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Pilot Plant Test Report - Extended Phase

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Revision History

Reason for Issue	Rev.	Date	Change
Issued for Information	01	11.11.2020	Updated NC03-KEA-P-RA-0017 rev. 2: Particle settlement analysis included (section 4.7.3)
Issued for Information	02	08.12.2020	Removed "Confidential" from front page, Deleted Ch 6 and other confidential information.

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1 SUMMARY

1.1 General achievements

The extended pilot plant test campaign started June 3rd and ended December 1st 2019 and the total number of successful pilot plant operational hours at Klemetsrud reached about 5100. The purpose of running the pilot plant beyond the original 2000 hours was to obtain additional knowledge of operating the plant at higher levels of solvent degradation and various upset conditions (as described in the test plans.

Between June and December, several specific tests were performed which can be summarised as follows:

- Aerosol Mitigating Device (AMD) tested for 3.5 weeks without any increase in pressure drop across the unit,
- Different flue gas (FG) temperatures into and out of the absorber to simulate seasonality,
- Different FG temperatures across the water wash,
- Different CO_2 removal efficiencies,
- Varying conditions to optimise steam consumption, and
- Varying conditions to test the effect on emissions with and without AMD.

The amine emissions remained well below the emission target level (0.4 ppmv) for most of the test period despite the varied test conditions. However, it should be noted that the tests between June and August were performed at slightly lower (than design) flue gas flow rates (the maximum achievable flue gas flow was reduced around July/August due to K3 maintenance break). Thus, the results indicate that the system is robust with regards to amine emissions when flue gas flow and CO_2 loading in the system is somewhat reduced compared to design capacity.

In September, the flow was kept at or above design conditions, and steam consumption optimisation tests were performed, followed by a prolonged period of AMD vs no AMD testing. A close to 10% reduction in steam consumption compared to the previously (first 2 000 hours) reported results was possible by adjusting operational parameters.

It was decided to extend the AMD vs. no AMD testing (at the expense of other tests) due to both the frequent variations in flue gas composition during the selected testing period and the importance of the results. From the results it can be seen that the AMD helps to reduce amine emissions to air during upset conditions (experienced between weeks 42 and 48), but that it's effect during normal operation appears less pronounced.

Amine degradation was continuously monitored and reached almost 6 wt% (wet basis, accuracy +/- 10%) by the end of the extended test campaign. The degradation rate remained fairly constant during the first 4000 hours. However, after reaching a degradation product concentration of around 3 wt%, the results indicate a shift/acceleration in the degradation rate (coinciding with both pilot and WtE plant upset conditions).

1.2 Lessons learned

The most significant lessons learned from the extended test campaign are as follows:

- Amine emissions to air can be controlled / maintained low by adjusting operational parameters (delta T over the water wash section being most critical).
    - The only exception found to the above is related to unusually high dust levels measured on K3 (due to upset conditions), for details see Chapter 4.2.1.
    - Acetaldehyde (as a known degradation product) levels in the treated flue gas did not increase with increasing degradation levels
    - The level of other contaminants (incl. NH_3) in the flue gas are effectively reduced (both by the pre-scrubber and the absorber) based on PTR-TOF measurements and also FTIR measurements during the first 2000 hours.
- Amine emissions to air have not been significantly[1] affected by the increased degradation level of the solvent and enables re-evaluation of the design/steady-

[1] Quantifying this is difficult due to the varied test conditions taking place, particularly during the last weeks of the extended test campaign. Reference is made to Chapter 0.

state degradation level for the full-scale plant, which in turn impacts TRU design (i.e. could be smaller)

- Solvent degradation rate remains constant up to at least 3 wt% degradation concentrations (despite varying test conditions). Above 3 wt%, data indicate a possible acceleration of degradation. However, this apparent acceleration coincides with upset conditions at the pilot plant as well as the WtE plant.
- The limited impact of high degradation products concentration on both emissions and amine degradation rate could allow to review and revise design degradation products concentration and TRU sizing - taking also into account actual measured NO_2 concentrations.
- The effect of operating with and without an AMD has been demonstrated.
    - o Unless the experienced high dust levels from K3 can be avoided by certain (not yet specified) methods, the AMD is necessary. Alternatively, modifications to the WtE plant (dust filter vs. ESP) are needed.
- Experience has been gained to optimize the steam consumption using specific methods (L/G vs stripping factor) during the test campaign to allow for later easier implementation for the full-scale plant.
- The importance of ensuring mass-balance closure by having only a few reliable instruments and flow meters in place at critical locations has been understood.
    - o For the pilot, focus has been on two meters experiencing fairly constant conditions; CO_2 product flow meter and the flow meter after the pre-scrubber
- Focus on the selection of an appropriate flue gas blower o Temporary fixes (such as the implemented blower safe mode setup) do not work in the long run. It is acknowledged that side channel blower type of design will not be used for the full-scale plant
- No unexpected/significant impurities in the CO_2 product have been found.
- No foaming issues in pilot plant operation, despite all foaming tests indicating high foaming tendency

1.3 Testing hours overview

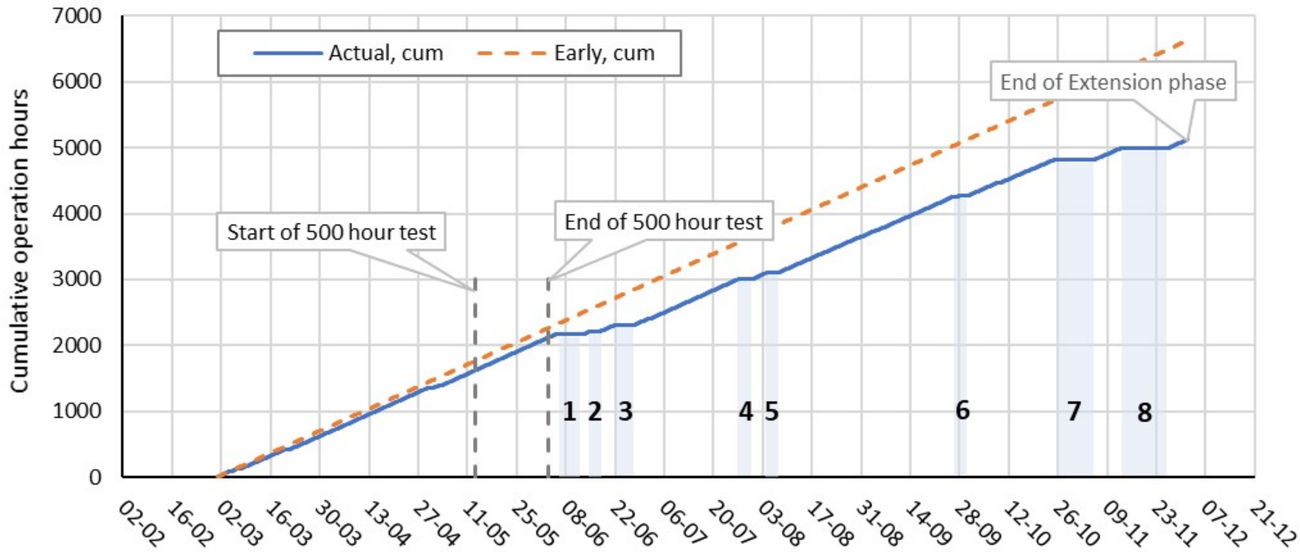


Figure 1. Cumulative operational hours for the pilot plant (including 24-hour test run).

From the above Figure it can be seen that the pilot plant has had some significant periods of downtime during the extension campaign. The significant downtime periods have been numbered and below is a brief explanation of the reason why:

1. Trip due to high-high level in stripper followed by blower fan being stuck and requiring maintenance.
2. Unstable incineration plant (resulting in e.g. loss of "permissive to start" signal)
3. Water wash pump failed and was replaced.
4. Plant tripped during the weekend (due to flashing and low level in the stripper) followed by blower fan being stuck and requiring maintenance
5. Plant tripped (possibly due to lightning strike) followed by blower fan being stuck and requiring maintenance
6. Planned shut down due to service at WtE plant drainage system.
7. Trip due to high level in reflux drum followed by blower fan being stuck and requiring maintenance.
8. Fan motor short circuited. Further inspection revealed water in the connector lid to the motor, extensive service required. (Plant tripped December 1st followed by same fan motor problem as before. Extensive service required and therefore no time for further pilot plant operation.)

Due to the multiple blower overhauls, it was decided to modify the control system to keep the blower fan running when the system trips (to avoid excessive downtime), in so called safe mode operation. This work was successfully completed (in September) and prevented the blower from being stuck during minor trips (not visible in the above Figure).

2 PLANT OPERATION

2.1 KEA flue gas supply

<u>Lines in operation</u>

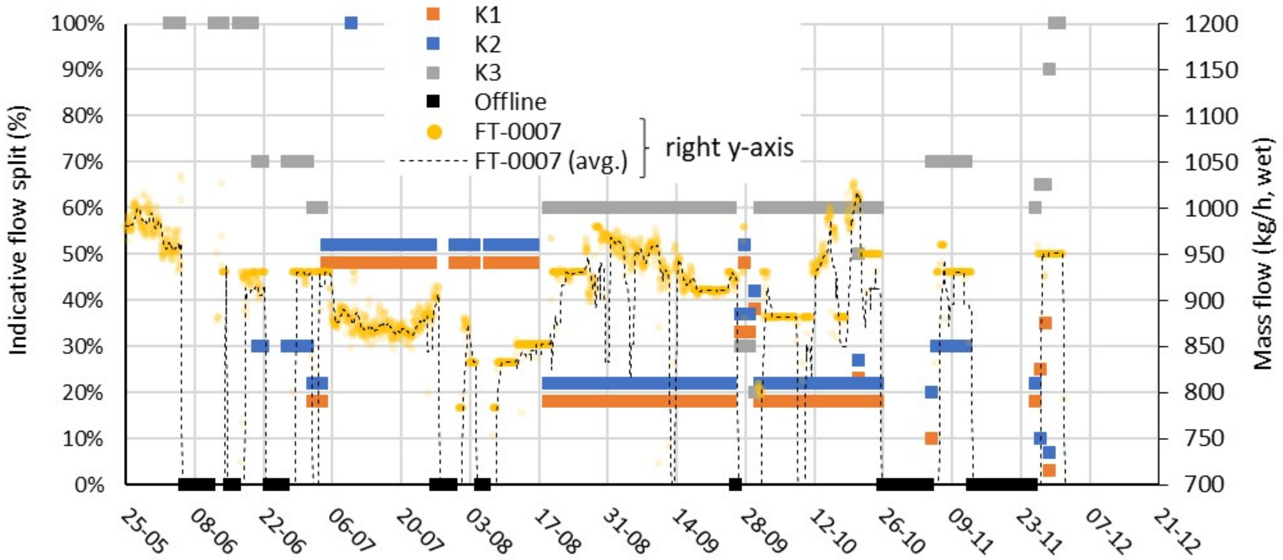


Figure 2. Indicative fraction (mass basis) of flue gas from each WtE plant line into the pilot plant during June to December together with mass flow of FG into the absorber (FT-0007 - actual)[2].

