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Report

WP 1 and 3 in the project: CCM TQP amine 6 – Emission Quantification and Reduction

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ABSTRACT

Emission quantification at Maasvlakte CO_2 capture pilot plant

The present report describes findings of a study of emissions from a CO_2 capture pilot plant. MEA (2-ethanolamine) was chosen as the solvent for the campaign. The main focus was on studying solvent emissions, but analysis has also been carried out for degradation products formed in MEA. Solvent emissions can be separated into three contributions: gas phase emissions, aerosols (also referred to as mist) and droplets of entrained solvents. For the emission campaign at Maasvlakte aerosols were found to be the major contributor to overall emissions. Entrainment was found to be a very small contributor to overall emissions. A Brownian demister unit (BDU) was tested as an emission reducing technology. It was found to be very efficient in reducing aerosol emissions. The emission levels at the CO_2 capture plant were higher than initially anticipated. There are several factors that may account for this. The exhaust gas may have a high content of condensation nuclei. This can in combination with temperature gradients in the absorber result in high solvent concentration in mist.

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APPENDICES

Executive Summary

The present report describes findings of a study of emissions from a CO_2 capture pilot plant at the Maasvlakte coal power station in The Netherlands. 30 wt% MEA (2-ethanolamine) was chosen as the solvent for the campaign. The main focus was on studying solvent emissions, but analysis has also been carried out for degradation products formed in MEA.

Solvent emissions can be separated into three contributions: gas phase emissions, aerosols (also referred to as mist) and droplets of entrained solvents. For the emission campaign at Maasvlakte aerosols were found to be the major contributor to overall emissions. Entrainment was found to be a very small contributor to overall emissions.

A Brownian demister unit (BDU) was tested as an emission reducing technology. It was found to be very efficient in reducing aerosol emissions.

The emission levels at the CO_2 capture plant were higher than initially anticipated. There are several factors that may account for this. The exhaust gas may have a high content of condensation nuclei. This can in combination with temperature gradients in the absorber result in high solvent concentration in mist.

The extent of aerosol emissions would most likely be lower in plant treating exhaust gas from a natural gas fired power plant. The reasons are lower content of condensation nuclei in the exhaust gas and that the magnitude of the temperature bulge in the absorber is lower due to lower CO_2 content in the exhaust gas compared to the exhaust gas from a coal based power plant.

The large contributions of aerosols to the overall emissions measured at Maasvlakte, means that it is a challenge to develop and apply a model for the plant emissions (see the report WP 2 in the project: CCM TQP amine 6 – Validation of simulation models).

Some of the samples collected during the campaign were quantitatively analysed for nitrosamines (NA). Gas concentrations of nitrosodiethanolamine (NDELA), nitrosodimethylamine (NDMA), and morpholine (MOR) were successfully obtained by enhancing concentration and mass sensitivities using liquid-liquid extraction (LLE). Since the gas concentrations of these products were in the range of nanogram per Nm^3, they have been just scarcely detected in previous measurement campaigns on the Esbjerg pilot plant (Cesar project).

1 INTRODUCTION

1.1 Background study

The CO_2 Capture Mongstad (CCM) project is in an early planning and development phase. The project is, at the moment, organized as a joint project between Gassnova and Statoil. The purpose of the project is to plan and build a large-scale CO_2 capture plant based on amine capture technology.

An amine-based CO_2 capture plant may cause harmful emissions to the atmosphere. Amines and degradation products from reactions in the process and in the atmosphere are of particular concern for the project, but there is limited knowledge about the behaviour of these chemical compounds. Thus, several studies have been initiated by the project to increase this knowledge.

The present study concerns measurement of total emissions, testing of emission reducing equipment and planning for installation of other emission reducing technologies that could be implemented in a full scale capture plant.

1.2 Organization of the study

The work was planned to be conducted within the following 5 work-packages (WP):

- WP1: Quantifying the amine-related gas and liquid phase emissions
- WP2: Validation of simulation model
- WP3: Testing Brownian diffusion demister
- WP4: Identification of other emission reducing technologies
- WP5: HSE

A separate report from each of the WPs was supposed to be delivered. Since the Brownian demister was tested during the same campaign as covered by WP 1 it was decided to combine the corresponding WP reports. Thus the present report covers the activities and results obtained within WP 1 and 3.

The main responsible for WP 1 and 3 was Herman Kolderup (SINTEF), while Peter van Os (TNO) was responsible for the Maasvlakte campaign and the work done at TNO.

1.3 Objectives

The main objective for WP 1 was:

> *Quantifying the amine-related gas and liquid phase emission loads in treated flue gas from a reference plant*

This work consisted of the following main activities within WP 1:

1. Review and selection of instruments and methods for characterising liquid emission droplet size distribution, rate and composition. Description and planning on of a test program for droplet measurements was included.
2. Procurement and installation of new analysers and equipment for measurement of droplet size distribution, rate and composition. Alternatively rental of equipment or services.
3. Operation of the facility and development and execution of the proposed measurement program. Sampling, measurement and analysis are performed.
4. Operation of the facility and development and execution of the proposed measurement program to identify gaseous emissions from the rig. Sampling, measurement and analysis are performed.

The objective for WP 3 was:

Test a Brownian diffusion demister

This work consisted of the following main activities within WP 3:

1. A candle filter will be selected for the demister.
2. Cost for equipment, procurement and installation
3. "Operation of the rig. Sampling, measurement and analysis are performed.

1.4 Test campaign

All the tests within WP 1 and WP 3 were conducted in the same pilot plant for CO_2 capture during a period of 2 weeks. This pilot plant is owned and operated by TNO and it is located at the Maasvlakte coal power plant in Rotterdam. The coal power plant is owned and operated by EON. Prior to the campaign the pilot plant was modified to include a Brownian Demister Unit and changes were made to the water-wash system.

1.5 Structure of report

The Maasvlakte pilot plant is described Chapter 2 while the Brownian Demister Unit (BDU) is described in Chapter 3. The basis for the campaign including the Test Plan and the description of the analysis equipment is given in Chapter 4. Finally, results are presented in the form of pilot plant operation parameters, emission levels and BDU efficiency in Chapters 5 and 6. Important conclusions based on these observations are summarized in Chapter 7. Several appendices are included at the end.

2 THE MAASVLAKTE PILOT PLANT AND THE TEST SYSTEM

In the following sections the Maasvlakte pilot plant and the Test System used during the campaign is described.

2.1 The Maasvlakte pilot plant

Early in 2008 TNO installed a post-combustion CO2 capture pilot plant at the Maasvlakte coal power plant in Rotterdam. The latter plant is a 1080 MW power station, producing between 4-7 thousand ton flue gas and 400-800 ton CO2 per hour. The installed CO2 capture plant can handle 1500 m3/h of flue gas and captures maximum 250 kg/h CO2 equivalent to 1/3 MW. A picture of the pilot plant can be seen in Figure 2-1 and a simplified P & ID of the pilot plant prior to modifications can be found in Appendix 1. The following section explains the pilot plant in more details.

Figure 2-1 The TNO CO_2 capture pilot plant at Maasvlakte coal power station

2.1.1 Flue Fas Conditioning System (SO_2 washer)

Initially, the volume of flue gas to be processed can be controlled with two modulating dampers adjustable from the COMPUTER. One modulating control is mounted at the inlet to the caustic scrubber and one modulating control is mounted on the by-pass piping directly connected to the flue gas booster compressor. Each stream has a temperature and pressure compensated flow meter to allow for various flow rate selections.

The caustic scrubber includes a recirculation system with a water cooled heat exchanger to control the flue gas temperature exiting the scrubber if required. The system is controlled through a pH selector that will automatically dose caustic soda into the scrubber when required. When the flue gas is directed to the caustic scrubber, the inlet SO_2 concentration to the absorber will less than 1 ppm. However, the concentration of sulphuric acid mist is unknown.

A power plant using medium sulphur coals will typically emit 2000 ppm (molar fraction) of sulphur dioxide (SO_2) prior to flue gas desulphurization (FGD). A probable 0.5 % oxidation of SO_2 to sulphur trioxide (SO_3), will give 10 ppm of SO_3 + H_2SO_4 at the FGD inlet. However, installation of selective catalytic reduction of NO_x (SCR) is expected to triple these levels, to 30 ppm. Assuming a capture efficiency of 30 % across the FGD, and 30 % across the caustic scrubber the absorber inlet concentration of sulphuric acid mist without dilution air might be 15 ppm = 65 mg H_2SO_4/Nm^3. The gas is diluted with a factor of 3.7, which gives a possible absorber inlet concentration of 4 ppm = 18 mg H_2SO_4/Nm^3.

Next, the flue gas enters a flue gas booster compressor that provides the necessary increase in pressure required to overcome the pressure drop in the caustic scrubber, absorber tower, Brownian Demister Unit (BDU) and related piping.

2.1.2 Absorber

After processing in the flue gas conditioning system, the flue gas enters the absorber tower for CO_2 extraction. In the absorber tower, the flue gas passes through a series of four (4) packed beds designed to allow sufficient contact time for efficient absorption of the CO_2 into the absorbing solvent. Liquid distributors and redistributors are installed between each packed section to protect against possible channelling of the liquid solution. Each packed bed has a valve to allow for various bed selections (1 to 4) depending on solvent characteristics and expected absorber efficiency. The absorber sump is specifically designed with sufficient volume to allow for continuous operation without nuisance low and high limit alarm and to store approximately 60 % of the solution during shut down (together with the Stripper Sump Section). The column and packing specifications are summarized in Table 2-1.

The balance of the flue gas, primarily nitrogen, is vented to atmosphere from the top of the absorber tower through a specialized demister section designed to minimize the loss of the recirculation solvent and BDU to reduce aerosol formation. This demister is delivered by Koch-Glitsch and the type is York mesh style 172. The Process and Instrument Diagram (PID) and drawings of the demister are shown in Figure 2-2. There is an automatic CO_2 analyser with flow meter on the absorber vent line.

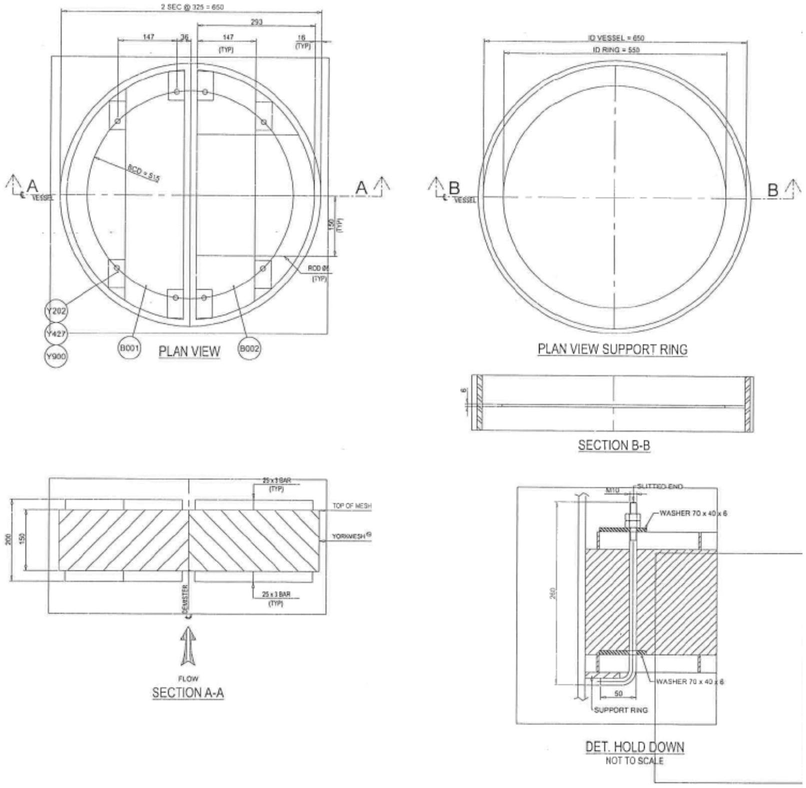


Figure 2-2 PID and drawings of the demister in the top of the absorber

Prior to the demister, the absorber vent passes through an integrally mounted water wash section where a portion of the water vapour and solvent is condensed, washed from the vent steam and returned to the system. Again, this design lowers the loss of the recirculation solvent. Based on solvent expectations, this wash section is over-designed to allow for various solvent tests. The temperature and circulation rate of the wash section is easily controlled from the PC. In 2010 an extra water circulation loop was connected to the absorber water-wash section. A water cooled plate heat exchanger is included in this loop as well as a 60 l stainless steel buffer vessel. A picture is shown in Figure 2-3 and its specifications are given in Table 2-2.

