ product from condenser	L/h	227,93			
	nL/h	216,24			
XFHE					
Cold rich inlet temperature	°C	39,9	Cold rich inlet temperature		31
Cold lean outlet temperature	°C	42,5	Cold lean outlet temperature	°C	47
Hot rich outlet temperature	°C	99,9	Hot rich outlet temperature	°C	66.8
Hot lean inlet temperature	°C	114,4	Hot lean inlet temperature	°C	98.9
Residence time in hot rich line	min	1.3	Residence time in hot rich line	min	4
OVERALL					
Solvent inventory	kg	12	Solvent inventory	kg	550
Inventory/circulation rate	mins	62	Inventory/circulatio n rate	mins	110
kg solvent/kg CO ₂ captured per day	kg/kg	1.18	kg solvent/kg CO ₂ captured per day	kg/kg	1.38

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The following components were part of the analytical program at SINTEF for these samples: OZD (497256), BHEOX (1871-89-2), HEA (142-26-7), HEGly (5835-28-9), HEPO (23936-04-1), HEF (693-06-1), HEI (1615-14-1), HEHEAA (144236-39-5), HEEDA (111-41-1), HEIA (3699-54-5), MEA urea (15438-70-7) and NHEGly (80556-89-4). Samples from both LAUNCH rig#2 (LR2) and TERC rig were analysed for the same components. Most of these components have also been reported for other cycled set-ups, either smaller degradation rigs (SDR) (Vevelstad et al. 2021) or larger pilots such as TCM (Morken et al. 2017). HEPO and HEGly are among the three components found in the highest amounts in this work as well as in the solvent degradation rig, TCM, and in samples from a MEA campaign at RWE. MEA-urea has also been quantified in samples from RWE, but it seems to be less important than is observed in smaller rigs such as LAUNCH rig#2, TERC and SDR. More pilot data for this component will therefore be required to understand the behaviour of this component. Investigating the sum of nitrogen from the degradation compounds guantified shows that higher degradation is observed in the LAUNCH rig#2 (campaign 1 and 2) followed by TERC which is followed by the SDR rig using 40 wt% MEA, and all of them has higher degradation compared to RWE (only looking at the sum of nitrogen from the quantified degradation compounds in this work, see Figure 7). HEPO is the single component with the largest contribution to the nitrogen balance for the degradation compounds, and how it behaves will therefore influence the data significantly.



Figure 17 Sum of degradation compounds in mmolN/L as a function of time (hrs) up to 700 hrs for the LR2 (Initial MEA: 5.3 molN/L), TERC 1 (Initial MEA: 5.5 molN/L) and the SDR campaign (Initial MEA: 6.7 molN/L).

Generally, both TERC and LAUNCH rig#2 seems to produce larger amounts of degradation components than the SDR rig. Specifically, there are some trends harder to explain. For example, looking at the data from the LAUNCH rig#2 for the amides (HEF, BHEOX, HEA, HEHEAA) and HEI, see Figure 18.

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Figure 18 Concentration (mmolN/L) of HEF, HEI, BHEOX, HEA and HEHEAA as a function of time (hrs).

HEF, HEI, BHEOX and HEHEAA seems to have similar curves and the start concentration exceeds the end concentration. This is not the case for HEA which seems to increase as a function of time, which is the expected behaviour. These components are either amide or imidazole which is dependent on acids/aldehydes and/or ammonia, and for the amides a similar formation or decomposition mechanism is expected. In later LAUNCH rig#2 campaigns, the behaviour of these components is similar to what is observed for HEA in Figure 18, increasing formation with time with low initial concentration.

For the TERC campaign there are some interesting behaviours for some of the degradation compounds at two different times during the operation. Firstly, some degradation compounds have a steep increase toward 200 hours followed by a steep decline (HEF, HEHEAA, OZD and BHEOX, see Figure 19), secondly a similar or small decline in concentration was observed for HEGly, HEPO, BHEOX, HEIA and MEA-urea (Figure 20) between 300-400 hours. Since the behaviour at 200 and between 300 and 400 hours is not the same for all degradation compounds this is hard to explain based on operational considerations. But, it is also hard to explain why some of the components has a steep increase toward 200 hours while build-up of some of the pauses between 300 and 400 hours.



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Figure 19 Concentration (mmolN/L) of HEF & HEHEAA (on primary y-axes), OZD and BHEOX (on secondary y-axes) as a function of time (hrs), a steep increase toward 200 hours followed by a step decline.