It should be noted that Figure 2 only represents indicative mass fractions of flue gas from the different WtE incineration lines. This is due to the fact that there have been problems with the

[2] FT-0007 actual is the modified instrument reading based on a mass balance around the pilot plant

flow transmitters located on the interconnection lines between the pilot and WtE plant. The pilot plant flow transmitters, however, have functioned normally.

Representative flue gas

In general, no significant differences in flue gas composition have been detected compared to BoD over the course of June to December (except for slightly lower average CO levels from K1 and K2 after the yearly service breaks). However, exceptionally high dust levels (hourly averages up to 10 mg/Nm^3, compared to typically less than 1 mg/Nm^3) were noted for K3 between mid-October and end of November (weeks 42 and 48), impacting the emission performance of the pilot plant (see Chapter 4.2.1).

2.2 Pilot plant key parameters

Table 1 represents the key parameters needing to be controlled for successful pilot plant operation and they have continued to be targeted during the extended test campaign. However, excursions (outside the typical range) in the name of testing the pilot plants performance have been made. Therefore, providing an overall average for the different parameters for the entire test campaign is not meaningful.

Table 1. Key process parameters for the pilot plant.

Variable	Unit	Target		Typical range		
Inlet absorber temperature TT-0005	°C	40		30	-	50
CO_2 removal efficiency Online calculation	%wt	90+		80	-	100
Lean loading (50%wt amine) Offline lab result	%wt	Note 1		Note 1	-	Note 1
Amine concentration Offline lab result	%wt	50		45	-	55
Reboiler pressure PT-0204	bar(g)	Note 1		Note 1	-	Note 1
Reboiler temperature TT-0108	°C	122		120	-	125
FG flow (absorber inlet) FT-0007	kg/h	937		750	-	1124

Note 1 - Confidential information

It should be noted that the AMD has been in operation for a longer continuous period in July as well as for shorter periods at a time from October onwards (see Chapter 4.3).

The carbon filter (operating on a slipstream of the lean amine flow) was not tested during the operational period of the pilot plant (due to reasons of pilot downtime when test was originally planned). However, it was tested after the extended test campaign was complete, i.e. when the solvent was circulated without flue gas flow through the absorber. It was tested for a long enough period to treat the entire inventory of solvent (see Chapter 4.7.2).

3 TEST PROGRAM OVERVIEW

3.1 June to August

The main purpose of the tests performed between June and August have been to:

- Monitor amine degradation over time (Test 1)
- Vary the inlet and outlet temperatures of the absorber with the purpose to track its effect on amine emissions and CO_2 capture efficiency. All tests also included a rapid/sudden reduction followed by a rapid/sudden increase in flue gas flow. (Tests 3 - 7)
- Test the effect on amine emissions when operating the plant without water wash both with (Test 9) and without (Test 8) AMD in operation
- Test long term AMD operation to verify stable operation (Test 10)
- Adjust steam flow to achieve various pre-determined CO_2 capture efficiencies (Tests 11 to 13)
- Test the effect of the temperature reduction across the water wash section (Tests 14 - 17)

For detailed information about the test program, see Attachment 1 (June to August test plan.pdf). For detailed information about the test results, see Chapter **Error! Reference source not found.**.

It should be noted that some changes were made to the program:

- Test 2 (test effect of polluting sample hose with amine) was not successfully performed as the pilot plant was not in operation during the test.
- Test 16 was modified to test the effect of 0°C temperature difference across the water wash section (instead of 6°C).
- The outcome of Test 18 (increasing stripper inlet temperature) was inconclusive due to operational upsets.

3.2 September to December

The main purpose of the tests performed between September and December have been to:

- Continue to monitor amine degradation
- Optimise steam consumption by altering key operating parameters
- Understand and quantify the impact of operating the pilot plant with and without the AMD in operation
- Test the carbon filter (although not needed to improve operation)

For detailed information about the test program, see Attachment 2 (September to December test plan.pdf). For detailed information about the test results, see Chapter **Error! Reference source not found.**.

It should be noted that changes were made to the program:

- Most of the tests planned after AMD testing were cancelled (as less important) in favour of getting more data about the impact of operating with and without AMD.

4 RESULTS - OVERALL

4.1 General

This chapter presents the main results obtained during the months June to December, while specific test series / weeks have been discussed / presented in Chapter 6.

The amount of flue gas into the pilot plant has been around the design value (937 kg/h) from mid-August onwards (see Figure 2). Between mid-July to mid-August the flow was at the lower range of acceptable, due to limitations in blower capacity (when WtE line K3 is not in operation).

The amine emissions (as measured by PTR-TOF-MS) have been low for most of the extended phase (see Figure 4), with a median value as low as 0.01 ppmv. Figure 3 below shows the distribution of average hourly emission values for the extended phase and e.g. that 80% of the time, the amine emissions have been below 0.05 ppmv. The average for the entire period was 0.12 ppmv (target <0.4 ppmv), including the intentionally induced amine emission tests and the high amine emissions experienced during weeks 42 to 48. It has been possible to provide explanations for all significant emission peaks / spikes and excursions, most noteworthy those caused by elevated dust concentrations in flue gas from K3.

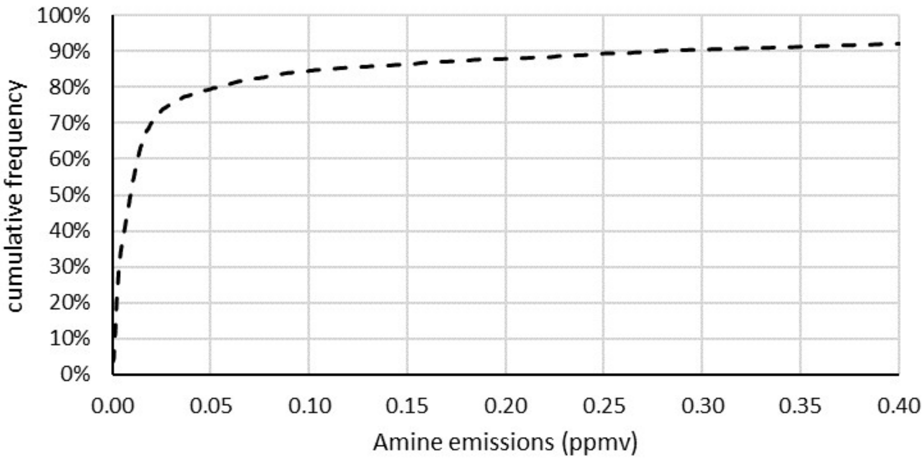


Figure 3. Cumulative frequency distribution of hourly amine emissions during the extension phase, representing around 3100 hours of operation.

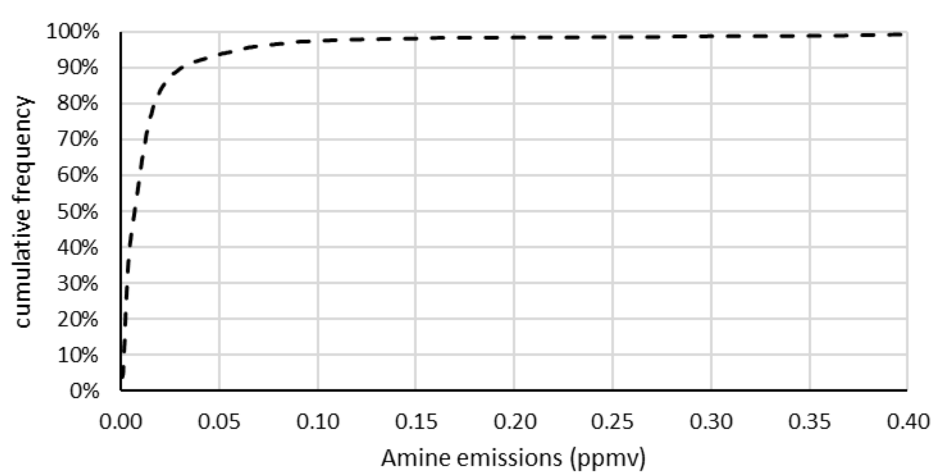


Figure 4. Cumulative frequency distribution of hourly amine emissions during the extension phase, but excluding weeks 42 onwards, representing around 2500 hours of operation.

The CO_2 capture efficiency has been allowed to vary, especially during the steam consumption optimisation testing. At other times, it has in general been maintained around

90% (noting that the incoming flue gas source has varied between K1 to K3). In addition, much of the fluctuations in CO_2 capture efficiency can be attributed to intentionally induced process upsets.

4.2 Emissions to air

According to information from UiO, the levels of other (than the main solvent) amine in the treated flue gas stream have not been significant during the entire extended test period (June to December). However, a new degradation product, called Deg 4, was detected in the emissions to air around August 28 and 29 coinciding with an extreme absorber temperature test of 60°C (representing absorber inlet and outlet FG temperatures). UiO confirms that this new degradation product was not detectable before August and that the concentration of Deg 4 in the gas is normally low (below 5 ppbv) and will not impact the 0.4 ppmv emission limit. Since August, Deg 4 has been reported/detected reaching levels up to 60 ppbv (hourly average), in general coinciding with the amine (Am 1) emission peaks reported below.