Figure 2-3 Water-wash recirculation loop

The rich solution (solution rich in carbon dioxide) from the absorber is pumped from the absorber sump to the lean/rich heat exchanger, where it is heated before entering the stripper tower. Pre-heating the rich solution before the stripper tower serves two purposes:

1. Minimizes the heat required for the stripper duty
2. Ensures maximum stripping in the top portion of the Stripper Tower

There is a common vent header with the stripper tower to allow for single connection back to stack. Both columns are skid mounted.

Table 2-1 Absorber column and packing material data

Column inside diameter (m)	Height of packing each section (m)/ number of sections	Total height (m)	Type of packing	Materials in equipment
0.65	2.1 / 4	23	Random packing IMPTP 50	SS-304 L

Table 2-2 Water-wash section and packing material data

Column inside diameter (m)	Height of packing (m)	Type of packing	Materials in equipment	Max liquid circulation rate (l/min)
0.65	2.0	Structured – Mellapak 252Y	SS-316 L	30

2.1.3 Desorber / Stripper

The lean solution (solution stripped of carbon dioxide) accumulates in the stripper sump and flows to the reboiler, where it is heated by the auxiliary steam. The solution stream generated in the reboiler is piped to the stripper sump, from which it flows upward counter-currently to the rich solution. The absorbed CO_2 is released from the rich solution and flows to the top of the stripper tower. At the top of the stripper tower there is a packed wash section and a specialized demister to minimize solvent carryover. Then the CO_2 exits the stripper tower and enters the product cooler and knock-out drum, where the solvent stream is condensed. The resultant condensate from the product cooler/knock-out drum is returned to the system, providing the condensate for the wash trays or packed section in the Stripper. The CO_2 exiting the CO_2 product cooler is combined with the absorber vent to be returned to the power plants flue gas stack. A picture of the absorber and stripper columns is shown in Figure 2-4.

Figure 2-4 Absorber and stripper columns of the TNO Maasvlakte pilot plant

Table 2-3 Stripper column and packing material data

Column inside diameter (m)	Height of packing each section (m/ number of sections)	Total height (m)	Type of packing	Materials in equipment
0.45	4.1 / 2	16.55	Random packing IMPTP 50	SS-304 L

2.1.4 Water balance

The quantity of liquid inventory (water and solvent) in the system can be increased or decreased by lowering or raising the vent gas temperature, changing the inlet temperature of the flue gas entering the absorber, changing the stripper product cooler vent temperature or changing the temperature of the lean solution entering the absorber For all the test solvents, appropriate parameters can be changed to maintain the water balance. The change in liquid inventory within the system can be adjusted by monitoring the liquid level in the stripper sump. Lost solvent is made up through the absorber sump located on the rich solution pump suction.

2.2 Modifications of the plant

As part of the project the following modifications were conducted at the Maasvlakte pilot plant during the early summer of 2011:

- Extended outlet at the top of the absorber with a 3 m section
- Replacement of random packing with structured packing in the water wash section
- Installation of a distributer plate in the absorber water wash section

In order to obtain the required flue gas condition in this project (similar to CHP conditions), an inlet pipe to the fan, which is normally not in operation was used for mixing air into the flue gas upfront the absorber. Then no modification was necessary for this special project requirement.

2.2.1 Extended outlet at the top of the absorber

The extended outlet at the top of the absorber was applied to ensure isokinetic conditions and to create a more laminar gas flow. The background for this is to have a long straight path after the water-wash and demisters to facilitate isokinetic sampling.

The extended length is 3 meters. This is about 5 times the column diameter, which is a general rule for this kind of measurements. The extended outlet was mounted on June 23, 2011 (see pictures before and after mounting in Figure 2-5 a) and b), respectively).

a) b)

Figure 2-5 Extended outlet at the top of the absorber, a) before mounting, b) after mounting

2.2.2 Replacement of the packing in the water wash section

The original stainless steel random packing (IMTP 50) was replaced by the structured packing Mellapak 252 Y.

2.2.3 Installation of a distributer plate in the absorber

Instead of only a spray ball, a liquid distributer was installed to ensure proper flow distribution into the absorber. The distributer was also mounted on June 23rd, 2011 (Figure 2-6).

Figure 2-6 New distributor plate in the bottom of the absorber, a) top view, b) bottom view

3 BROWNIAN DEMISTER UNIT (BDU)

3.1 Introduction

The Brownian Demister Unit (BDU) is intended primarily for the removal of very fine mist particles of less than 2 microns. A combination of impingement for removing greater than 1-2 microns and diffusion for finer particles where Brownian motion becomes increasingly predominant is the mechanism of operation for a BDU.

3.2 Description and working of equipment

Each filter is composed of millions of fibres, and although the efficiency of each individual fibre is low, the cumulative effect is very high. Low approach velocities are necessary in order not to mask the diffusion velocities associated with Brownian movement. Figure 3-1 shows a sketch of BDU with a candle filter. They are installed vertically and gases pass horizontally through the filter wall, the trapped particles coalesces and drains through the filter bed.

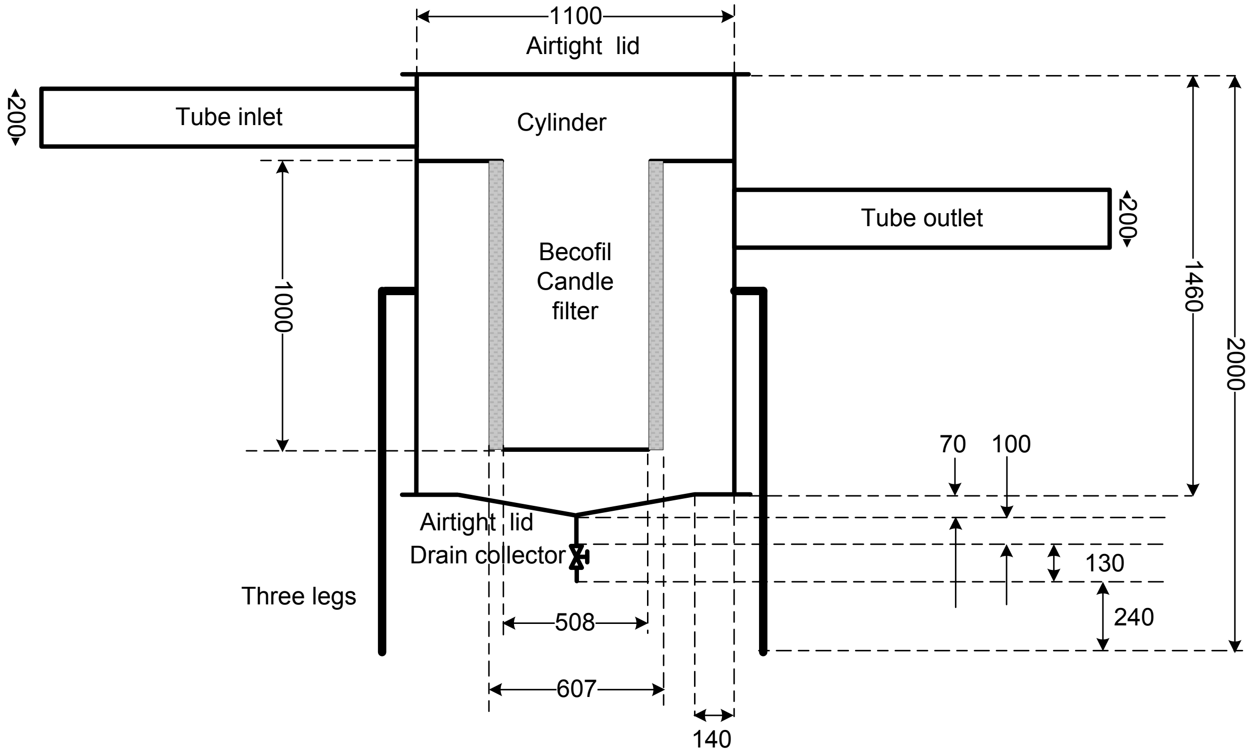


Figure 3-1 Sketch of BDU designed for installation at the Maasvlakte CO_2 capture pilot plant

Gas from demister enters top of cylinder housing and into the candle filter element. Gas outlet is from the middle part of the cylinder. The cylinder is supported with three legs. The top and bottom of the cylinder has a clamp on airtight lid, which is easy to open. This is for inspection and for washing out amine into collection drain after a test run of the filter. All liquid droplets separated in the filter need to be drained and collected for analysis of amine compounds. The upper part of the candle filter is fastened to a concentric ring (flange), which is welded to the inner wall of the cylinder below the tube inlet. This flange has 12 threaded holes matching the holes in the top flange of the filter candle element. The candle is supported with a threaded plug in the bottom plate to empty residual rinsing water used for washing out amines from the filter. The upper horizontal inlet duct and the filter compartment must be insulated in order to avoid condensation. Figure 3-2 and Figure 3-3 show some pictures of the BDU at the pilot plant.

Figure 3-2 (L) Side view showing inlet and outlet pipes, (R) Vessel for candle filter in BDU

Figure 3-3 (L) Inserting the candle filter in the filter vessel, (R) BDU at the pilot plant

Figure 3-4 shows the connection of the BDU to the absorber at the pilot plant at Maasvlakte. It shows the measurement points for process parameters such as temperature and pressure as well sampling points for gas concentration. The 150 mm ID vertical duct from the pilot absorber is not insulated. Some water and amines will therefore condense and be drained into the watertight butterfly valve. PID of the BDU can be found in Appendix 2.

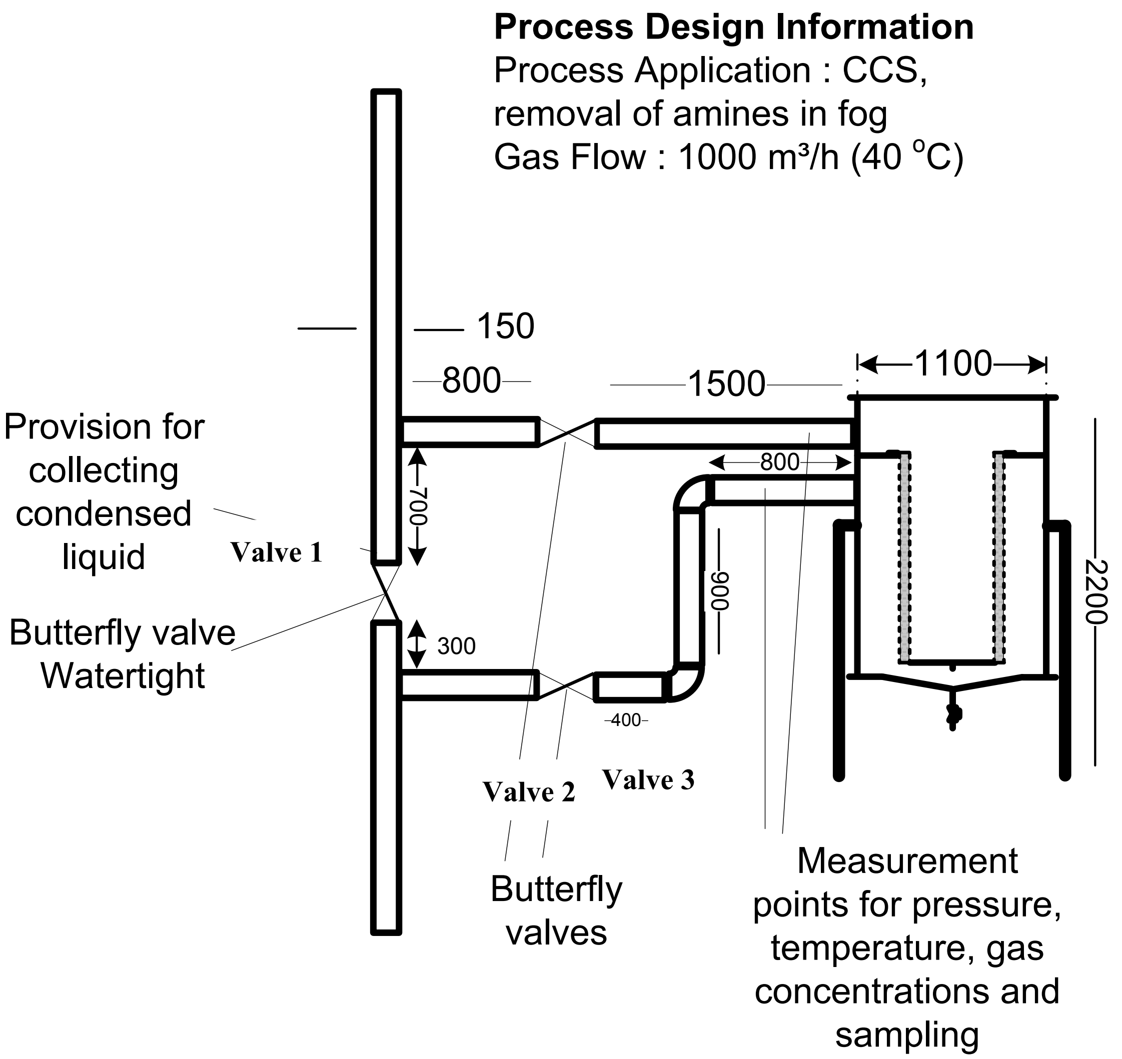


Figure 3-4 Connection of BDU to the absorber at Maasvlakte pilot

3.3 Fog Sensors

Two small optical fog sensors (OFSMk2) (see Figure 3-5) were mounted in-situ before and after the candle filter element in the filter compartment of the BDU.