Figure 20 Concentration (mmolN/L) of HEGly, HEPO & MEA-urea (on primary y-axes) and BHEOX and HEIA (on secondary y-axes) as function of time (hrs), the build-up stops or slowly decline between 300 and 400 hours before it increases again.

In the last part the corrected data for comparison between the rigs are given for all the degradation compounds analysed using LC-MS, see Figure 21.

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Figure 21 Concentration (mmolN/L) for the LAUNCH rig#2 and the TERC ACP for all degradation compounds analysed using LC-MS.

Table 12 provides quantitative comparison of individual degradation products for the two rigs.

$\mathbf{T}_{\mathbf{A}}$	Table 12 Com	parison between	the LR2 and the	TERC ACP for eac	h specific degradatior	l compound.
---------------------------	--------------	-----------------	-----------------	------------------	------------------------	-------------

Component	Comment	Concentration
OZD	Low concentration in both, the build-up more visible and the slope larger for the LAUNCH rig.	LR2 > TERC
BHEOX	Both shows scattering. LAUNCH curve similar as for HEF & HEI (to some extent also HEHEAA)	
HEA	Significantly higher build-up in TERC compared to LAUNCH.	TERC > LR2
HEGly	Higher initial concentration in LAUNCH, overall, the build-up relatively constant and low. For TERC a larger build-up observed, and concentration higher than for LAUNCH.	TERC > LR2
HEPO	Similar up to 300 hrs, the slope of the LAUNCH curve possible slightly larger than TERC, after 300 hrs the slope seems to be the same as for LAUNCH.	LR2 > TERC
HEF	High initial concentration in LAUNCH, declining over time, relatively scattered (similar curve as for HEI and BHEOX and to some extent HEHEAA). TERC increases over time.	
HEI	High initial concentration in LAUNCH, declining over time, relatively scattered (similar curve as for HEF and BHEOX and to some extent HEHEAA). TERC increases over time.	
HEHEAA	Similar initial and end concentration for the LAUNCH rig. The curve shows similarity to HEF, HEI and BHEOX. For TERC a steeper build-up initially, a turning point with smaller slope than initially.	TERC > LR2
HEEDA	No HEEDA observed for LAUNCH, while HEEDA first was observed after 500 hrs in TERC.	
HEIA	Higher concentration observed in TERC compared to LAUNCH.	TERC > LR2
MEA-urea	Higher concentration and steeper formation in LAUNCH compared to TERC.	LR2 > TERC



6 Conclusions

The most abundant degradation compounds observed in this work were HEPO, HEGIy and MEA-urea, this is comparable to other laboratory rigs and some larger pilots. HEPO dominated and has therefore a large influence on the overall degradation behaviour. More degradation was observed in LAUNCH rig#2 compared to the TERC ACP*, and both showed more degradation than the SDR rig, as well as more than has been observed at the RWE pilot⁺. There are some interesting trends observed for some of the degradation compounds which cannot be explained by water, CO₂ or MEA concentration variation. More work is required to relate these observations to degradation mechanisms.

Footnotes - added May 2023:

*Subsequent investigations suggest that the unexpected presence of zinc (thought to be from corrosion of a heater element) – see Table C4 - may have had a role in catalysing degradation in LAUNCH rig#2.

⁺ A recently published study (referenced below) identified a likely stabilising role for the fly ash present in RWE pilot trials in MEA degradation tests and also noted higher oxidative degradation rates with solutions containing 'metal ions like chromium, magnesium, and zinc, making these suspects of deteriorating solvent stability'.

Buvik, V. *et al* (2023) Degradation behaviour of fresh and pre-used ethanolamine, Carbon Capture Science & Technology, Volume 7. <u>https://doi.org/10.1016/j.ccst.2023.100110</u>