The results presented here consist of PTR-TOF-MS measurements that account for all amine emissions above 5 ppbv (reporting limit). It should be noted that including Deg 4 to the below graphs does not change the appearance of the graphs significantly nor the conclusions and therefore the reported total amine emissions consist of only one type of amine, called Amine 1 (Am 1).

Below graphs have been created using 1-hour average data. Figure 5 emphasises the fact that the 24-hour average data (black-dotted line) has stayed below the 0.4 ppmV target level for the entire period, except for the period of FG from K3 with unusually high dust concentration. Figure 6 highlights the emission peaks that have been detected (mainly related to pilot plant trips or testing purposes). Items number 9, 10 and 11 in Figure 6 are discussed in more detail in Chapter 4.2.1.

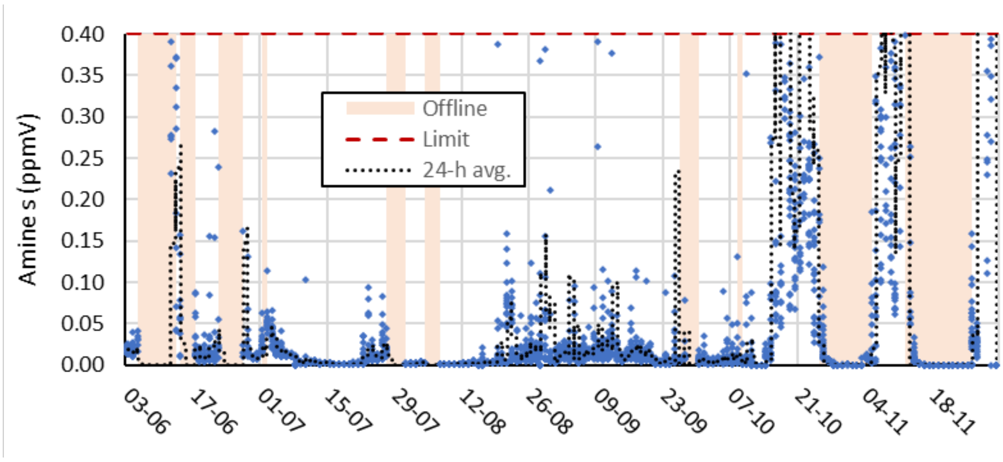


Figure 5. Total amine emissions measured in the period between June and August based on hourly averages. The black dotted line represents 24-hour averages and the red dashed line represents the long-term average emission limit.

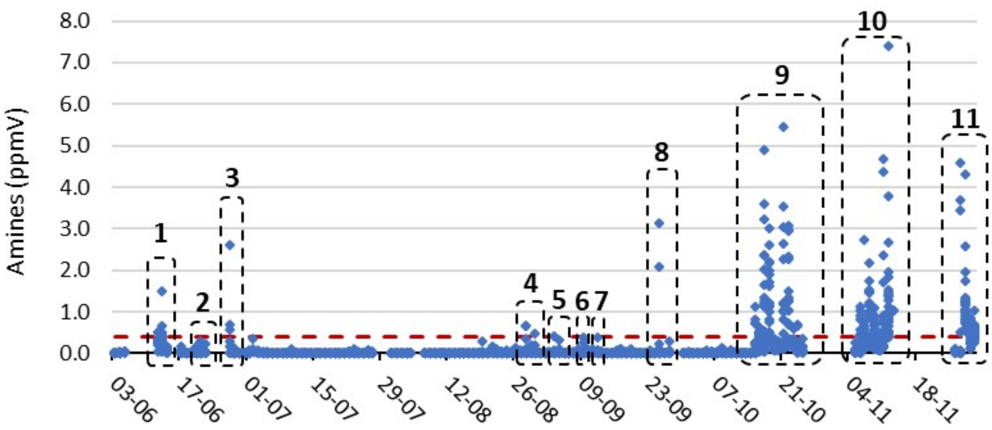


Figure 6. Total amine emissions measured in the period between June and August based on hourly averages (same data, but different scale than Figure 5). The most significant emission peaks have been indicated with numbers and the red dashed line represents the long-term emission limit.

In Figure 6, the most significant emission peaks have been indicated and below is an explanation for each of the instances:

1. Multiple pilot plant trips and subsequent restarts
2. Absorber inlet and outlet temperature testing
3. Plant restart after trip
4. Absorber temperature test (60°C) combined with rapid changes in flue gas flow
5. Rapid changes in FG flow amount
6. Suspected (missing data for K3) FG compositional change
7. Sudden drop in FG flow (and negative delta T over water wash section)
8. Sudden drop, followed by sudden increase in CO_2 conc. (Smaller peak under #8 related to starting and stopping the pilot plant)
9. Induced emissions testing (with and without AMD) coinciding with unusual dust levels from K3. See Chapter 4.2.1 below for more information.
10. Induced emissions testing (with and without AMD) coinciding with unusual dust levels from K3. See Chapter 4.2.1 below for more information.
11. Induced emissions testing (with and without AMD) coinciding with unusual dust levels from K3. See Chapter 4.2.1 below for more information.

4.2.1 Emissions in week 42 and 48

Week 42 to 48 represented weeks of unusually high amine emissions at the pilot plant as can be seen from Figure 6. The high amine emissions correlate well with unusually high dust levels measured at K3 (at the WtE plant) as can be seen in the Figures below. Unfortunately, there is no size distribution data for the dust in question and it can only be suspected that submicron sized particles and/or aerosols have been present causing the emission peaks.

The reasons for why K3 has periods with more dust being emitted is related to the operational logic of the electrostatic precipitator (ESP). It is possible to see that the ESP outlet flue gas dust concentration and voltage across the ESP are correlating. A sometimes lower voltage is related to the flue gas composition, moisture, dust concentration and composition etc., which in turn affect the breakdown voltage (or sparkover). When sparkover occurs, the high voltage load is rapidly turned off and on again until a new sparkover has occurred at a new level of voltage. This is considered as an upset condition and will most likely not occur frequently (did not take place during entire 2018 for instance).

Note that the periodic (every 4 hours) peaking dust levels seen in the Figures below are related to the cleaning of the ESP equipment. These, cleaning induced peaks, do not correlate with emissions at the CC plant. In addition, note that the pilot plant was not in operation during weeks 44 and 47 and therefore no graphs for these weeks can be presented.

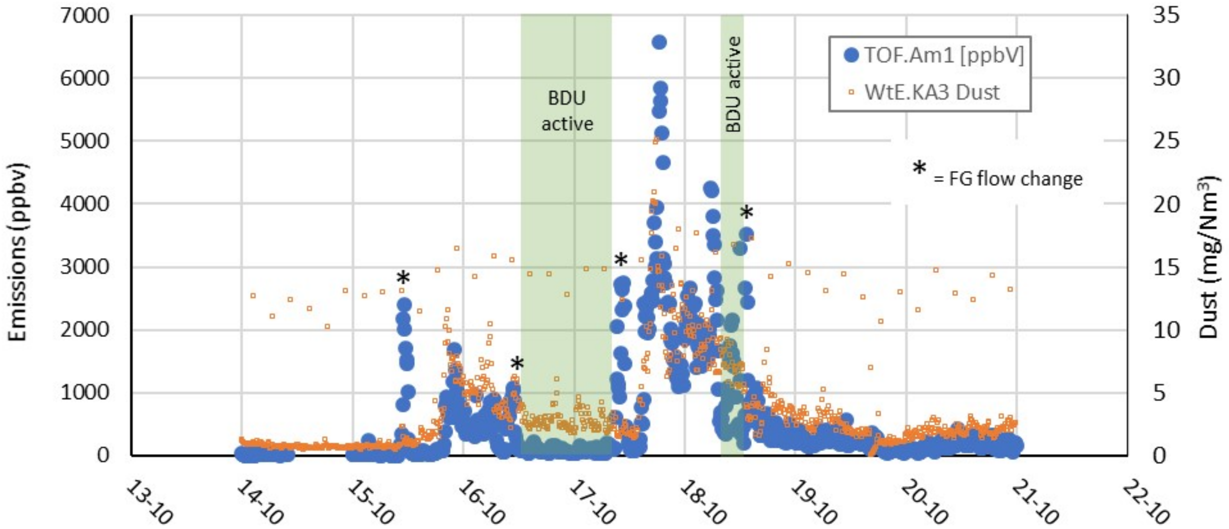


Figure 7. Week 42 - Amine emissions together with dust measurements of K3 flue gas. AMD in operation has been indicated, as well as instances of induced process upsets / FG flow change. Note that the periodic peaks, every 4 hours, visible in the dust data are related to the ESP cleaning cycle.

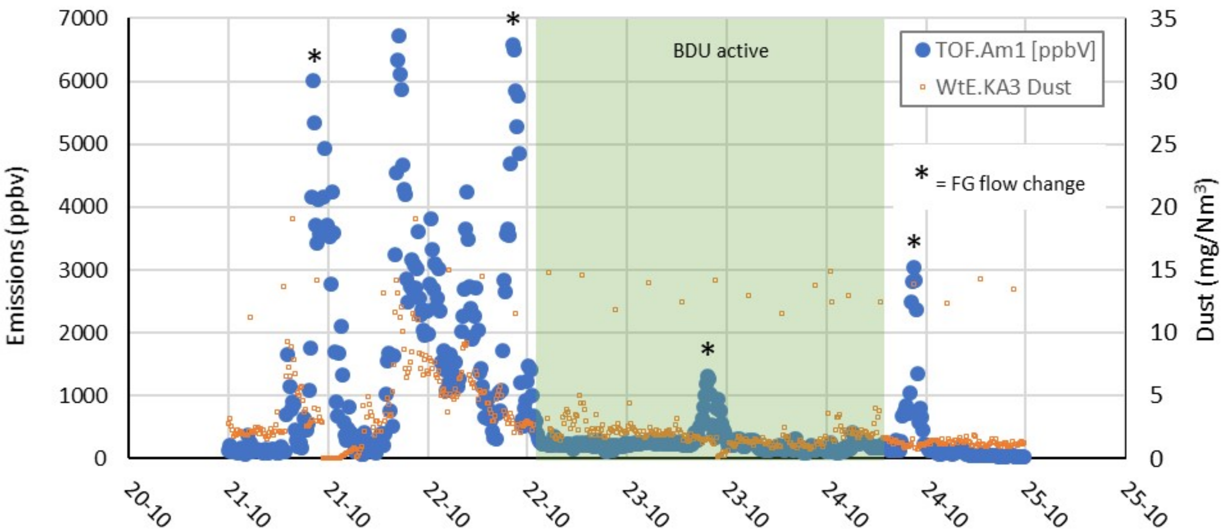


Figure 8. Week 43 - Amine emissions together with dust measurements of K3 flue gas. AMD in operation has been indicated, as well as instances of induced process upsets / FG flow change. Note that the periodic peaks, every 4 hours, visible in the dust data are related to the ESP cleaning cycle.