Figure 3-5 Photo of the optical fog sensor OFSMk2

The measurement principle of the instrument is shown Figure 3-6. A narrow beam of red light from a laser is emitted into fog from the instrument front. A sensitive photo detector mounted in the same front measures the back scattered light in a narrow lobe that overlaps the laser beam in the sensitive zone shown in the figure. The volume of this zone, located approximately 30 cm ahead of the sensor, is less than 1 cm^3.

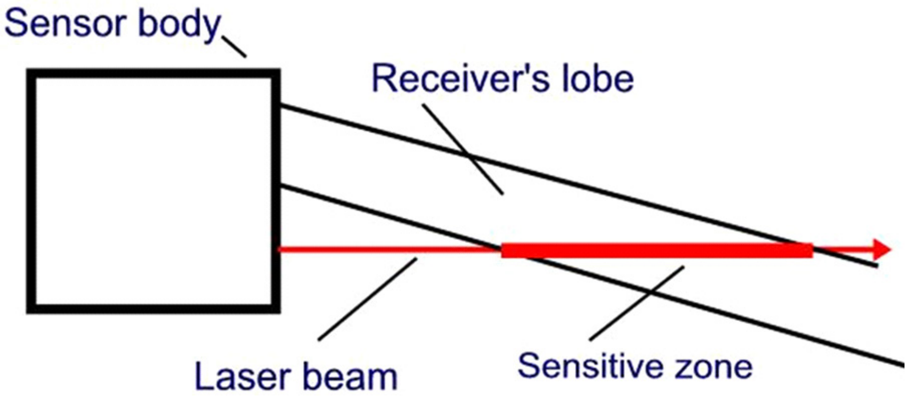


Figure 3-6 Fog sensor detecting back scattered light from a laser beam (OFSMk2)

The amount of backscattered light reaching the receiver is lower than would be obtained from forward scattering. Therefore the signal to noise ratio normally becomes lower compared to forward scattering. However, this deficiency is compensated by using high performance electro optic solutions (low noise optical receivers) making a metrological range, L_V, of 10 km possible.

The dimension of the OFSMk2 installed at Maasvlakte is 12 x 12 x 9 cm^3. Its weight is approximately 1 kg.

The standard application of this instrument is early warning of fog formation along roads, but it can also be made applicable for in-situ measurements in process equipment. A light trap was installed to prevent disturbing reflections from the hit point of the laser beam and the sensitivity was adapted to the mist concentrations in the filter.

Minimum 0 % and maximum 100 % readings for the extinction coefficients for the sensor mounted before the filter was respectively 0.006 and 3 m^{-1}. Minimum 0 % and maximum 100 % readings for the extinction coefficients for the sensor mounted after the filter was respectively 0.003 and 1.5 m^{-1}.

The fog sensors give a signal X_{in} (0 – 100 %) and X_{out} (0 -100 %) for the inlet and outlet gas respectively. It must be stressed that the sensors have different sensitivities. The light extinction coefficients, $b_{e,in}$ and $b_{e,out}$ from the two signals are calculated as follows:

Inlet gas: $b_{e,in} = (3 - 0.006) \cdot X_{in}/100 + 0{,}006$ **3.1**

Outlet gas: $b_{e,out} = (1.5 - 0.003) \cdot X_{out}/100 + 0{,}003$ **3.2**

Figure 3-7 shows the mounting and the position of the optical fog sensors 1 and 2 at each side of the filter in order to monitor light extinction by measuring backscattered light from laser beam and avoiding beam reflections.

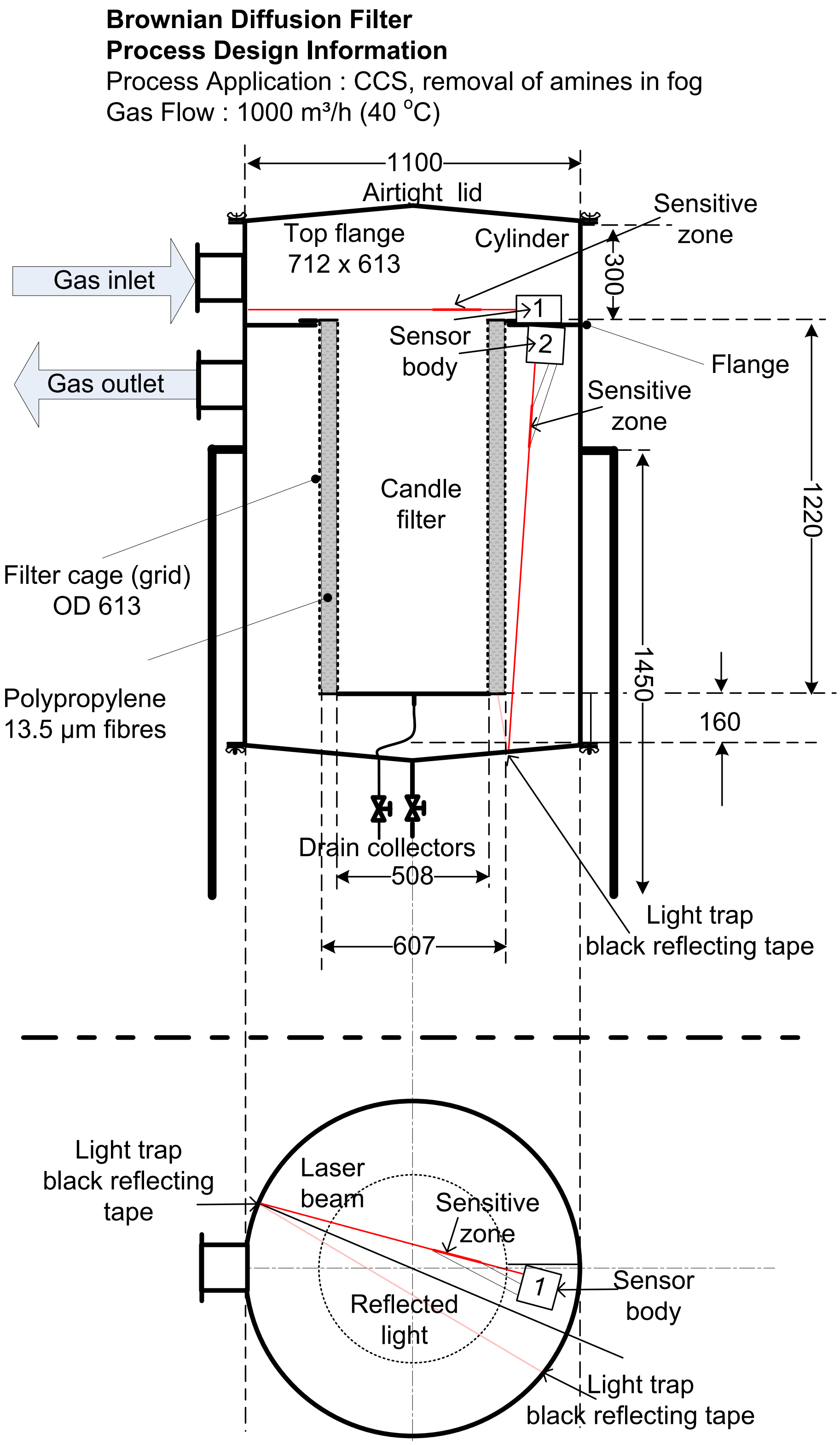


Figure 3-7 Side and Top view of optical fog sensor position in the BDU

4 BASIS FOR THE CAMPAIGN

4.1 Main basis

The present emission campaign was intended to provide emission data relevant for a full scale CO_2 capture plant at Mongstad. Some changes were made to the pilot plant and the exhaust gas composition in order to obtain results relevant for a natural gas fired power plant. The campaign was also intended to provide insight into the nature of emissions and relative importance of aerosol emissions, gas phase emissions and entrainment.

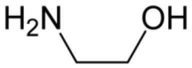
4.2 Chemistry

MEA: mono-ethanol-amine

CAS no. 141-43-5

Molecular Formula:	C_2H_7NO
Molecular Weight:	61.08

Molecular structure:



4.3 Pre-campaign operation

The plant was filled with fresh 30 wt. % MEA at the beginning of week 30 and was circulated in operation with standard operating conditions until the campaign started in week 39. The plant was shut down in week 37 and 38 during the installation period of the BDU.

4.4 Test program and operating conditions

The plan for the campaign was described in a memo, which is attached in Appendix 3. The main plan for the operating conditions were to change the solvent settings in terms of the solvent circulation flow rate (6 and 3 ton/h) and temperature of the lean solvent stream entering the absorber (35/40/46 °C) to see its effect on the aerosol formation and the efficiency of the BDU. The actual test program included main operating conditions is listed in Table 4-2. Each of the test settings were in operation for sufficient time (at least 2.5 h) to give stable operating conditions. The flow of the solvent was difficult to control as the circulation flow rate of 3 ton/h is on the lower range for the PID control of the pump.

The flue gas flow rate to the absorber over the entire period of the campaign is shown in Figure 4-1. The flow meter is calibrated at 40 °C to give results in normal m^3. The standard set point is 900 Nm^3/h. As can be seen from the graph the gas flow is not always at 900 $Nm^3/$ h especially at the last half of the campaign. This is due to the fact that pressure drop over the BDU increases over a period of time due to the accumulation of trapped particles as can be seen in Figure 5-6. Flue gas flow was reduced to 550 m^3/h on 7th October in order to investigate the effect of flow on the pressure drop across the BDU.

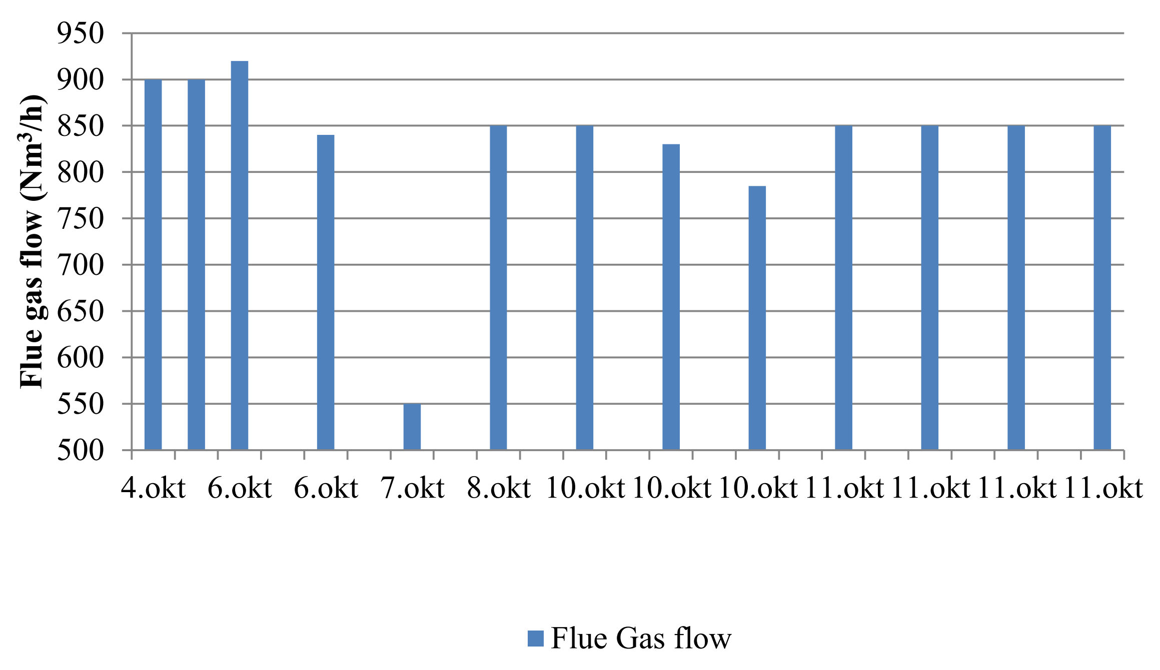


Figure 4-1 Average flue gas flow during the campaign

Table 4-1 shows the average gas volumes through the BDU during the different periods of gas sampling, liquid collection in the BDU and upstream condensate collection. These volumes are calculated from average gas flows during the different periods and their duration.