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Appendix

Appendix A. Data collection template

Sensor			Mean	
Tag	Parameter	Unit	value	Comments
		Case definition	n	
	Flue gas source	[-]		
	Start date and time	[-]		Please indicate the date and time of the data point below
	End date and time			
	Time since beginning of campaign	hours		
		Absorber		
	Temperature Gas inlet	°C		
	Pressure Gas inlet	barg		
	Gas inlet flowrate	kg/h		
	Gas inlet composition, CO2	vol(%) dry		
	Gas inlet composition, CO2	vol(%) wet		
	Gas inlet composition, O2	vol(%) dry		
	Gas inlet composition, O2	vol(%) wet		
	Gas inlet composition, H2O	vol(%)		
	Lean solvent inlet temperature	°C		
	Lean solvent inlet pressure	barg		
	Lean solvent inlet flowrate	kg/h		
	Lean solvent inlet flowrate	m3/h		
	Solvent inlet composition, MEA	g/100g		
	Solvent inlet composition, water	g/100g		
	Solvent inlet composition, CO2	g/100g		
	Solvent inlet composition, CO2	mol/mol		
	Gas outlet to water wash	°C		
	Gas flow outlet to water wash	kg/h		
	Pressure outlet to water wash	barg		
	CO2 outlet to water wash	vol(%) dry		
	CO2 outlet to water wash	vol(%) wet		
	H2O outlet to water wash	vol(%)		
	Rich solvent outlet temperature	°C		
	Rich solvent outlet pressure	barg		
	Rich solvent outlet flowrate	kg/h		
	Rich solvent outlet flowrate	m3/h		
	Solvent outlet composition, MEA	g/100g		
	Solvent outlet composition, water	g/100g		
	Solvent outlet composition, CO2	g/100g		
	Solvent outlet composition, CO2	mol/mol		
	abs T profile	°C		
	abs T profile	°C		
	abs T profile	°C		Diagona incerti anno 11
	abs T profile	°C		Please insert more rows if needed.
	abs T profile	°C		each sensor, and if liquid or gas
	abs T profile	°C		temperature is being measured



abs T profile	°C	
abs T profile	°C	
Absorber packing height	m	
 Absorber packing type	[-]	
Height of liquid inlet	m	Please indicate if not at the top of the packing
Height of gas inlet	m	Please indicate if not at the bottom of the packing
Liquid volume in absorber sump	m3	· · · · · · · · · · · · · · · · · · ·
Temperture in the aborber sump	°C	
· · · ·	Water wash	
Water inlet temperature	°C	
Water inlet pressure	barg	
Water inlet flowrate	kg/h	
Water inlet flowrate	m3/h	
Water inlet composition, MEA	g/100g	
Water inlet composition, water	g/100g	
Water inlet composition, CO2	g/100g	
Water inlet composition, CO2	mol/mol	
Gas outlet to vent	°C	
Gas flow outlet to vent	ka/h	
Pressure outlet to vent	barg	
 CO2 outlet to vent	vol(%) drv	
 CO2 outlet to vent	vol(%) wet	
H2O outlet to vent	vol(%)	
MEA outlet to vent	mg/Nm3	
NH3 outlet to vent	mg/Nm3	
Water outlet temperature	°C	
 Water outlet flowrate	ka/h	Please indicate average flowrate of drain to the absorber sump
Water outlet flowrate	m3/h	
Water outlet composition. MEA	a/100a	
Water outlet composition, water	g/100g	
Water outlet composition, CO2	g/100g	
Water outlet composition, CO2	mol/mol	
 Water outlet composition, O2	ma/ka	
Water wash T profile	°C	
 Water wash T profile	°C	Please insert more rows if needed.
 Water wash T profile	°C	Please indicate packing height of
 Water wash T profile	°C	temperature is being measured
Water wash packing height	m	
 Water wash packing type	[-]	
	Stripper	
Rich solvent inlet temperature	°C	
Rich solvent inlet pressure	barg	
Gas outlet to condenser	°C	
Gas flow outlet to condenser	ka/h	
Pressure outlet to condenser	barg	
CO2 outlet to codenser	vol(%) dry	
CO2 outlet to condenser	vol(%) wet	



H2O outlet to condenser	vol(%)		
Lean solvent outlet temperature	°C		
Lean solvent outlet pressure	barg		
stripper T profile	°C		
stripper T profile	°C		
stripper T profile	°C		
 stripper. T profile	℃ ℃		
stripper. T profile	<u>.</u> 		
stripper. T profile	<u></u> 2°		Please insert more rows if needed.
stripper. T profile	0°C		Please indicate packing height of
stripper T profile	<u>ິ</u>		each sensor, and if liquid or gas
Reboiler duty			temperature is being measured
Stripper packing beight	 		
Stripper packing type	[-]		Please indicate if not at the top of
Height of rich solvent inlet	m		the packing
			Please indicate if it is returned back
Height of condensate inlet	m		to another point in the plant
Height of gas inlot	m		Please indicate if not at the bottom
	m3		
Tomporature in reboiler liquid	•C		
	C		
Brossure in rebeiler	borg		
	barg		
Flaw CO2 product from condenser			
Flow CO2 product from condenser	kg/n		
 Pressure CO2 product from condenser	barg		
 CO2 product from condenser	Vol(%) dry		
CO2 product from condenser	vol(%) wet		
H2O in CO2 product	vol(%)		
F	Reboiler (steam	ר?)	
hot amine	kg/h		
cold amine	°C		
 Steam	kg/h		
Steam	bar g		
condensate	kg/h		
condensate	°C		
Crc	ss Heat Excha	anger	
Cold rich inlet temperature	°C		
Cold lean outlet temperature	°C		
Hot rich outlet temperature	°C		
Hot lean inlet temperature	°C		
	Other		
Addition of process water	kg/h		Please indicate where
			Please include any other relevant parameter