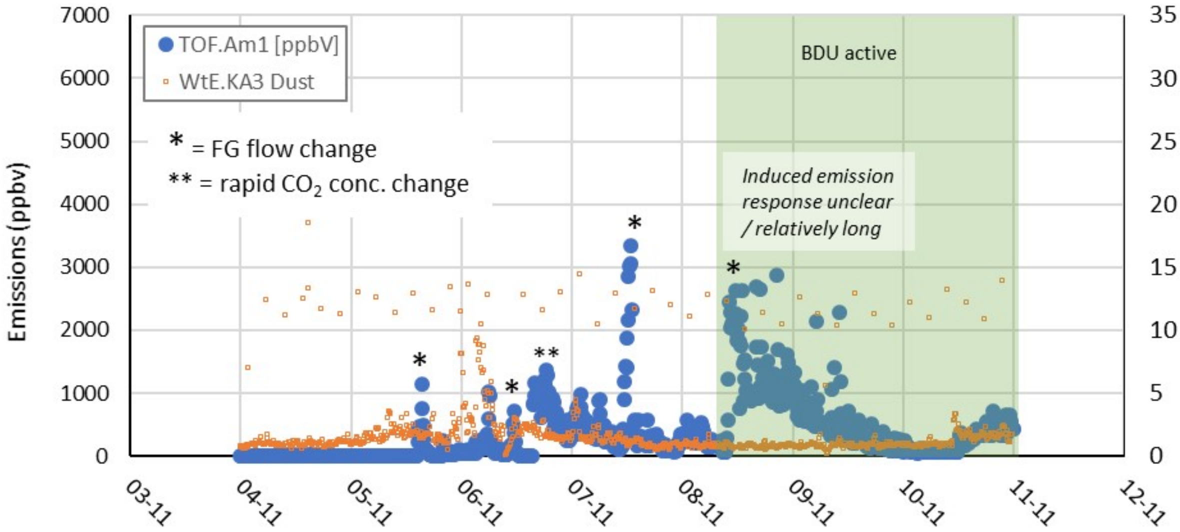


Figure 9. Week 45 - Amine emissions together with dust measurements of K3 flue gas. AMD in operation has been indicated. Note that the periodic peaks, every 4 hours, visible in the dust data are related to the ESP cleaning cycle. Also note that the reasons for the relatively intensive amine emission response following the induced upset conditions November 8th are unclear.

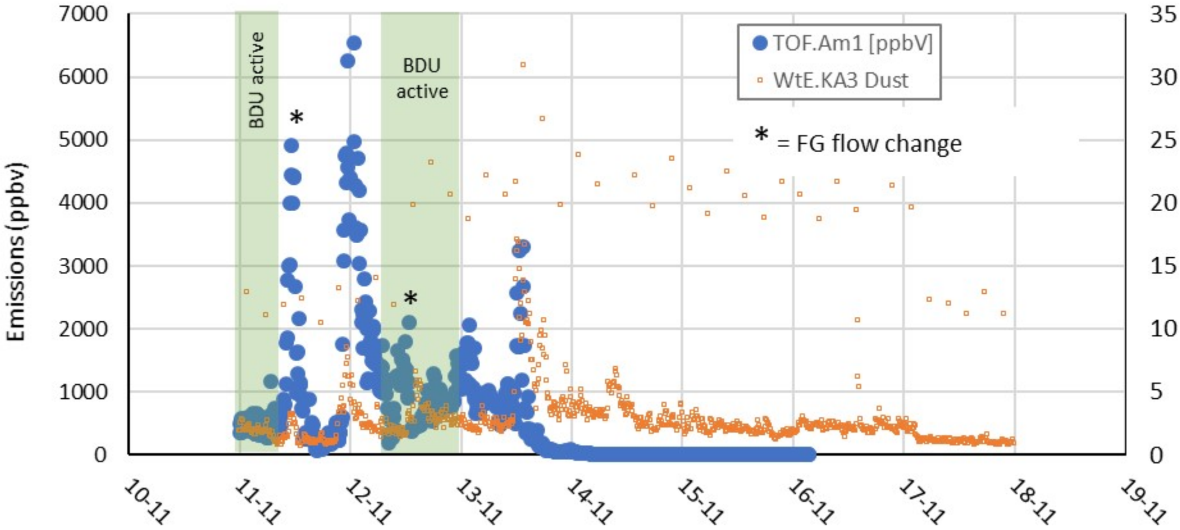


Figure 10. Week 46 - Amine emissions together with dust measurements of K3 flue gas. AMD in operation has been indicated, as well as instances of induced process upsets / FG flow change. Note that the periodic peaks, every 4 hours, visible in the dust data are related to the ESP cleaning cycle.

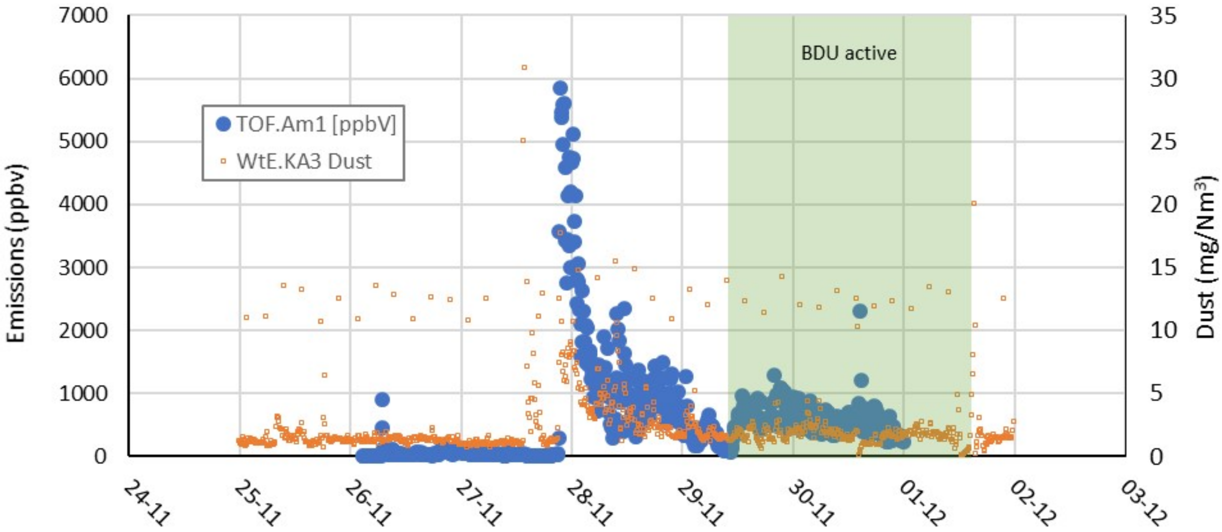


Figure 11. Week 48 - Amine emissions together with dust measurements of K3 flue gas. AMD in operation has been indicated. Note that the periodic peaks, every 4 hours, visible in the dust data are related to the ESP cleaning cycle.

4.3 AMD operation

While the AMD was operated continuously for a prolonged (3.5 weeks) period in July, it did not become clear how much the AMD was reducing/impacting the amine emissions. Therefore, a number of subsequent testing weeks (week 41 onwards) were committed to quantifying the amine emissions with and without AMD in operation.

During normal operation, the amine emissions are very low both with and without the AMD in operation (see Figure 12) and therefore the target was to simulate (by rapidly changing the FG flow) upset conditions in order to quantify the effect of the AMD on amine emissions.

4.3.1 Stable operation

In addition to referring to previous pilot plant result reports regarding low amine emissions during stable operation without AMD, a graph is given below (Figure 12) with representative data obtained during stable operation. Each data point represents average values over significant periods of time and while comparative enough for the purposes here, does contain varying operating conditions (capture efficiency, CO_2 concentration, L/G etc.). All of these have been plotted in the same graph, essentially showing that during normal operation the difference between operating with and without AMD is negligible in terms of amine emissions.

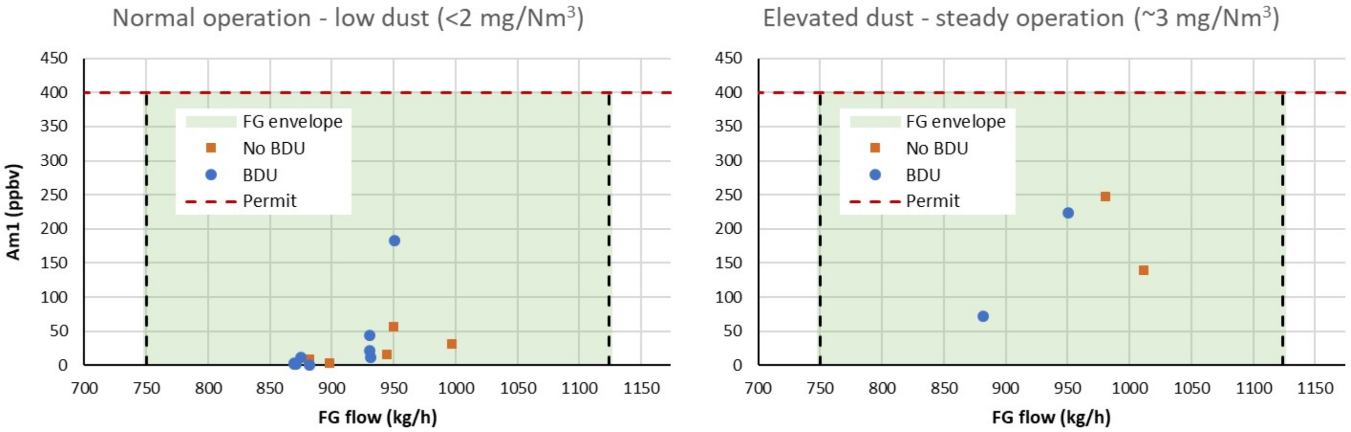


Figure 12. Average amine emissions both with and without AMD in operation during normal (left figure) and elevated FG dust conditions (right figure). The highlighted area (FG envelope) represents the acceptable operational range for the pilot plant in terms of FG flow and amine emissions.