Figure 4-2 shows the various set-points for lean solvent circulation flow and absorber inlet temperature. Initially, the lean solvent circulation flow rate was maintained at 6 ton/h and later switched to 3 ton/h. The temperature was maintained at either 35, 40 or 45°C. The idea was that at higher temperature there would be higher emissions as well as higher water evaporation leading to higher aerosol formation.

Table 4-1 Average gas volumes through the BDU during the different periods of gas sampling, liquid collection in the BDU and upstream condensate collection

Date of sampling and liquid collection		2011-10-04	2011-10-05	2011-10-06	2011-10-06	2011-10-07	2011-10-08	2011-10-10	2011-10-10	2011-10-11
Test number same day		1	1	1	2	1	1	1	2	1
Flue gas flow	Nm^3/h	700,5	881,5	884,2	833,2	560,1	880,2	839,2	812,4	857,4
Sampling by impingers	Time Start	10:04	11:27	13:30	19:05	16:52	16:02	12:08	18:04	16:10
	Stop	13:30	15:12	17:10	21:15	18:46	18:32	16:05	21:05	18:10
	Hours	3,43	3,75	3,67	2,17	1,90	2,50	3,95	3,02	2,00
Gas volume	Nm^3	2405	3306	3242	1805	1064	2201	3315	2451	1715
Flue gas flow	Nm3/h	789,7	908,7	880,2	844,4	554,3	839	852	818,9	862,2
Collection by BDU by	Time Start	09:40	11:06	13:15	19:06	16:25	15:32	11:36	17:55	15:10
	Stop	16:45	17:15	17:42	21:40	19:45	19:10	16:30	21:50	18:45
	Hours	7,08	6,15	4,45	2,57	3,33	3,63	4,90	3,92	3,58
Gas volume	Nm^3	5594	5588	3917	2167	1848	3048	4175	3207	3089
Flue gas flow	Nm^3/h	691,8	908,7	879,1	840,2	555,3	868,1	838,6	812,4	864,2
Condensate collection	Time Start	09:45	11:06	13:15	19:06	17:30	15:45	12:10	18:00	15:50
	Stop	17:50	17:15	17:30	21:38	19:25	18:45	16:15	21:20	18:40
	Hours	8,08	6,15	4,25	2,53	1,92	3,00	4,08	3,33	2,83
Gas volume	Nm^3	5592	5588	3736	2129	1064	2604	3424	2708	2449
Comments	No data available before 15:00 for 5th Oct									
	Flue gas flow is 0 after 1650 for 4th Oct									
	Flue gas flow is 0 after 1842 for 11th Oct									

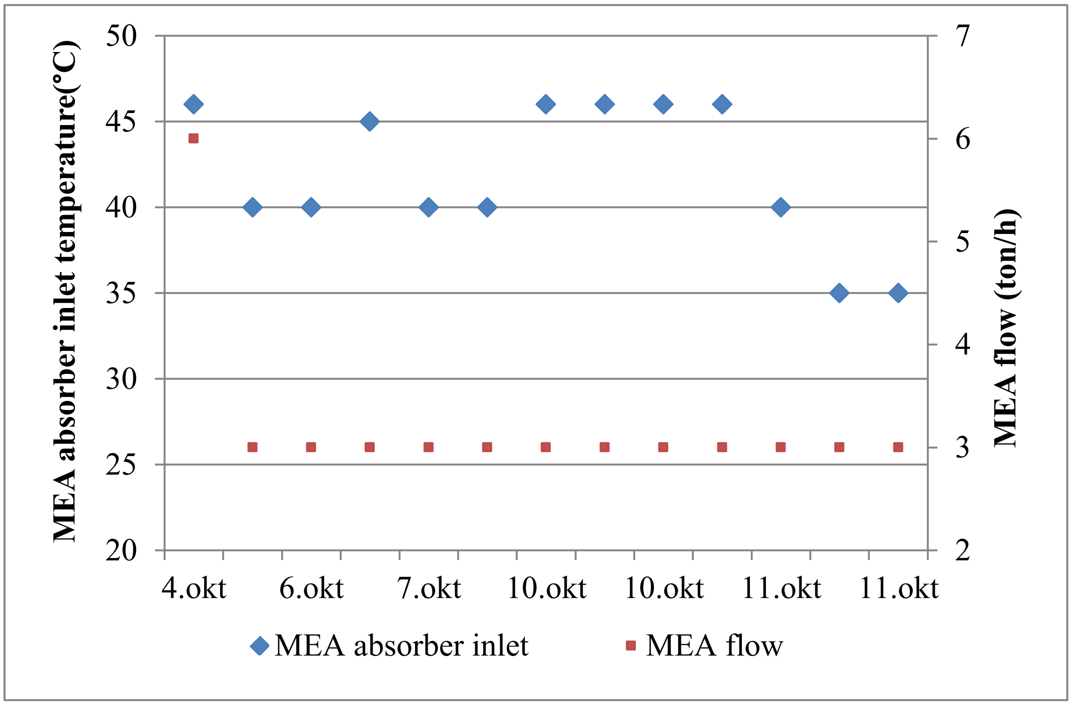


Figure 4-2 Set points for lean solvent temperature and flow (inlet absorber) during the campaign

Table 4-2 Actual test-plan and main conditions in the plant

Date	Description	Flue gas flow (Nm^3/h)	MEA circulation flow (ton/h)	MEA absorber inlet temperature (°C)	Flue gas outlet temperature (°C)	Stripper pressure (barg)	Stripper Temperature (°C)	CO_2 inlet (%)	CO_2 outlet (%)	SP-BDU Temperature (°C)
4-okt-11		900	6	46	32	1.1	114	4.4	0.22	35
5-okt-11		900	3	40	29	1.1	115	4.3	0.37	31
6-okt-11	A*	920	3	40	29	1.1	115	4.4	0.40	31
6-okt-11	B*	840	3	45	32	1.1	115	4.4	0.43	31
7-okt-11	Running low gas velocity to get optimal pressure drop in BDU	550	3	40	22	1.1	113	4.3	0.34	31
8-okt-11**	Running "standard" conditions	850	3	40	27	1.1	114	3.9	0.40	31
10-okt-11	A – Running "high mist" conditions	850	3	46	33	1.1	115	3.4	0.38	35
10-okt-11	B – Running "high mist" conditions	830/785	3	46	32/42	1.1	115	3.3	0.36	35
11-okt-11	APS + ELPI measurement at BDU	850	3	46/40/35	33/28/25	1.1	115	3.7	0.40	35/33/31
11-okt-11	Gas sampling at BDU	850	3	35	25	1.1	115	3.4	0.30	32

*A and B designates first and second test on one day. ** For 8th October the power plant was running on part load.

4.5 Flue Gas composition

The flue gas from the Maasvlakte power station is obtained from combustion of hard coal. The flue gas composition of this flue gas is given in Table 4-3. However, for the testing of the Brownian Demister Unit this flue gas was mixed with outside air to obtain a flue gas composition as given in Table 4-4 . More specifically the flue gas was mixed with air with a fixed proportion of 1:2.67, respectively. The resulting composition resembles closely the gas composition relevant for flue gas derived from a natural gas fired operated power station, such as Mongstad CHP (see Table 4-4 for comparison).

Table 4-3 Composition of the flue gas from the coal power station at Maasvlakte

Component	Composition	Unit
N_2	70.6	Vol. %
CO_2	12.6	
O_2	7.0	
H_2O	12.8	

Table 4-4 Composition of the actual flue gas to the CO_2 capture pilot plant at Maasvlakte and the specification for the Mongstad CHP

Component	Flue gas for CCM	Mongstad	Unit
N_2	76.0	79.0	Vol. %
CO_2	3.4	3.4	
O_2	17.2	13.8	
H_2O	4.0*	6.8	

*Depending on the humidity of outside air

4.6 Measurements

The following measurements were conducted during the campaign:

- Gas concentrations of MEA and degradation products by sampling through impingers containing sulfamic- or sulphuric acid
- Droplet- and amine gas concentration by collecting droplets in the BDU
- Droplet size measurements
- Light extinction by fog sensors
- Continuous gas concentrations of MEA by FTIR
- Concentrations of MEA and degradation products in rich and lean absorption liquid, in water wash liquid, in liquid collected by the BDU and in condensate from duct surface upstream BDU
- Volumes of liquid collected in BDU and of condensate from duct surface
- The use of lithium (Li)- an rubidium (Rb) carbonate as tracer for quantification of drop entrainment from the absorber and water wash liquids
- Registration of temperatures, pressures and gas- and liquid flows in the carbon capture pilot plant

4.7 Online measurements

4.7.1 Overview

Several process parameters such as temperature, pressure, flow and CO_2 content have been measured online at various locations across the pilot plant. The measurements points are shown in the PID in Appendix 4. The description of the corresponding tags in the PID can be found in Appendix 5. A flow sheet with the measurement points can also be found in Appendix 6. An FTIR measures the flue gas composition entering and leaving the BDU. More information about the FTIR can be found in the following section.

4.7.2 FTIR instrument

A 0.95 cm diameter stainless steel tube is inserted in the 12.7 cm diameter flue gas stream inlet and outlet to the BDU. This tube is heated up to a temperature of 120°C using an electric heat tracer. The tube leads to a heated ceramic filter which is kept at a temperature of 180°C (see Figure 4-3). From this filter, a 5 m heated sampling line (180°C) leads to a second heated filter and a heated pump (all at 180°C), which pumps the flue gas sample in the FTIR analyser. The analyser is also kept at 180°C (see Figure 4-4). This implies that the flue gas is kept above dew point temperature when analysed thus preventing condensation of water and loss of water soluble components.

The FTIR was specifically calibrated for the following components: water, CO_2, CO, N_2O, NO, NO_2, SO_2, Methane, Acetaldehyde, Formaldehyde, Mono Ethanol Amine (MEA), NH_3, Diglycolamine (DGA), and Diethanolamine (DEA). Formic acid and acetic acid are also monitored by the FTIR, but the FTIR system was not calibrated for these components. Instead, publically available spectra were used to analyse these components, which undoubtedly increased the detection limits and uncertainties. Oxygen is also measured using a zirconium cell. The FTIR analyser is controlled using an on-site laptop which also records the data. A GPRS connection with this laptop allows remote access.

Figure 4-3 Installation of the sampling tube and heated filter (in blue)

Figure 4-4 FTIR analyser and the controlling and recording laptop

4.8 Sampling

4.8.1 Gas sampling

The gas sampling trains consisted of two impingers filled with 50- and 100 ml 0.1 N acid solution, respectively and an empty bottle at the end for drop of entrainment collection.

The gas sampling points were:

1. Between absorber (A) and water wash (WW) unit
2. Between WW and demister
3. Above demister (highest point)
4. Inlet BDU
5. Outlet BDU

A sketch of the locations is given in Figure 4-5. For more details, see Appendix 4.

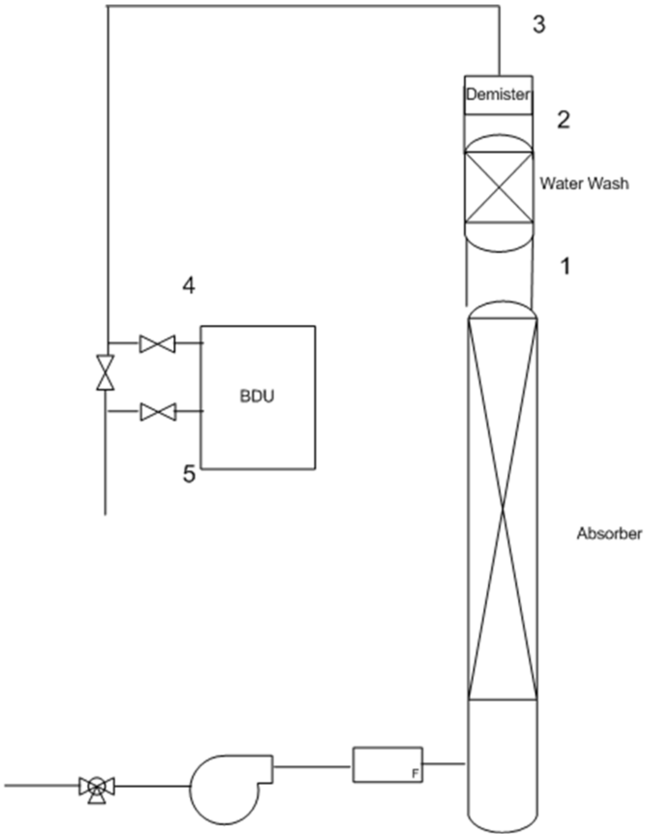


Figure 4-5 Simplified sketch of the sampling points

4.8.2 Aerosol measurements

Part of the emission of the CO_2 removal plant is in the form of small water droplets containing MEA and other non-volatile components. This mist or aerosol is characterized by its concentration and particle size distribution. The concentration is generally reported as the number of particles per cm^3 (i.e. per ml of air) or as mg per m^3. This concentration is dominated by the large number of submicron particles present. The mass concentration is dominated by the larger particles because mass increases with the particle diameter cubed. The focus of the aerosol measurements during this campaign was on the Brownian Demister Unit, BDU. The inlet and outlet concentrations were measured

For the aerosol measurements three instruments were used:

1. The two fog sensors installed before and after the BDU (see Section 3.3)
2. An aerodynamic particle sizer or APS (TSI 3321) for measuring the particle size dependent concentration in samples
3. An electrical low pressure impactor ELPI (Dekati) for measuring the particle size dependent concentration in samples

The two last instruments are further described in the following sections.