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Appendix B. Sample overview and selection

Daily lean and rich samples were taken and several of them were analysed in intervals of 2-3 days by FT-IR in TNO for amine, water and carbon dioxide content. A selection of samples were also analysed using other analysis methods and for other components. Table B1 shows the overview of time series and available samples.

Date	Hours	FT-II (MEA,	R (TNO) H ₂ O, CO ₂)	IC, LC-N (accordin	IS, TIC-TOC
		Analysed	Sample type	Analysed	Sample type
11-1-2021	24	Yes	lean, rich	Yes	lean
12-1-2021	48	Yes	lean, rich	No	lean
13-1-2021	72	No	lean, rich	No	lean
14-1-2021	96	Yes	lean, rich	Yes	lean
15-1-2021	120	No	lean, rich	No	lean
16-1-2021	144	No	lean, rich	No	lean
17-1-2021	168	No	lean, rich	No	lean
18-1-2021	192	No	lean, rich	No	lean
19-1-2021	216	Yes	lean, rich	Yes	lean
20-1-2021	240	No	lean, rich	No	lean
21-1-2021	264	No	lean, rich	No	lean
22-1-2021	288	Yes	lean, rich	Yes	lean
23-1-2021	312	No	lean, rich	No	lean
24-1-2021	336	No	lean, rich	No	lean
25-1-2021	360	No	lean, rich	No	lean
26-1-2021	384	Yes	lean, rich	Yes	lean
27-1-2021	408	No	lean, rich	No	lean
28-1-2021	432	No	lean, rich	No	lean
29-1-2021	456	Yes	lean, rich	Yes	lean
30-1-2021	480	No	lean, rich	No	lean
31-1-2021	504	No	lean, rich	No	lean
1-2-2021	528	Yes	lean, rich	Yes	lean

 Table B1 – Time series and samples overview

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Appendix C. Analytical measurements for TNO LR #2

The data obtained for MEA, water and CO₂ that were measured with FTIR.

		Le	ean		Rich					
Date	MEA	H₂O	CO2	CO ₂	MEA	H₂O	CO2	CO ₂		
	wt%	wt%	wt%	mol/L	wt%	wt%	wt%	mol/L		
14-1-2021	33.26	60.44	6.18	1.58	31.94	57.88	10.12	2.57		
19-1-2021	34.40	58.26	7.27	1.85	33.48	55.45	10.96	2.78		
22-1-2021	34.1	58.71	7.1	1.81	33.66	55.46	10.85	2.75		
26-1-2021	33.63	59.55	6.76	1.73	32.15	57.2	10.57	2.68		
29-1-2021	33.38	59.74	6.81	1.74	32.37	56.95	10.57	2.68		
1-2-2021	32.67	60.02	7.23	1.84	31.72	57.56	10.64	2.70		

Table C1 – Lean and rich sample analysis for MEA, water and CO₂ by FTIR.

The measured data for MEA, water and CO_2 that were measured with two different methods are provided in Table C2C2.

Table C2 – Lean sample ana	lysis for MEA,	water and CO ₂
----------------------------	----------------	---------------------------

	MEA			Water		CO ₂		
Date			LC-MS	KF				
		FTIR	4	titration	FTIR	PA titration	FTIR-Petten	
	mg/kg	wt%	mg/kg	wt%	wt%	mol/L	mol/L	
14-1-2021	318780.0	33.26		65	60.44	1.47	1.50	
19-1-2021	324049.6	34.40		67	58.26	1.60	1.85	
22-1-2021	329054.8	34.10		64	58.71	1.58	1.67	
26-1-2021	337245.4	33.63		65	59.55	1.58	1.67	
29-1-2021	311990.0	33.38		65	59.74	1.58	1.71	
1-2-2021	310483.4	32.67		65	60.02	1.65	1.68	

Table C3 – Lean sample analysis for acids and inorganic compounds using IC.