While the pilot plant operation was not very stable during dusty conditions from K3, it has been possible to obtain periods of time (of at least 1 hour) with fairly stable operation even during elevated dust conditions as those plotted in the right graph of Figure 12. These have been further plotted against the average dust content measured during that time in the below Figure.

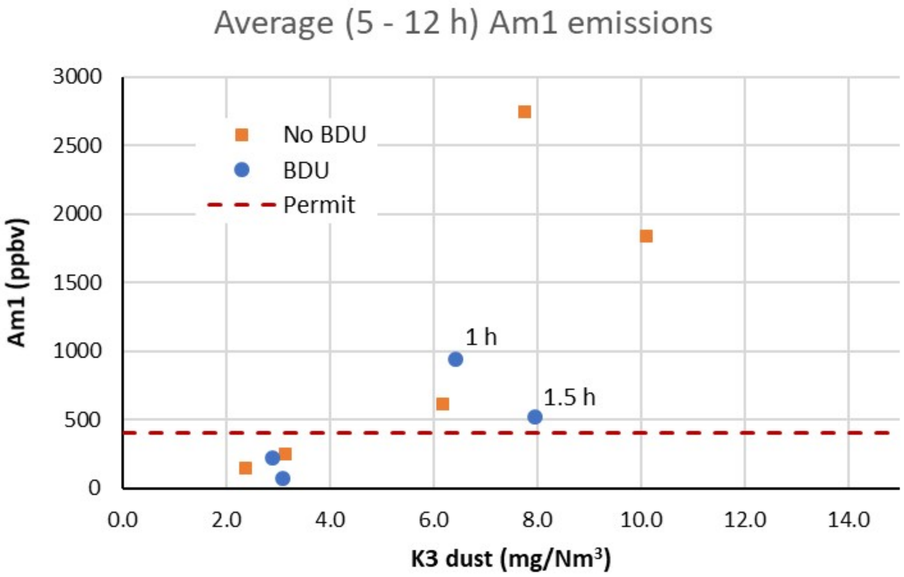
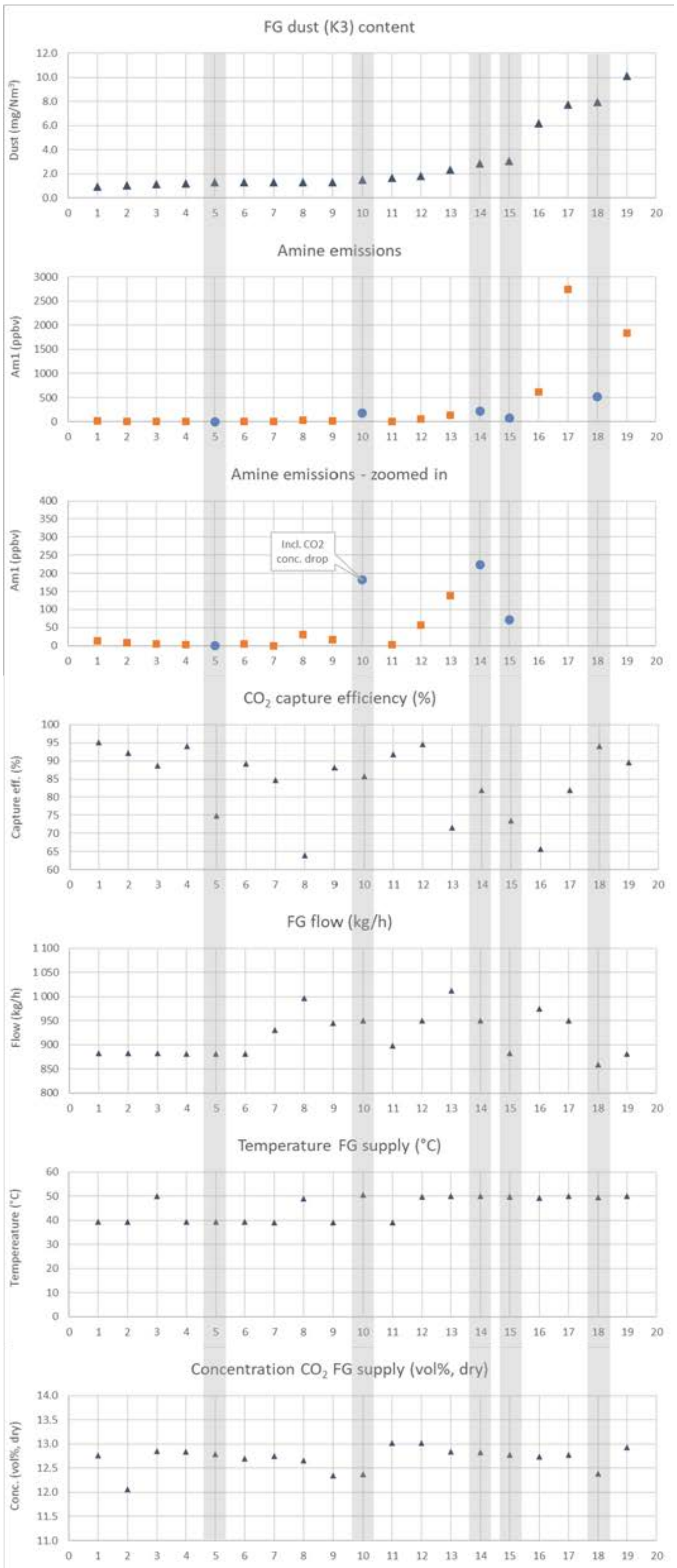


Figure 13. Prolonged (5 to 12 hours) amine emission levels with and without AMD in operation during unusually high dust concentrations from K3. Each data point, except when marked otherwise, represent average values between 5 and 12 hours of duration.

Another way to visualise the impact of running the pilot plant with and without AMD in operation is presented below. Here (Figure 14), key parameters have been plotted on individual graphs above each other, each data point being the average value for the same stable period of time with the ones directly above or below. The start time and length of the time intervals have been provided in the table next to the graphs.

ID	Start time	Duration (h)
1	05-10-2019 00:00	8.5
2	07-10-2019 01:00	9.0
3	10-10-2019 22:00	6.0
4	03-10-2019 20:00	8.0
5	09-10-2019 22:00	6.0



6	02-10-2019 19:00	10.0
7	11-10-2019 23:00	6.0
8	14-10-2019 22:00	6.0
9	14-10-2019 00:00	5.0
10	23-10-2019 15:00	6.0
11	02-10-2019 00:00	7.0
12	24-10-2019 14:00	13.0
13	21-10-2019 00:00	5.0
14	22-10-2019 15:00	12.0
15	16-10-2019 23:00	6.0
16	15-10-2019 22:00	6.0
17	21-10-2019 20:00	10.0
18	18-10-2019 07:30	1.5
19	17-10-2019 23:00	6.0

Figure 14. Average key parameters for pilot plant operation with and without AMD in operation. X-axis is a sequential number indicating a stable operational period, arranged according to increasing FG dust content. Grey highlighted data points represent stable operational periods with AMD in operation.

4.3.2 Upset conditions

Upset conditions were generated by rapidly decreasing the FG flow through the absorber both with and without the AMD in operation. Representative/comparative data from these experiments have been collected in the below Figures. Because, at the time of testing, the FG contained unusually high amounts of dust (originating from K3), the data has been plotted against K3 dust level.

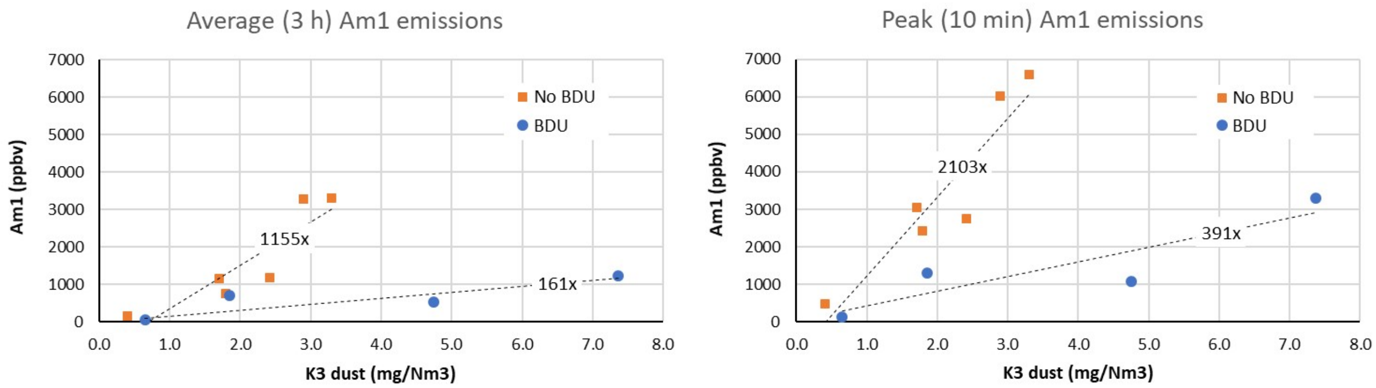


Figure 15. Amine emissions as a function of K3 dust content both with and without AMD in operation. The left figure represents 3-hour average amine emissions, while the right figure represents peak/maximum 10-minute amine emissions around the time of the simulated upset conditions. The dotted lines indicate the slope for a linear fit of the data.