4.8.3 Instrument description

2. APS

An APS takes a sample of one litre air per minute. The air with the aerosol particles is led through a nozzle to a laser beam where the light scattered by the particles is detected. This is depicted in Figure 4-6. The intensity of the laser beam is crested resulting in a double peaked signal. The time between the two peaks is a measure for the particle velocity.

At the nozzle exit the particles have a velocity v_1. Through an outer nozzle air without particles is pumped at a rate of 4 l/min resulting in air velocity v_2. Small particles adapt quickly to the new velocity. Large particles have barely changed their velocity by the time they pass through the laser beam. Based on the time of flight between the peaks in the laser particles between 0.5 and 20 µm can be sized in 50 size classes.

Particles down to 0.3 µm can be detected but not sized. If two or more particles are present in the measurement section at the same time the particles are also detected but not sized. All such measurements are accumulated and represented in the first channel < 0.523 µm. For practical purposes (linearity in Excel) this channel is represented as 0.3 µm.

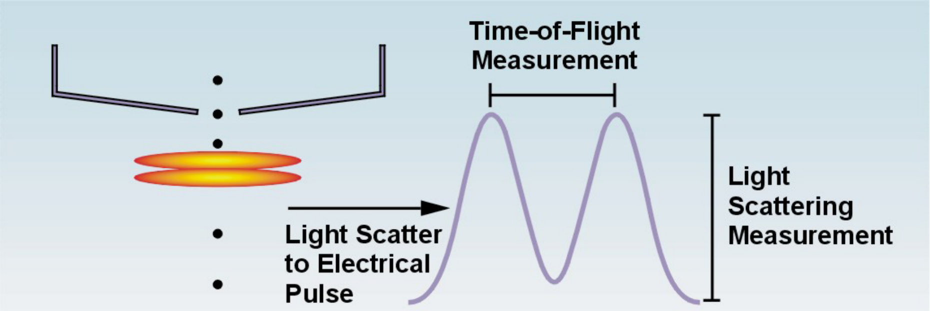


Figure 4-6: APS measurement principle, illustrated

3. ELPI

Multi stage or cascade impactors can be used to collect aerosol particles in the different size classes as illustrated in Figure 4-7. Air is pumped through a small nozzle or set of nozzles with a certain flow rate. Below the air entrance is a collection plate. The air has to make a sharp turn around this collection plate and due to their inertia larger (heavier) particles cannot follow the air flow and impact on the plate. Smaller particles remain entrained. Next the air is led through subsequently smaller nozzles, increasing the air velocity and causing increasingly smaller particles to impact and be collected on the collection plates. The cut-off size is not absolute but has an S-curve. Each stage is characterized by particle size (D50) and the inner diameter or characteristic size Di. D50 is the particle size of which 50% is collected and 50% moves on to the next stage. These sizes are valid for a particle density of 1 g/cm^3, which in the case of water droplets is valid and no corrections are required.

A low pressure is needed to collect particles with an aerodynamic diameter smaller than 0.5 µm. The ELPI consists of 12 stages and pressure at the smallest ELPI stage is 100 mbar. The flow through the ELPI is 10 l/min and a sample measurement can be taken each second. The pressure reductions through the ELP stages will cause droplets to evaporate resulting in too high measured concentrations of the smallest size fractions.

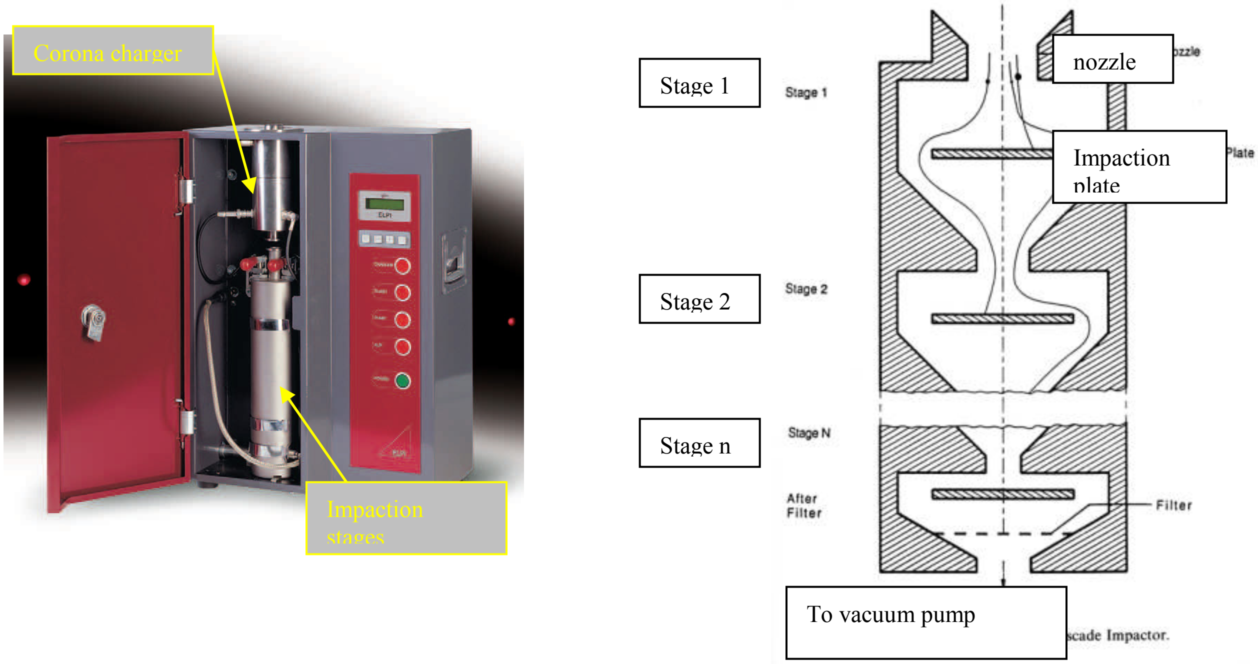


Figure 4-7: ELPI picture on the left, on the right a schematic diagram of the working principles of an impactor

The ELPI is able to count the trapped particles in real time. At the entrance a corona charger charges the particles and when the particles hit the metal collection plates this charge is transferred as a current. The smallest particles the ELPI can measure have a diameter of 30 nm which result in a current of the order of 1 femto ampere.

4.8.4 Sampling strategy

The aerosol in the ducts from the CO_2 plant consists of water droplets with some non-volatile components. Water is a fast evaporating liquid. Typically a 1 µm water droplet evaporates in less than a second, even at high relative humidity (see Figure 4-8). On the other hand, when the temperature of the aerosol sample decreases the air can contain less water and droplets may grow due to condensation. Precautions have to be taken to keep the aerosol sample measured representative of the mist in the stack. Condensation inside instrument's nozzles or pinholes may cause additional problems.

In order to retain the particle size distribution of the sample as it leaves the ducts, the sampling lines and measurement apparatus were kept at an optimal temperature equal to or slightly (1-2 °C) above the temperature in the ducts to prevent condensation and minimize evaporation. Also the sampling lines are kept as short as possible without sharp turns.

In Figure 4-9 schematic of the sampling and measurement set up with the APS is shown. A temperature controller and hot air supplied by a hair dryer is used to keep the optimal temperature in the box with the measurement equipment.

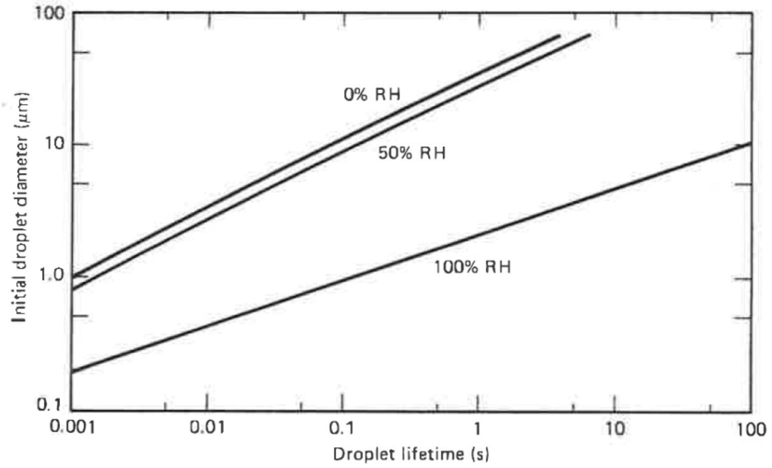


Figure 4-8: Water droplet lifetimes as a function of droplet size for 0, 50 and 100% relative humidity at 20°C (from W.C. Hinds, Aerosol Technology: properties, behaviour and measurement of airborne particles, chapter 13)

Ideally, sampling from a flow should be performed iso-axially and iso-kinetically, so the sampling flow has the same direction and velocity as the main flow. To prevent losses due to particles colliding with the walls of the sampling tube the bends should be smooth and gradual. The entrance of the sampling line must be thin walled. Due to practical constraints, sampling was not performed iso-kinetically, but this should have little influence for the particle sizes and velocities encountered around the BDU as can be seen from calculation.

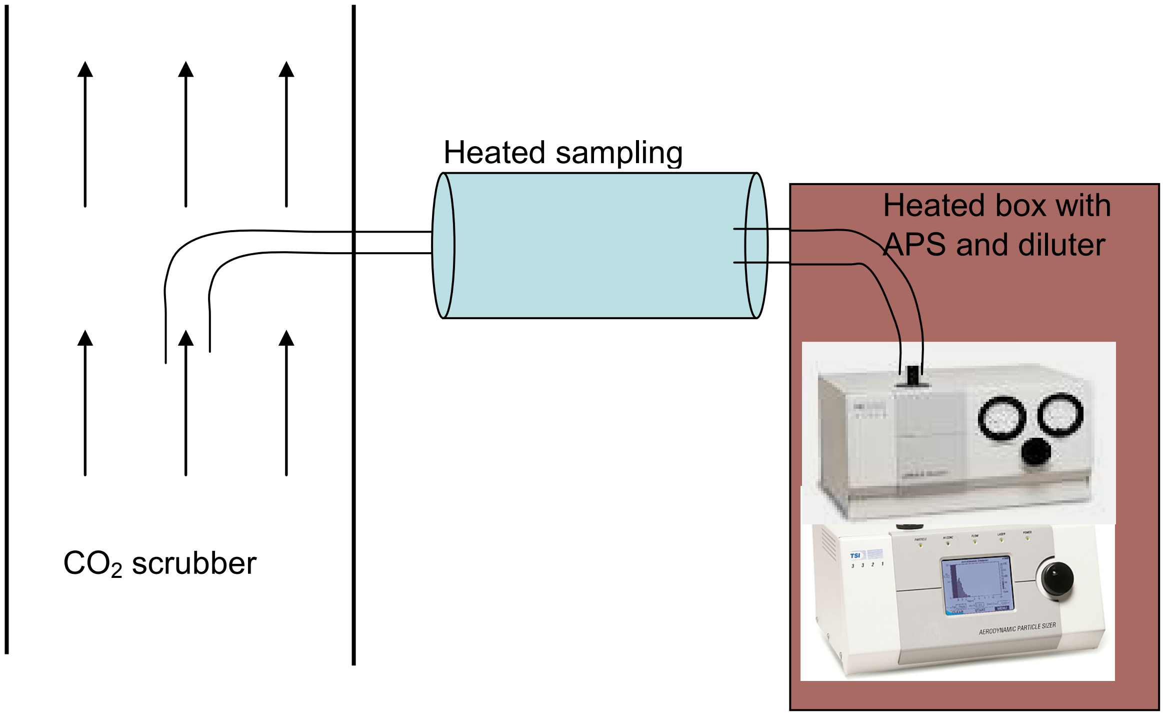


Figure 4-9: Schematic of the sampling and measurement set up with the APS

The diameter of the tube, from which the BDU inlet and outlet samples are taken, is 12.7 cm (5 inch). The flow through the Brownian demister, hence through the tube is 800-850 m^3/hr. Thus velocity in the pipe is 17.5-18.6 m/s.