Data	Acetic acid	Formic acid	Oxalic acid	Nitrate	Nitrite
Dale	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
14-1-2021	-	78.5	8.3	30	21
19-1-2021	-	97.1	15.2	48	19
22-1-2021	-	115.6	18.6	56	23
26-1-2021	-	119.3	22.7	72	15
29-1-2021	-	131.4	25.6	76	10
1-2-2021	-	151.2	28.2	85	14

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Table C4 – Lean sample analysis for metals using ICP-MS.

Data	Cr	Fe	Ni	Mn	Cu	Zn	Мо	Ba	Pb
Date	mg/kg								
14-1-2021	0.19	6.54	0.32	0.09	0.1	2.82	0.07	0.25	<0.01
19-1-2021	0.23	7.01	0.35	0.09	<0.1	3.11	0.08	0.22	<0.01
22-1-2021	0.29	6.54	0.50	0.09	<0.1	3.56	0.11	0.29	<0.01
26-1-2021	0.36	6.61	0.67	0.09	<0.1	3.86	0.14	0.29	<0.01
29-1-2021	0.41	6.96	0.81	0.10	<0.1	4.54	0.16	0.27	<0.01
1-2-2021	0.19	6.54	0.32	0.09	<0.1	2.82	0.07	0.25	<0.01

Table C5 – Lean sample analysis by LC-MS

	MEA-Urea	ΗΕΗΕΔΔ	HEEDA	HEEDA 2-oxazoline		HEE	HEPO
Date	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
14-1-2021	703	281	< 1		1196	1217	723
19-1-2021	1955	318	< 1		1250	1066	3007
22-1-2021	2344	317	< 1		1615	1329	4268
26-1-2021	2912	284	< 1		1056	799	6853
29-1-2021	3068	227	< 1		683	503	9549
1-2-2021	3171	241	< 1		793	605	11579
14-1-2021							
					Nitroso-		
HeGly	HEA	BHEOX	HEIA	OZD	mg/kg		
mg/kg	mg/kg	mg/kg	mg/kg	mg/kg			
993	83.4	35.9	10.8	19.1	< 0.50		
1250	156	38.4	25.3	43.6	< 0.50		
1305	207	57.4	34.5	49.0	< 0.50		
1373	219	27.8	54.8	55.0	0.5		
1413	217	10.2	79.9	56.8	0.9		
1407	259	14.4	101	67.1	1.0		
993	83.4	35.9	10.8	19.1	< 0.50		



LAUNCH D4.1.1 Comparison TERC and LR#2 20/05/2023 Public 41/41

Appendix D. Analytical measurements for TERC pilot plant

Oper hrs	CO ₂	CO2	MEA	HEI	HEF	HEPO	HeGl y	HEA	BHEO X	HEIA	OZD	MEA- Urea	HEHEAA	HEEDA	Nitroso -HeGly
	mol/kg	•		•			•		mg/kg			•			
0	0.000	0.000	329	2.84	11.5	0.88	47.0	2.25	1	0.1	0.20	0.294	14.1	1	1
113	0.702	30890.513	273	179	205	1290	1650	262	5.43	55.4	3.24	722	178	1	1
193	0.702	30890.513	301	529	513	2540	2320	503	26.9	108	12.0	1090	357	1	1
299	0.599	26370.380	302	602	345	4370	3230	636	16.0	139	2.01	1410	301	1	1.57
407	0.599	26370.380	295	890	446	4430	3190	681	16.1	141	2.50	1340	327	1	1.56
500	0.966	42514.299	298	979	469	6480	3510	796	25.6	153	5.85	1600	377	1.04	2.13

Appendix E. Procedure for cleaning and passivation:

- 1. Circulated water (90 °C) for few hours and drained
- 2. Cleaned the strainers
- 3. Repeated water washing for few times
- 4. Circulated Sodium carbonate (3%) solution at 90 °C for one day and pumped out to waste IBC
- 5. Cleaned the strainers
- 6. Circulated water (90 °C) for few hours and pump out to waste IBC
- 7. Cleaned the strainers
- 8. Circulated Monoethanolamine (3%) solution at 90 °C for 2 days and pump out to waste IBC
- 9. Cleaned the strainers
- 10. Circulated clean water for couple of hours and pumped out to waste IBC
- 11. Cleaned the strainers
- 12. Circulated Monoethanolamine (14%) solution at ~20 °C for ~ one day and at 90 °C for ~ one day and pumped out to waste IBC
- 13. Cleaned the strainers
- 14. Filled the plant with 35% MEA, ready for the test campaign

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