From Figure 15 it can be seen that the AMD reduces both the intensity (right-hand Figure) and the duration (left hand Figure) of the emission peaks. Ideally, all other parameters except for AMD in operation or not, would be exactly the same allowing for a clean comparison. However, since this has not been the case, especially with regards to FG composition (dust content), quantifying the emission reduction capacity of the AMD is not straightforward. However, an attempt to quantify the emission reduction capacity has been made based on the data in Figure 15 by estimating the difference in the linear slopes. In case of average amine emissions, the reduction factor was 7 times and in case of peak emission intensity, the reduction factor was 5.

4.4 CO_2 Capture efficiency

Below are two Figures showing the capture efficiency as a function of time for the period June to August and September to December, respectively. The data for CO_2 capture has been based on 10-minute averages.

The continuously (assuming similar pressure and temperature conditions) estimated capture efficiency shown below is based on:

$$CCCC_2\ (sscccccccssee) - CCCC_2\ (cccccccccccctt\ FFFF) VV\dot{}_{CCCC2,sssssssssss}$$

$$-VV\dot{}_{CCCC2,tttttttttttttttt}$$

$$CCCC2\ ccccccccccccc\ cceeeeeecceeecceeccee = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ \quad \_\_\_\_\_\_\_\_\_\_\_\_\_\_CCCC\ (sscccccccssee) \quad = \quad VV_{CCCC2,sssssssssss}$$

2

Where,

$$\dot{VV}_{CCCC2,tttttttttttttttt} =$$
$$ee(mm\dot{m}_{ttFF-0011}, cc_{PPFF-0012}, TT_{FFFF-0013}, xx_{CCCC2,tttttttttttttttt,RRRRCCRR})$$

The concentration of CO_2 is not continuously measured at the outlet of the absorber, instead occasional manual (spot value) checks are done by shifting the CO_2 measurement device to the outlet of the absorber (*Efficiency = 1 - [$CO_{2,\ treated\ FG}$ / $CO_{2,\ Supply\ FG}$]*). The results of both the CO_2 measurement estimates and the spot checks have been presented in the Figures below. For testing purposes, the CO_2 capture efficiency has been allowed to vary considerable during the second half of the extended pilot campaign (see Figure 17).The CO_2 mass balance, indicating the accuracy of the various flow measurements has been fluctuating around +/- 10 kg/h (or +/- 8 wt%).

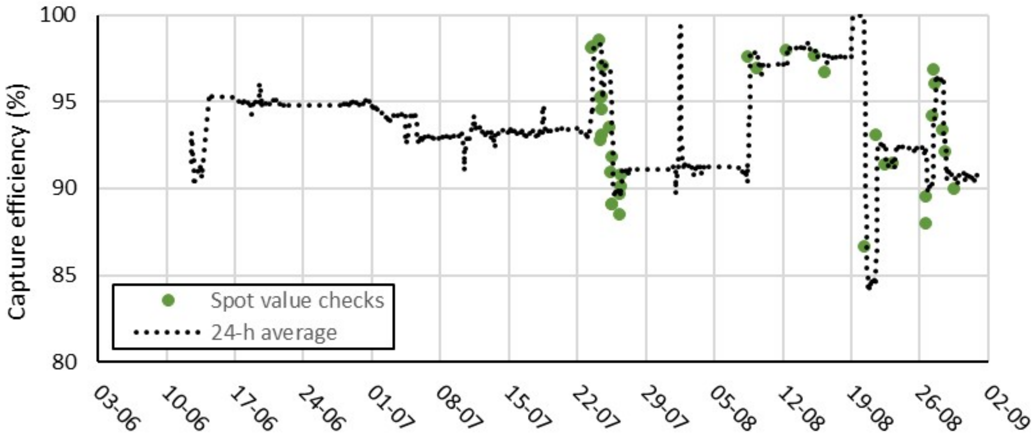


Figure 16. CO_2 capture efficiency based on absorber balance for June to August. The dotted line represents 24-hour running averages.

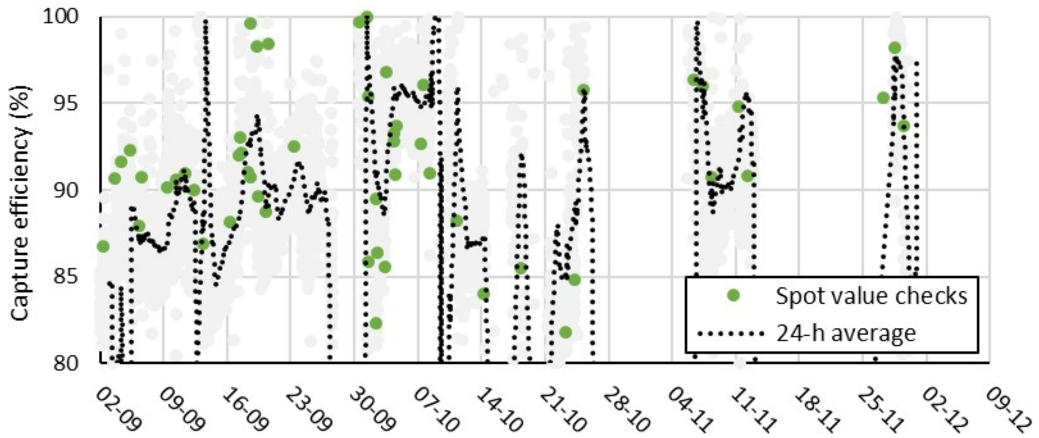


Figure 17. CO_2 capture efficiency based on absorber balance for September to December. The dotted line represents 24-hour running averages (i.e. slightly shifted compared to spot analysis data), while the light grey background data represents 1-hour averages.

4.5 Steam consumption

Although the specific steam consumption (per ton CO_2) measured at the pilot plant is not comparable to that designed for the full-scale plant, a good comparison with simulated results was obtained earlier (see NC03-KEA-P-RA-0016).

During the extended testing campaign (weeks 38 to 40), tests were carried out to optimise the specific steam consumption (GJ/t CO_2). In practice this was done by changing the lean amine and steam flow to predetermined values, while maintaining other parameters as stable as possible.

The below graph shows the capture efficiency obtained for different lean amine to gas flow ratio (L/G) and stripping factor (SF) conditions. All points represent average values during representative/ comparative time periods of various durations (~6 hours, see also Chapter **Error! Reference source not found.**). The lines represent 2nd order polynomial fits to the available data points. Note that for the 93% capture efficiency, there are only two periods of representative data, i.e. only two data points, thus the straight line.

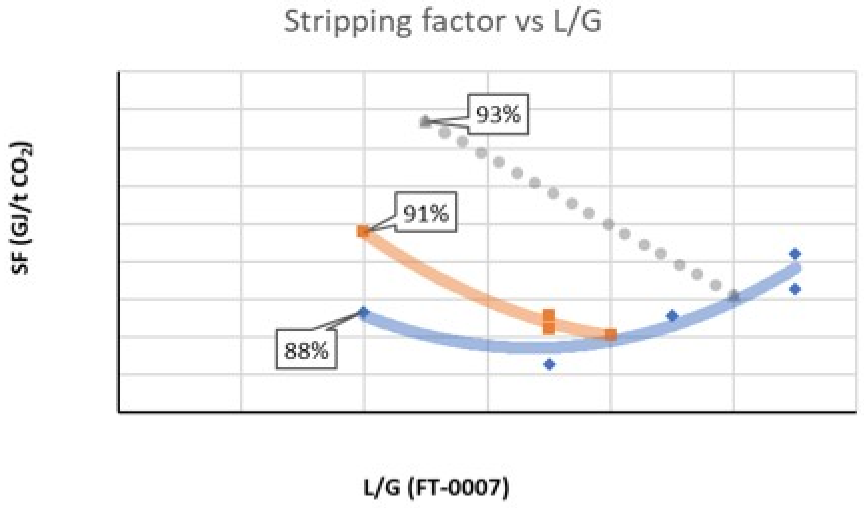


Figure 18. Specific steam consumption / stripping factor as a function of L/G for different capture efficiencies (88, 91 and 93%).

4.6 Amine degradation

The Figures below show the progression of amine degradation products over time (as measured by LCMS). Note the difference between Figure 19 and Figure 20. Figure 19 shows degradation as a function of the cumulative operational hours from start of the pilot plant until end of November, while Figure 20 shows the degradation as a function of when the sample was taken (i.e. irrespective of pilot plant up/downtime).

The total amount of degraded products (measured as received, i.e. on a wet basis) is based on the sum of three known degradation products called Deg 1, Deg 2 and Deg 3. Work was performed to validate if Deg 4 (or any other significant amine-based degradation components) could be detected in the solvent.

From the Figure below, it can be seen that the typical design range for degradation products in the solvent has been exceeded after around 3000 operational hours and that there is a notable upwards trend in the otherwise fairly linear curve of total degradation products after around 3 wt%. Additional operational hours would be needed to show if the build-up rate of degradation products in the solvent is indeed accelerating. It should be noted that this apparent acceleration also coincides with the elevated dust conditions experienced during the last phase of testing.

Plotting the data against time (i.e. including both operational and non-operational hours), shows a more linear trendline, indicating that degradation could have continued during downtime. Normally this not the case as most of the solvent stays cold and lean, but in case of repeated trips where rich amine in the stripper just falls to the bottom, where it remains hot for some time, this could have happened.