Sampling was performed with a 3/8 inch sampling tube with an inner diameter of 7 mm. With a flow of 26 l/min (sampling plus excess flow) through the probe the velocity in the sampling tube is 11.3 m/s. Before the BDU the sampling flow is approximately 56 l/min. This results in a flow velocity of 24 m/s.

The effect of an-isokinetic sampling is based on the Stokes number. This is the ratio of the stopping distance S of a particle to a characteristic dimension of the obstacle, in this case the tube diameter.

The Stokes number for 10 µm particles in an air flow through a 12.7 cm cylindrical tube at 18 m/s is about 0.03. The Stokes number in the probe is 0.6. For 1 µm particles the Stokes number in the sampling tube is 0.06.

A factor 2 between the main stream velocity and the velocity in the sampling tube for particles with a Stokes number of less than 1 results in inlet and transmission efficiencies of 80%-120% (In the plant U_0/U is about 1.65. The probe is in the direction of the flow, so the aspiration angle is 0°. Inlet efficiency more than 1 (100%) means oversampling (see Figure 4-10).

Also, with a Reynolds number of approximately 10^5 in the inlet and outlet tube of the BDU, turbulence is so high that this should influence the sampling efficiency more than an-isokinetic sampling.

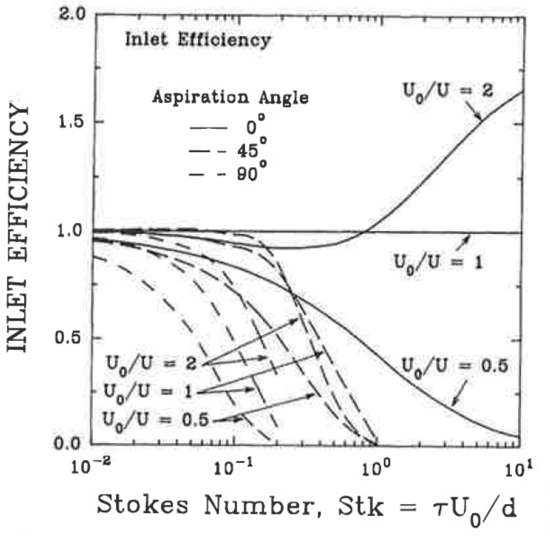
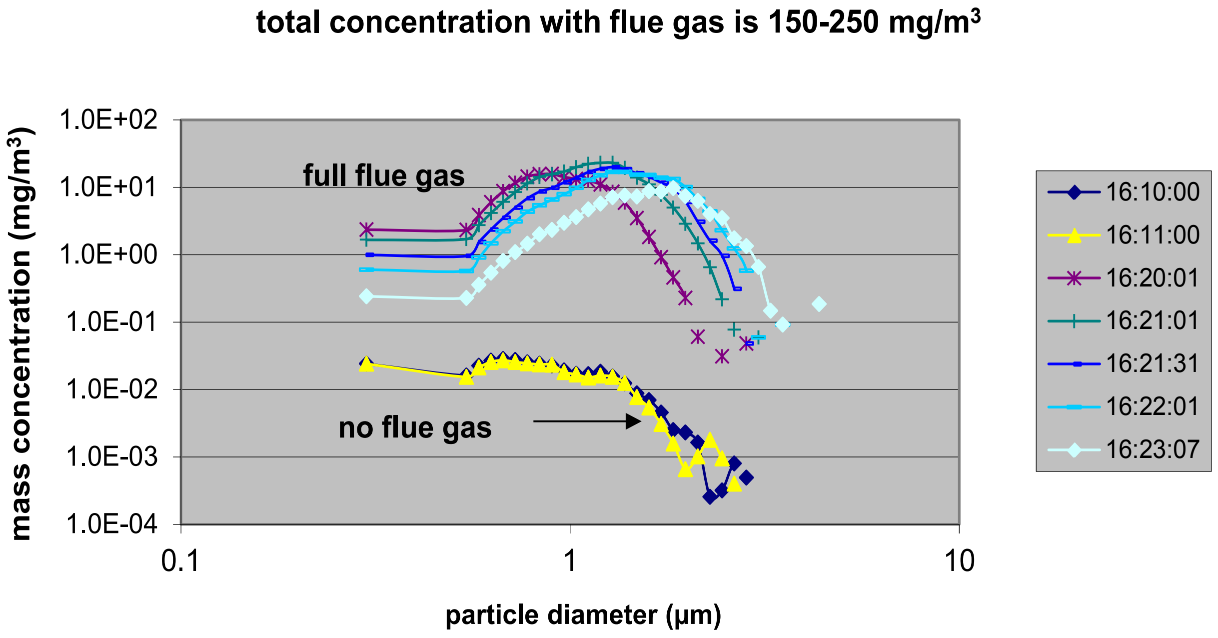


Figure 4-10: Inlet efficiency of a probe as a function of St and the ratio of the velocity in the pipe, U_0, and the velocity in the probe U (***source: Brockman in Aerosol Measurement: principles, techniques, and applications, Chapter 6***)

4.8.5 Testing of the APS and ELPI

Preliminary testing in a lab scale set-up of the washing tower showed low concentrations of particles that could well be measured with an APS without diluter and with an ELPI. The first tests in the pilot plant in the riser after the washer showed very high mist concentrations. The APS had to be used in combination with two diluters (1:100 and 1:20) giving a combined dilution ratio of 1:2000. This leads to loss of especially the larger droplets and thus significant underestimation of the total mass concentration. However, Figure 4-11 clearly shows that the aerosol concentrations in the pilot plant are very high and increase by orders of magnitude when flue gas is added to the mixture. The flue gas contains a lot of particles and components that can act as nuclei for the formation of droplets.



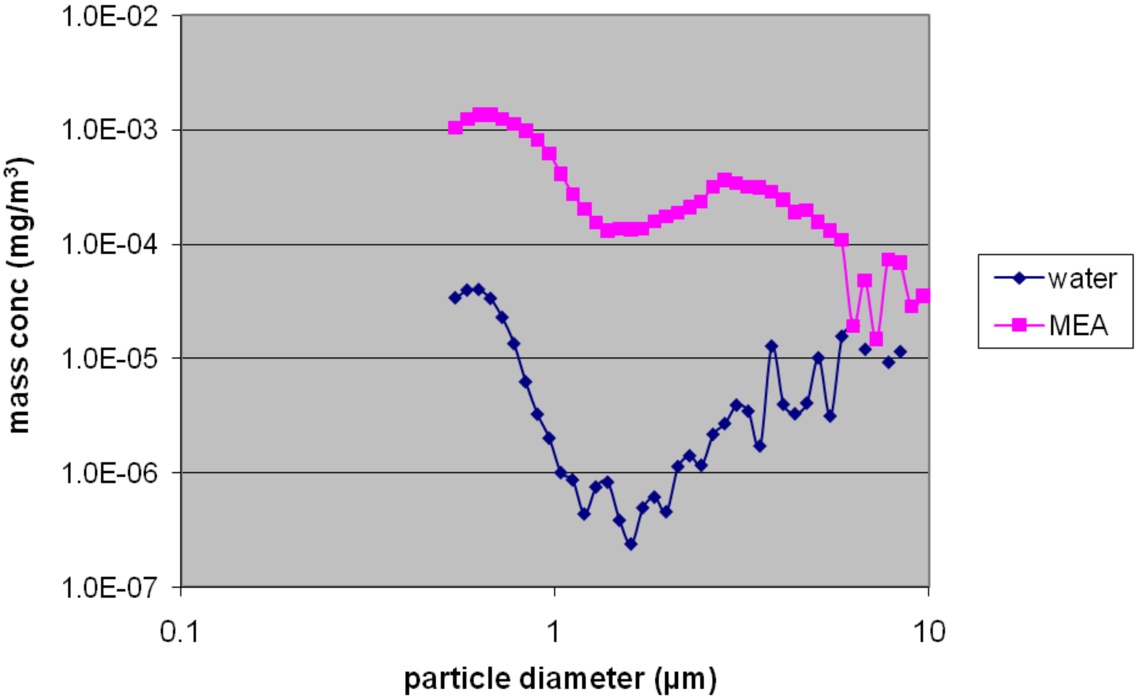


Figure 4-11: The top graph: aerosol concentration with and without flue gas after the washer at the CATO pilot plant, the Bottom graph: the concentration in the laboratory set-up with and without MEA in the liquid

The concentration was so high that the ELPI had to be protected against the moisture by using a pre-impactor plate that removes particles larger than 15 µm. The maximum size measured by the ELPI is 10 µm (D50) this should not influence on the measurement. Several grams of water were collected on the pre-impactor plate within 5 minutes. The highest stages on the ELPI also give us an idea of the losses in the APS due to dilution and less sensitivity to larger liquid particles.

Background concentration (outside air) and mist concentration with full flue gas is shown in Figure 4-12. The total concentration measured by the ELPI is 3 g/m3. Larger particles taken out by the pre-impactor constitute another estimated amount of 10 g/m^3. Even with the pre-impactor the maximum concentration on one stage (nr 3) was exceeded.

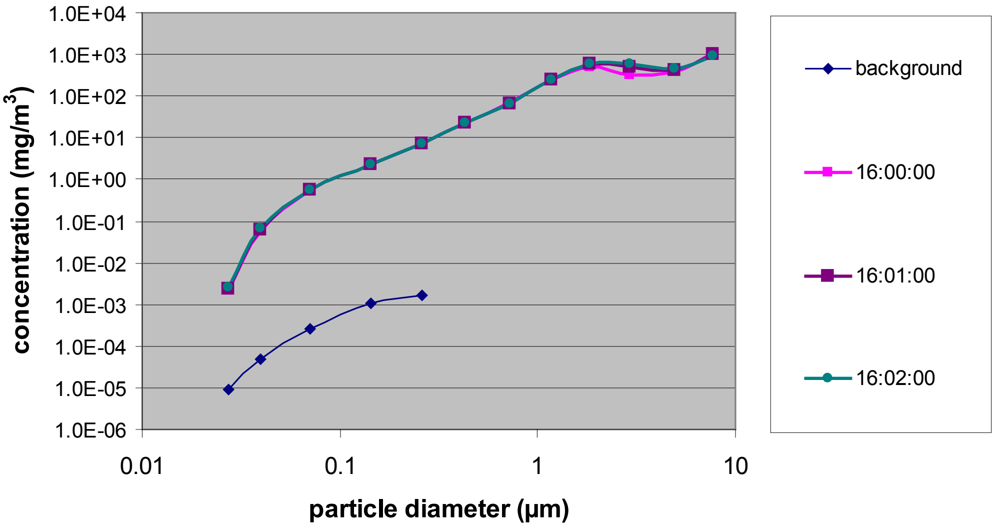


Figure 4-12 **Background concentration (outside air) and mist concentration with full flue gas.**

In Figure 4-13 the effect of flue gas on aerosol concentration measured with the ELPI is shown. Total concentration rises from ca. 10 mg/m^3 to over 200 mg/m^3.

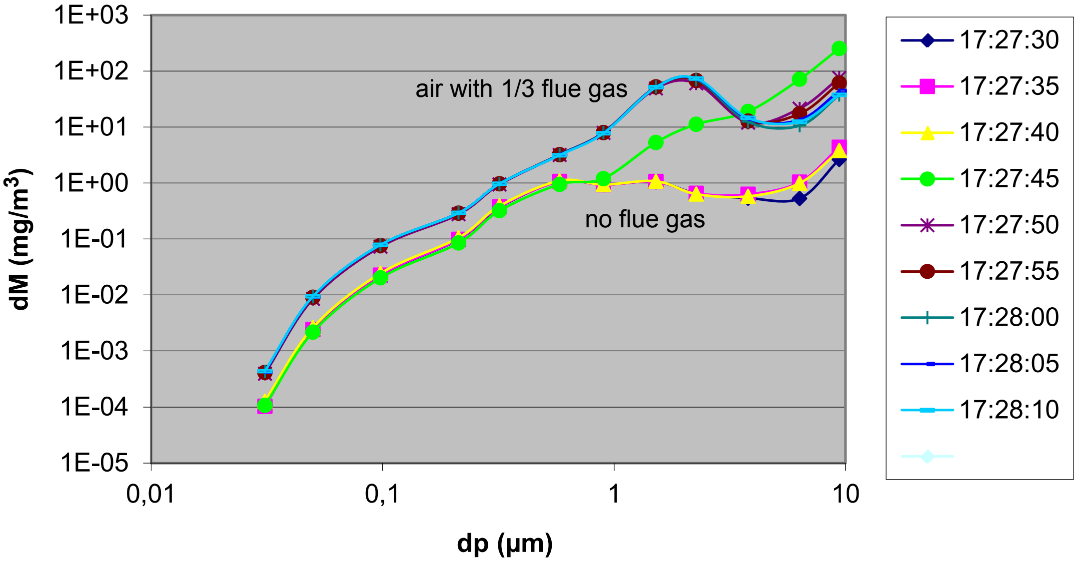


Figure 4-13: The effect of flue gas on aerosol concentration measured with the ELPI

4.9 Testing of BDU

The operation of the BDU is explained in brief here. At the start of each day of the experiment, stable operation of the pilot plant is achieved with mixed flue gas. Subsequently, inlet and outlet valves of the BDU are opened while slowly closing the bypass valve (to BDU). The BDU induces a pressure drop and thus flue gas flow is corrected for by adjusting the flue gas to the absorber valve. Gas sampling across the different locations in the pilot plant is only started after stable operation over the BDU is reached (at least 30 min). The condensate from the down comer pipe wall is then collected. Gas sampling measurements are done for at least 2.5 h during which the operation conditions are maintained. At the end of the experiment the bypass valve to BDU is opened while closing the inlet and outlet BDU valves. Finally, the condensate from the down comer and the condensate in the BDU (BDU catch) are collected.

4.10 Analysis of samples

4.10.1 Mass spectrometric analysis

The components (MEA, NDMA, NMOR, NDELA and MEA-nitramine) were analysed in samples (wash water, solvent, condensate, BDU catch, sulfamic acid and sulphuric acid) with tandem liquid chromatography triple quadruple mass spectrometry (LC-MS-MS-QQQ) technique on an Agilent 1290/6460 instrument (Agilent Technologies, Palo Alto, CA, USA). The samples were prepared by dilution, direct injection or sample preparation by liquid-liquid or solid phase extraction. The limits of quantitation were 0.6 µg/L for MEA and 0.1 µg/L for NDMA, NMOR, NDELA and MEA-nitramine in solution.

4.10.2 ICP-MS analysis

Sample handling

A simple dilution of 1+99 was chosen as sample preparation to reduce matrix effects, maintain detection power and reduce risk of severe memory effects. To be able to detect the elements in the samples with the lowest concentrations, no dilutions were done. Quality controls were prepared to monitor method performance by diluting a reference solution.

Instrumentation

Determinations of lithium (Li), rubidium (Rb), sodium (Na), sulphur (S) and potassium (K) in the samples were carried out with a high resolution ICP-SFMS (Element 2, Thermo Scientific, Bremen, Germany). This instrument consists of both a magnetic sector and electric sector field and has three modes of predefined resolutions. The monitoring of Li, Rb, Na, S and K were performed in medium resolution (4000 m/$\boxed{m}$ at 10% valley definition). Internal standardisation was used to compensate for analytical issues like drift and matrix effects. Indium was chosen as a suitable internal standard, and applied at a final solution of 1.000 ng/L to all samples and calibrators.

Method performance

The limit of detection and quantification for this method are presented in Table 4-5. Detection limit is based on 3 times the standard deviation of a series of blanks. Quantification limit is based on 10 times the same standard. The calibration ranges and intra- sequential precisions (for one level) are also reported. For Rb a few results are outside the calibration range. But the linear range is proven to be broader.

Table 4-5Limit of detection and quantification

Element	Limit of detection	Limit of quantification	Calibration range	Intra- sequential precisions (%RSD)
Li	0,01 µg/L	0,03 µg/L	0,03-1000	4
Rb	0,075 µg/L	0,25 µg/L	0,25-4500	12
Na	60 µg/L	200 µg/L	200-100000	5
S	60 µg/L	200 µg/L	200-100000	4
K	6 µg/L	20 µg/L	20-100000	4

4.11 Assessment of the data from Maasvlakte

The emission data can in general be assessed based on internal consistency and by comparison with other relevant emission data. In addition we can look at the uncertainty in reported experimental data.

In general it must be noted that there is little emission data available for direct comparison.

Besides the very efficient BDU it is noticed that the water wash is not capturing much of the MEA. However, the results from the campaign must be evaluated by taking into account the specific conditions and circumstances at the pilot plant in Maasvlakte.

The pilot plant is treating a flue gas from a coal fired power plant. Compared to a standard gas fired flue gas this gas has much higher concentrations of CO2 and dust particles. During the campaign the CO2 level was adjusted for by mixing with surrounding air. However, the temperature and humidity of the outside air was low during the campaign, and since the amount of air to flue gas was about 2:1, also the gas entering the absorber column was very low in temperature (below 30°C) and thus the water content (it is assumed that the gas absorber inlet stream is saturated at this low temperature). For the capture rate low temperature condition in the bottom of the absorber is advantageous, but it makes it difficult to keep the water balance in the pilot. Since the absorber outlet temperature was around 40°C and there was no make-up water supply to the water wash, there was a net loss of water from the plant during the campaign.

The circulation rate of the water in the water wash section was only 3.6m3/m2h. It is SINTEFs view that it should have been around 7-10 m3/m2h in order to wet the packing material sufficiently. However, this cannot explain the total low efficiency of the water wash. Much more important is the high degree of mist particles.

High particle concentration and presence of sulphur-acid will increase the amount of kernels for water condensation and mist generation. Condensation will happen in zones in the absorber where the gas is cooled down like in the upper part of the absorber where the lean liquid enters the absorber and in the water wash section. Also in the bottom of the absorber, where the unsaturated cold flue gas enters the column could possibly provide some special conditions for mist generation.

For modelling of emission in the water wash it is important with accurate data from on-line sensors in the process. Unfortunately, some of the sensors (e.g. the gas flow measurements out of the absorber and inlet to the pre-scrubber) were not working properly during the campaign and back-calculations based on other sensors were needed, which also implied additional assumptions and thus higher uncertainties. This is explained more in detail in Mejdell et al., 2012.

5 MAIN RESULTS FROM THE EMISSION MEASUREMENTS

5.1 Gas sampling, analysis and fog sensor results

Table 5-1shows the concentrations of total MEA and liquid aerosols in the gas flow at different sampling points (see simplified sketch in Figure 4-5). It is assumed that amine containing liquid collected in the BDU during each test approximately equals the amount of liquid drained from the BDU after each test. This collected liquid or BDU catch is divided by the gas volume through the BDU filter unit to give average liquid gas concentrations during each test in in unit g/Nm^3. In the same way the amount of MEA in the BDU catch is divided by the gas volume through the BDU filter to give average MEA concentrations during each test in unit mg/Nm^3 as MEA BDU catch.

In average, concentrations of total MEA is reduced from 370 to 150 mg/Nm^3 dry gas through the water wash and demister unit. A concentration of 14 mg/Nm^3 is condensed at the inner wall of the duct leading to the BDU and a concentration of 137 mg/Nm^3 is collected in the BDU. The average MEA concentration out of BDU is just 2 mg/Nm^3. The aerosol concentration based on the collected liquid in the BDU (BDU catch) was in average 2.9 g or ml/Nm3 wet gas. If the inlet gas to the BDU is supersaturated an unknown amount of water vapour will condense on the tiny fibres in the BDU. The calculated concentration of liquid aerosol at the inlet will then be too high.

Table 5-2 Shows concentrations of entrained droplets in the gas flow at different sampling points calculated from tracer experiments. These concentrations are very small compared to fog concentrations calculated from the BDU catch.

Table 5-3 shows the average value of the signals from the two fog sensors during the periods for aerosol collection in the BDU and the belonging light extinction coefficients, $\boldsymbol{b_{e,in}}$ and $\boldsymbol{b_{e,out}}$, calculated from equation 3.1 and 3.2. The extinction coefficients depends on the droplet surface concentration, which can be calculated from the droplet volume fraction, $\boldsymbol{y}$, and the Sauter or volume to surface mean diameter of the droplets, $\boldsymbol{D_{3,2}}$. The relation between $\boldsymbol{D_{3,2}}$, $\boldsymbol{y}$, and $\boldsymbol{b_e}$ is given by equation 5.1.

$$D_{3,2} = \frac{\sum n_i D_i^3}{\sum n_i D_i^2}$$

Where $\boldsymbol{n_i}$ = number of droplets with diameter, $\boldsymbol{D_i}$.

$$\boldsymbol{D_{3,2} = \frac{3y}{b_e}} \tag{5.1}$$

The volume fraction of droplets, $\boldsymbol{y_{in}}$, measured in the BDU catch can be used to calculate inlet droplet diameter, $\boldsymbol{D_{3,2,in}}$. The volume fraction of droplets, $\boldsymbol{y_{in}}$, measured in the BDU catch and the inlet, $\boldsymbol{C_{MEA,\ in}}$, and outlet, $\boldsymbol{C_{MEA,\ out}}$, MEA concentrations can be used to calculate outlet volume fraction, $\boldsymbol{y_{out}}$, and outlet droplet diameter, $\boldsymbol{D_{3,2,out}}$.

$$D_{3,2,in} = \frac{3y_{in}}{b_{e,in}} \quad , \qquad D_{3,2,out} = \frac{3y_{out}}{b_{e,out}}$$

y_{out} can be estimated based on different assumptions. If we assume that that the gas concentration of MEA in aerosols is much larger than of vaporous MEA, equation 5.2 can be used.

$$y_{out} = y_{in} \frac{c_{MEA,out}}{c_{MEA,in}} \tag{5.2}$$

An alternative approach is to predict a reasonable penetration of the inlet aerosol through the BDU. For example 0.7 % and calculate y_{out} from equation 5.3

$$y_{out} = y_{catch} \frac{0.007}{1-0.007} \tag{5.3}$$

Table 5-3 shows the Sauter mean droplet diameter ($D_{3,2}$) estimates before and after the BDU for the two alternative assumptions: **a** based on equation 5.1 and 5.2 assuming all of the penetrated MEA through the BDU is in liquid phase as aerosol droplets and **b** based on equation 5.1 and 5.3 assuming 0.7 % penetration of inlet aerosol droplets.

For inlet gas the results of these calculations show a variation of $D_{3,2,\ in}$ in the range 0.8 – 8 µm and an average value of 4.4 µm.

The resulting $D_{3,2,\ out}$ for assumption **a** shows range and average value of 0.2 – 1.8 µm and 0.64 µm respectively, while $D_{3,2,\ out}$ for assumption **b** shows range and average value of 0.09 – 0.57 µm and 0.32 µm respectively. Droplet diameters around 0.3 µm are more likely to penetrate the BDU because neither Brownian motion nor inertial forces are effective separation mechanisms in this size range. Assumption **b** might therefore be the one most appropriate.

A third alternative **c** is to calculate the MEA gas concentration which is in equilibrium with the liquid MEA concentration in the BDU catch. Droplet concentration can then be calculated by deducting the equilibrium MEA concentration from total outlet MEA concentration. The result of this calculation is shown in the last four rows of the table.

If inlet gas to the BDU contains supersaturated vapour that condenses in the BDU, this will result in a too large $D_{3,2}$ estimate at the inlet. On the other hand if not all of the collected liquid in the BDU is drained and measured, this will result in a too low $D_{3,2}$ estimate at the inlet.

The fog sensors did successfully register in-situ extinction coefficients continuously during the measurement campaign. The extinction coefficients are useful measurements because their values are proportional with the surface concentration of aerosols in gas flow and tell if there is a high or low content of aerosols in the gas. Extinction coefficients can be used to calculate surface to volume diameter, $D_{3,2}$, of the aerosols. However, because the droplet size distribution including $D_{3,2}$ of the aerosol varies it is difficult to correlate the extinction coefficients quantitatively with mass concentrations of aerosols and MEA.