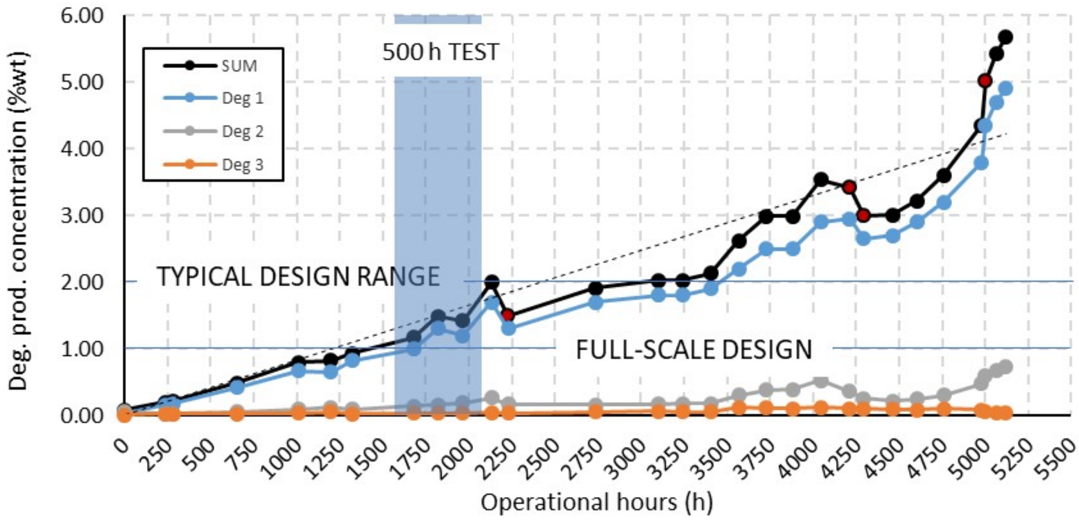


Figure 19. Amine degradation as measured by LCMS as a function of operational hours. The red-filled dots represent average values of samples that have been analysed twice.

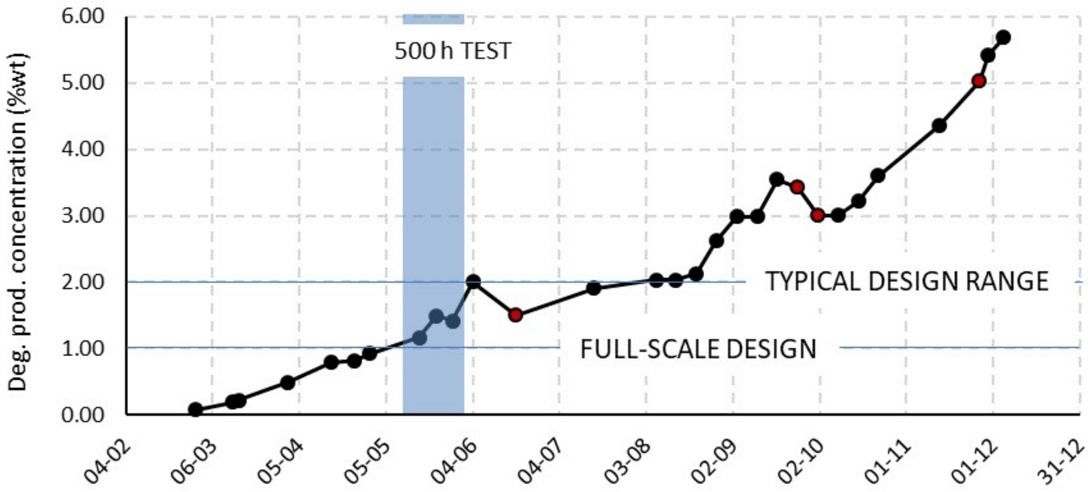


Figure 20. Amine degradation as measured by LCMS as a function of time regardless of operational hours. The red-filled dots represent average values of samples that have been analysed twice.

Results of a comparison between LCMS and titration are provided below, where the amine concentration determined by LCMS is the sum of both amines and degradation products. The previously (during the first 2 000 hours) noted small difference between the two methods indicated that no significant (unknown) amine species have been omitted. However, this trend is broken by a number of samples throughout the extended test campaign and the reason is not fully clear. There appears to be a correlation between the distance/delta from the linear regression line (Figure 19) to that of the difference/delta between the LCMS and titration results (Figure 21), but further work is needed to verify this and how results should be interpreted.

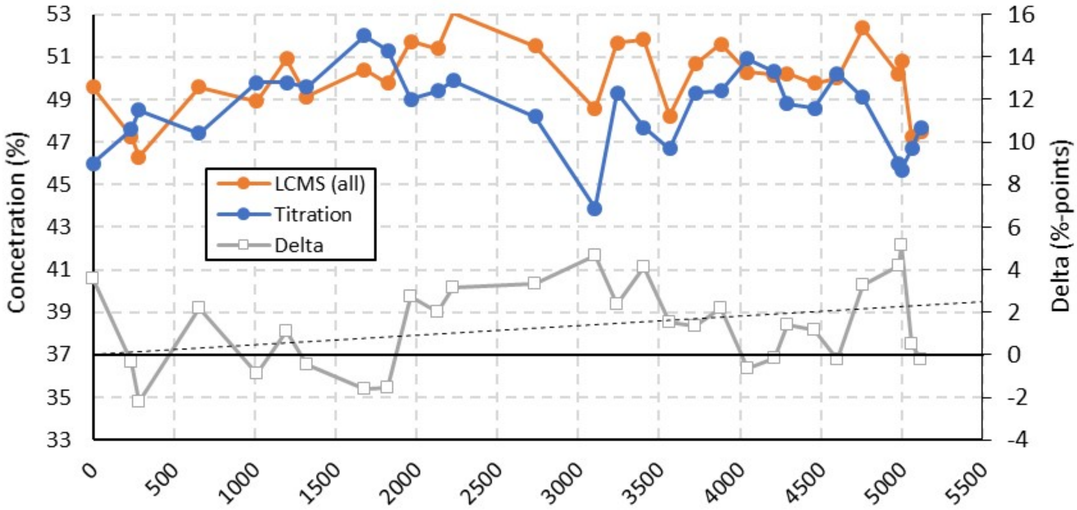


Figure 21. Comparison of amine titration results for lean amine samples to amine concentration as determined by LCMS method.

4.6.1 IC and ICP-MS analysis results

A number of lean amine samples have been analysed by both IC (anions) and ICP-MS (cations). This information is useful to determine the requirements for TRU operation as the anions would also need to be removed with the TRU. The below Figures present the results from the analyses in question.

With regards to the organic acids presented in Figure 22, the results are as expected and the findings from before [ref. NC03-KEA-P-RA-0016] remain valid; that the "accumulation rates of these compounds are orders of magnitude below the main degradation products accumulation rate". Furthermore, it should be noted that the levels detected so far are considerably lower than those experienced in full-scale plants using solvent DC-103.

The ICP-MS results show low ingress of contaminants, but it appears as if the high dust loads from K3 are also visible in the final results.

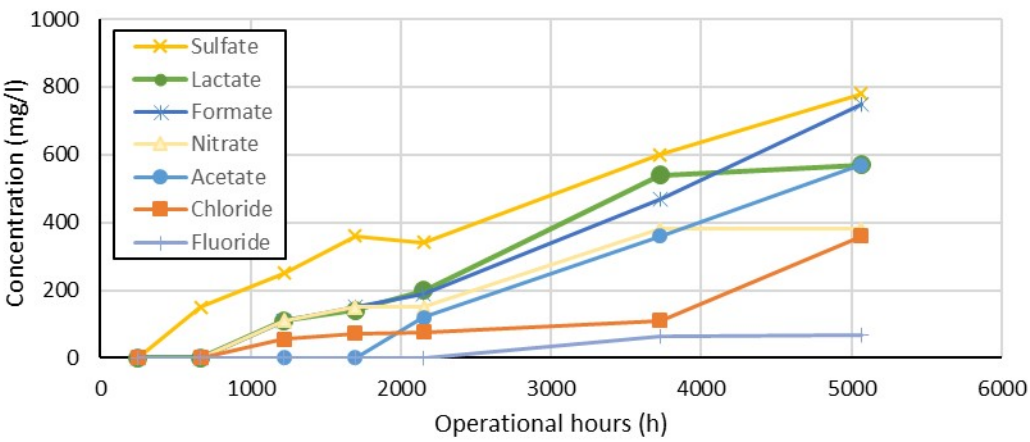
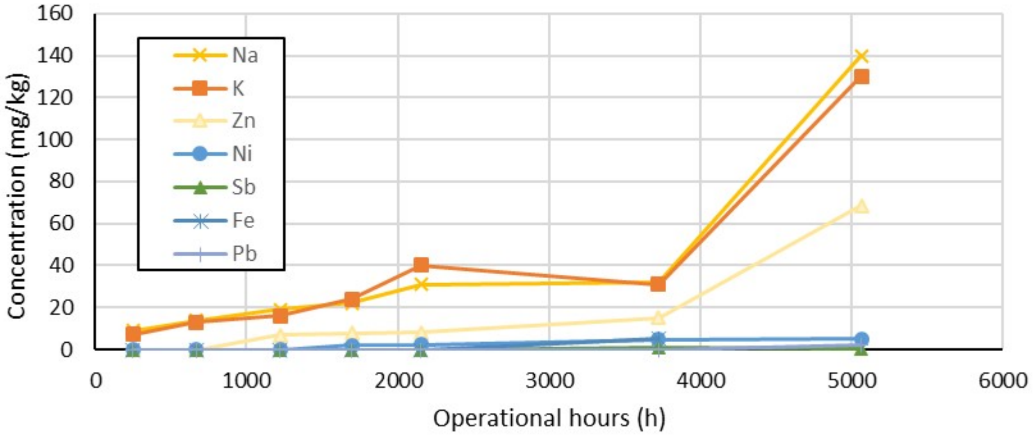


Figure 22. Anions in lean amine samples to date (above detection limits)

The following anions were not detected (with detection limit in parenthesis): thiosulfate (<100 mg/l), butyrate (<100 mg/l), propionate (<100 mg/l).



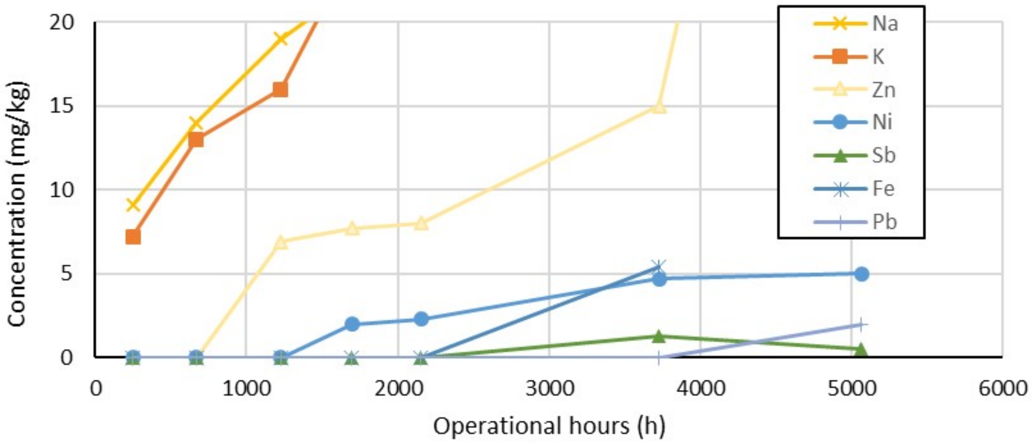


Figure 23. Two graphs showing cations in lean amine samples to date (above detection limits). Same data, but different y-axis scales.

In addition to the seven cations (Na, K, Zn, Ni, Sb, Fe and Pb) shown in the Figure above, the following Table shows the (during operational hours)[3] not detected cations together with their detection limits.

Table 2. Detection limits, in mg/l, for cations not detected in lean amine samples during operational hours.

Mg	Al	Ca	V	Cr	Mn	Co	Cu	As	Mo	Cd	Sb	Ba	Hg	Tl
2.0	2.0	8.0	1.0	1.0	5.0	1.0	5.0	1.0	2.0	0.2	0.5	1.0	0.1	1.0

4.7 Other results

In addition to the main results discussed above, the following measurements were performed between June and December (1. and 2), in addition to one (3) measurement performed in September 2020:

1. CO_2 product (after stripper condenser) measurements using PTR-TOF-MS
2. Additional foaming tests in conjunction with testing the activated carbon filter 3. Analysis of particle settlement sample from lean amine tank

[3] Following the normal operational hours, the pilot plant was operated without FG and having solvent circulation on to test the impact of the active carbon filter. The ICP-MS results showed elevated concentrations of Mg, Ca and traces of Cd. More information in Chapter 4.7.2.

4.7.1 CO_2 product purity

One additional PTR-TOF-MS measurement campaign on the CO_2 product line was performed during 11-14 October 2019. The results were in line with the previously presented data.

Table 3. Trace components in CO_2 product as reported/measured by PTR-TOF-MS during 1114.10.2020. Where available, previously reported values in parenthesis.

	Acetaldehyde [ppbV]	Formamide [ppbV]	Acetone [ppbV]	C4H6O2 [ppbV]	C5H6N2 [ppbV]	C6H8N2 [ppbV]	Am1 [ppbV]
Max	718 (2400)	76	24	28	11	25	3 (83)
Avg.	587 (800)	59	21	24	7	15	1 (3)

In addition, the main results from the CO_2 bag sample taken during the first 2 000 hours are:

- Acetaldehyde measured at 0.5 ppmv, fairly consistent with average value of 0.6 (Table above) and 0.8 ppmv as measured previously by PTR-TOF-MS (ref. NC03-KEA-P-RA0016)
- Confirmation that all components are within CO_2 product specification, including O_2.

4.7.2 Activated carbon filter operation and foaming tests

The activated carbon filter (ACF) was not tested during the logged (around 5 100) operational hours due to the fact that the pilot plant was down when the testing was planned. Thus, it ended up being tested without flue gas from the WtE plant, having only the solvent circulation pumps turned on. The solvent circulated (with partial flow) through the filter between 5 - 9.12.2019. Solvent samples were taken both before and after the ACF test to be analysed for foaming tendency. Both samples showed reduced tendency towards foaming compared to earlier samples, see Figure 24.

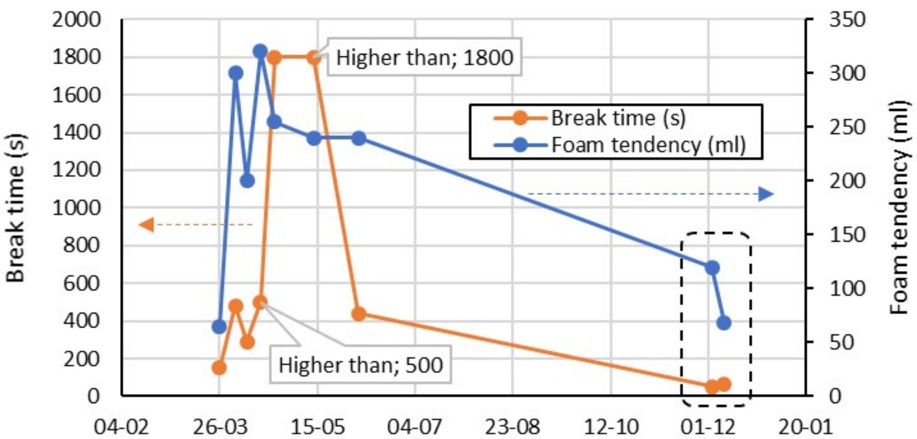


Figure 24. Foaming tendency and foam break time results for entire pilot plant test campaign. The dotted square indicates the results of the samples taken before and after ACF testing.

In addition to testing for foaming tendency and break time, the samples before and after ACF were analysed with IC and ICP-MS instruments.

Table 4. ICP-MS analysis results (mg/kg) for samples taken before and after ACF tests.

Sample	Na	Mg	K	Ca	Ni	Zn	Cd	Sb	Pb
Before ACF test	140	<2.0	130	<8.0	5.0	68.6	<0.20	0.54	2.00
After ACF test	120	120	130	120	<2.0	<5.0	0.48	0.55	<1.0

Table 5. IC analysis results (mg/l) for samples taken before and after ACF tests.

	Analysis results IC (mg/l)									
Sample	**Chloride**	**Fluoride**	**Sulphate**	**Thiosulfate**	**Nitrate**	**Acetate**	**butyrate**	**Formate**	**Lactate**	**Propionate**
Before ACF test	**360**	**66**	**780**	<100	**380**	**570**	<100	**750**	**570**	<100
After ACF test	**440**	**71**	**860**	<100	**420**	**590**	**120**	**670**	**600**	<100

From the above tables it appears as if the activated carbon filter did not have a significant impact on species analyzed by IC. The ICP-MS results however indicate that zinc has been bound to the activated carbon, while the concentration of both magnesium and calcium have increased significantly. It is not unlikely that the ACF contains both of these elements, explaining the observed increase in concentration.

4.7.3 Particle settlement

After de-commissioning of the pilot plant, a sample of particle settlements was retrieved from the bottom of the lean amine tank. This sample have been analysed by Eurofins and the results are summarized in Table 6. The pilot plant was emptied of solvent and flushed with water prior to sampling.

Table 6. ICP-MS analysis results of particulates

Element	Concentration [mg/kg dry matter]	Percentage [%]
Aluminium (Al)	2800	0,4 %
Antimony (Sb)	490	0,1 %
Arsenic (As)	55	0,0 %
Barium (Ba)	1200	0,2 %
Mercury (Hg)	0,63	0,0 %
Cadmium (Cd)	11	0,0 %
Potassium (K)	200	0,0 %
Calcium (Ca)	3600	0,5 %
Cobalt (Co)	1000	0,1 %
Chromium (Cr)	82000	12,0 %
Copper (Cu)	2000	0,3 %
Lead (Pb)	6200	0,9 %
Magnesium (Mg)	1200	0,2 %
Manganese (Mn)	7300	1,1 %
Molybdenum (Mo)	6400	0,9 %
Sodium (Na)	220	0,0 %

Nickel (Ni)	48000	7,0 %
Iron (Fe)	520000	76,0 %
Zinc (Zn)	1500	0,2 %
Thallium (Tl)	0	0,0 %
Vanadium (V)	330	0,0 %
Element	**Concentration [mg/kg dry matter]**	**Percentage [%]**
		100,0 %

All elements >1% corresponds to the composition of stainless steel (316SS).

5 SUGGESTIONS FOR FURTHER WORK

This chapter represents suggestions for further work based on the operation of the Klemetsrud CC pilot plant. The work suggestions have been divided to purely desktop studies (requiring no experimental work) and other work requiring at least some experimental work.

5.1 Desktop studies

5.1.1 Improve understanding of observed degradation rate behaviour

Re-evaluate and compare data and knowledge related to the degradation of the solvent obtained during Klemetsrud pilot plant operation with other parties:

- CCS knowledge centre
- Saskatchewan / Boundary Dam
- TCM

5.1.2 Operational experience report for future CC plant operators

- Custom-made test reports for future FOV pilot and full-scale plant operators.

5.2 Desktop and experimental studies

5.2.1 Quantify the effect from various upset conditions from the WtE plant

Investigate the impact of different treatment and combustion processes and equipment in the WtE plant on the carbon capture plant. Some examples of parameters to be looked into are:

- Dust type (concentration and origin)
- Dust particle size distribution (ESP/baghouse)
- Dust amounts
- Effect of acid gases on degradation (different flue gas treatment)
    - o Noting, that unlike during the pilot operation, the pre-scrubber in the full-scale plant will be operated with caustic injection to reduce the impact of acid gases further

5.3 Experimental work

5.3.1 Further pilot plant testing suggestions

- Additional stable operation with and without AMD o With degraded solvent o With new/undegraded solvent
- Collect more data (potentially re-evaluate existing data) for stripping factor vs capture efficiency
- Investigate the effect of active carbon filter (different types) in continuous operation
- Prolonged operation under extreme conditions (high temperature in absorber)

6 RESULTS - DETAILED

The purpose of this chapter is to present additional details regarding individual test series as listed in Chapter 3.1 (June to August) and individual test weeks as listed in Chapter 3.2 (September to December). The main results and findings have been presented in the preceding chapters and the below represents the results as reviewed during weekly pilot plant follow-up meetings.

The detailed results are confidential information and, thus, retained by FOV.