Table 5-1: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Concentrations of total MEA and liquid aerosols in the gas flow at different sampling points

M = Mole weight MEA	61.08	kg/kmol	ppm = (mg/Nm^3)□V_{NM}/M = (mg/Nm^3(dry)) X(dry)□V_{NM}/M		
V_{NM} = Mole volume	22.414	Nm^3/kmol	X(dry) = Dry gas volume fraction	Mole weight of water 18	
For saturated air at, 30 ° C, X(dry) = 0.956				Water cont. 44000 ppm	35,3 g/Nm3

Time of gas sampling by impingers	Date	**2011-10-04**	**2011-10-05**	**2011-10-06**	**2011-10-06**	**2011-10-07**	**2011-10-08**	**2011-10-10**	**2011-10-10**	**2011-10-11**	
	Time Start	10:04	11:27	13:30	19:05	16:52	16:02	12:08	18:04	16:10	
	Stop	13:30	15:12	17:10	21:15	18:46	18:32	16:05	21:05	18:10	
Test number same day		1	1	1	2	1	1	1	2	1	
Average flue gas flow during impinger sampling	Nm^3/h	900	900	920	840	550	850	850	807.5	850	
Temperatures °C	Flue gas outlet	32	29	29	32	22	27	33	37	25	
	BDU gas	**25,5**	**27,9**	**27,7**	**31,2**	**21,3**	**25,9**	**33,9**	**34,9**	**23,2**	**Average**
MEA concentrations mg/Nm^3 dry gas	Before WW			**407**	**466**	**213**	**337**	**385**	**460**	**336**	**372**
	After WW									**206**	
	After demister			**241**	**252**	**111**				**272**	**219**
	In BDU	**87**	**129**	**157**	**173**	**86**	**159**	**146**	**177**	**256**	**152**
	Out BDU	**1.18**	**0.97**	**1.15**	**2.10**	**4.37**	**1.27**	**1.89**	**3.95**		**2**
	Condensate collection	**8,4**	**16,6**	**23,9**	**20,9**	**7,9**	**12,7**	**11,3**	**14,8**	**8,8**	**14**
	BDU catch	**58.9**	**112.0**	**134.4**	**245.5**	**34.4**	**147.4**	**140.8**	**188.1**	**161.0**	**136**
Liquid collected in BDU	g/Nm^3 wet	**1.12**	**2.34**	**2.72**	**4.91**	**0.70**	**3.02**	**3.22**	**4.90**	**3.67**	**3.0**
MEA conc. in collected liquid	mmol/l	841	765.5	789	797.5	780	779.5	697.5	613.5	699.5	751
	g/l	51.4	46.8	48.2	48.7	47.6	47.6	42.6	37.5	42.7	46

Table 5-2: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Concentrations of entrained droplets in the gas flow at different sampling points calculated from tracer experiments.

Time of gas sampling by impingers	Date			**2011-10-08**	**2011-10-10**	**2011-10-10**	**2011-10-11**	**Average**
	Time		Start Stop	16:02 18:32	12:08 16:05	18:04 21:05	16:10 18:10	
Test number same day				1	1	2	1	
Droplet concentration from Li entrainment	mg/Nm3 dry	Before WW	Abs basis	65.0	77.5	91.1	46.2	**69.9**
		After WW	Abs basis				1.9	
		After demister	Abs basis				41.5	
		In BDU	Abs basis	3.2	0.7	1.5	1.3	**1.7**
		Out BDU	Abs basis	3.1	0.6	0.2		**1.3**
Droplet concentration from Rb entrainment	mg/Nm3 dry		WW basis		934.3	1090.2	492.1	**839**
		Before WW	Abs basis		67871.9	4847.2	1069.0	
		After WW	WW basis				54.6	
		After demister	WW basis				0.2	
		In BDU	WW basis			0.4	0.005	
		Out BDU	WW basis			0.1		
Li conc. in BDU catch		µg/l		4.8	12.7	18.4	2.8	
Li conc. in abs liquid		µg/l		1924	1990	2030	1948	
Entrainment catched by BDU	Liquid entrainment from absorber mg/Nm3 wet			**7.6**	**20.6**	**44.4**	**5.2**	**19.4**
	Rb conc. in BDU catch µg/l			0.7	0.9	2.1	1.1	
	Rb conc. in WW liquid µg/l			< 0.25	56446	25201	13688	
	Rb conc. in abs liquid µg/l			< 0.25	3225	4696	6301	
	Liquid entrainment from WW mg/Nm3 wet				**0.05**	**0.41**	**0.30**	**0.3**
Fog formation catched by BDU g/Nm3 wet				2.971	3.377	4.673	3.721	3.7
Entrainment collected in condensate	Li concentration in condensate µg/l			0.074	0.073	< 0.03	0.14	
	Droplets from absorber mg/Nm3 wet			**0.115**	**0.118**		**0.264**	**0.17**
	Rb concentration in condensate µg/l			< 0.25	0.417	< 0.25	0.4	
	Droplets from WW mg/Nm3 wet				**0.02**		**0.12**	**0.07**

Table 5-3: Gas and aerosol sampling analysis results from the campaign at Maasvlakte.
Sauter mean droplet diameter estimate before and after the BDU based on light extinction coefficient and collected concentrations of liquid and MEA

Time of gas sampling by impingers	Date	2011-10-04	2011-10-05	2011-10-06	2011-10-06	2011-10-07	2011-10-08	2011-10-10	2011-10-10	2011-10-11	Average
	Time Start	10:04	11:27	13:30	19:05	16:52	16:02	12:08	18:04	16:10	
	Stop	13:30	15:12	17.10	21:15	18:46	18:32	16:05	21:05	18:10	
MEA Concentrations mg/Nm^3 dry gas	In BDU	87	129	157	173	86	159	146	177	256	152
	Out BDU	1.18	0.97	1.15	2.10	4.37	1.27	1.89	3.95		2
	BDU catch	53	115	131	251	35	148	151	184	166	137
Liquid collected in BDU	g/Nm^3 wet	1.12	2.34	2.72	4.91	0.70	3.02	3.22	4.90	3.67	3.0
Fog sensor measurements	In BDU % of scale	72.8	71.3	74.8	58.8	83.1	70.1	61.7	53.2	52	66.4
	Out BDU % of scale	14.3	10.2	9.07	11.63	9.41	8.71	11.38	10.63	26.7	12.4
Light extinction coefficient	In BDU 1/m	2.186	2.141	2.246	1.766	2.494	2.105	1.853	1.599	1.563	2.0
	Out BDU 1/m	0.217	0.156	0.139	0.177	0.144	0.133	0.173	0.162	0.403	0.19
Sauter mean diameter D(3,2)	In BDU µm	1.36	2.94	3.26	7.49	0.76	3.86	4.62	8.14	6.29	4.3
	Out BDU µm	0.19	0.30	0.39	0.91	0.67	0.49	0.64	1.80	0.20	0.62 [a]
		0.10	0.28	0.37	0.53	0.09	0.43	0.35	0.57	0.17	0.32 [b]
		0.15	0.19	0.28	0.39	1.67	0.39	0.32	1.228		0.58[c]
MEA Conc. mg/Nm^3 dry gas	Gas Eq. out BDU	0.562	0.452	0.440	0.779	0.146	0.350	0.962	1.000	0.219	0.546[c]
	Aerosol out	0.613	0.520	0.713	1.321	4.220	0.924	0.927	2.953		1.524[c]
Droplet conc.	g/m^3	0.011	0.010	0.013	0.023	0.080	0.017	0.018	0.066		0.030[c]

a. Assuming all of the penetrated MEA through the BDU is in liquid phase as aerosol droplets
b. Assuming 0.7 % penetration of inlet aerosol droplets
c. Calculating outlet droplet concentration by deducting equilibrium MEA concentration from total outlet MEA concentration

Table 5-4: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Estimate of condensation nuclei of sulphuric acid

Time of gas sampling by impingers	Date		2011-10-08	2011-10-10	2011-10-10	2011-10-11
	Time Start		16:02	12:08	18:04	16:10
	Stop		18:32	16:05	21:05	18:10
MEA Concentrations mg/Nm^3 dry gas	In BDU		159	146	177	256
	Out BDU		1.27	1.89	3.95	
	BDU catch		147.4	140.8	188.1	161.0
Collected in BDU	Liquid conc. g/Nm3 wet		3.02	3.22	4.90	3.67
Sulphur collected in BDU	Sulphur concentration µg S/l		3087	4917	4878	9733
	Gas conc.	mg S/Nm3 wet	0.0093	0.0158	0.0239	0.0358
		mg H2SO4/Nm3 wet	0.0285	0.0485	0.0731	0.1095
		mg 40 % H2SO4/Nm3 wet	0.0713	0.1213	0.1828	0.2738
		µl/Nm3 wet	0.0549	0.0933	0.1406	0.2106
Condensation nuclei of 40 % H2SO4		number/Nm3 wet	$1.64\cdot10^{12}$	$2.78\cdot10^{12}$	$4.2\cdot10^{12}$	$6.28\cdot10^{12}$
		number/Ncm3 wet	$1.64\cdot10^{6}$	$2.78\cdot10^{6}$	$4.20\cdot10^{6}$	$6.28\cdot10^{6}$
Droplet diameter growing from 40 nm		µm	1.52	1.30	1.31	1.04
Condensation nuclei growing from size 0.04 µm						
Condensation nuclei density 1.3 g/ml						

A possible absorber inlet concentration of 18 mg H_2SO_4/Nm^3 was predicted in Section 2.1.1. This concentration will be reduced through the absorber and the demister possible to a value in the range of 10 mg H_2SO_4/Nm^3. However, only small quantities of sulphur were analysed in the BDU catch giving gas concentrations varying from 0.03 to 0.11 mg H_2SO_4/Nm^3 as shown in Table 5-4. The estimate of condensation nuclei in Table 5-4 is based on this small concentration and that the acid is in the form of 40 nm droplets containing 40% H_2SO_4 and growing to a size corresponding to the aerosol liquid concentration collected in the BDU.

Table 5-5 shows the effect of sulfamic- and sulphuric acid on MEA analysis in liquid. Here, it seems to be hardly any effect or difference between the results using these acids. Table 5-6 shows the effect of sulfamic- and sulphuric acid on MEA concentration measurements in gas samples. The concentrations of MEA in the liquid samples are here 10 times lower. In this case it seems that sampling in sulfamic acid give 6 – 7 % higher concentrations of MEA. This effect has to be studied further in order to determine which of the acids gives the most reliable results.

Table 5-5: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Effect of sulfamic- and sulphuric acid on MEA analysis in liquid

Sample-ID	Journal ID	MEA conc. sample [mmol/l]	Mixture vol. [ml]	Code	MEA conc. BDU Catch not diluted [mmol/l]	Ratio C_A/C_B	Ratio C_A/C_C	Ratio C_B/C_C
2011-10-10 LS Test 1 BDU Catch sample 3 100ml H2SO4 0.1N	P112543	508	268	A	810.4		1.10	1.04
2011-10-10 LS Test 2 BDU Catch sample 2 100ml 0.1N H2SO4	P112563	417	288	A	638.8		1.00	1.00
2011-10-10 LS Test 1 BDU Catch sample 17:00 100ml sulfamic acid 0.1N	P112544	471	261	B	763.5	1.06		
2011-10-10 LS Test 2 BDU Catch sample 3 100ml 0.1N sulfamic	P112564	432	306	B	641.7	1.00		
2011-10-10 LS Test 1 BDU Catch sample 1, without acid addition	P112541	734		C	734			
2011-10-10 LS Test 2 BDU Catch sample 1, without acid addition	P112562	639		C	639			

Table 5-6: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Effect of sulfamic- and sulphuric acid on MEA concentration in the gas.

Sample ID	Gas concentrations (mg MEA/Nm3 dry)		Ratio
	Collecting acid		
	B 0.1 N Sulfamic	**A** 0.1 N Sulphuric	C_A/C_B
2011-10-08 In BDU	165.2	152.1	0.92
2011-10-10 Test 1 In BDU	152.0	139.3	0.92
2011-10-10 Test 2 In BDU	178.9	174.5	0.98
Average			0.94
% increase by method B			6.7

5.2 ELPI and APS measurements

5.2.1 ELPI measurements

The results of the ELPI (Electrical Low Pressure Impactor) measurements around the BDU are summarized in Table 5-7. The concentrations at the top of the tower after the demister are much higher than in the BDU inlet. This is probably due to condensation and impaction of droplets on the walls of the down comer.

Table 5-7: Overview ELPI results

MEA absorber inlet temperature	**date**	**BDU Efficiency number based**	**Inlet concentration BDU [particles/cm^3]**	**BDU Efficiency mass based**	**Inlet concentration BDU [mg/m^3]**
35 °C standard settings	23-11-2011	89.4 %	$6.9*10^5$	99.2 %	370
40 °C standard settings	11-10-2011	94.8 %	$2.4*10^5$	99.4 %	29.4*
40 °C standard settings	13-10-2011	93.3 %	$3.9*10^6$	99.1 %	$1.1*10^3$
45 °C standard settings	11-10-2011	93.2 %	$2.7*10^5$	93.6%	23
45 °C standard settings	23-11-2011	94.1 %	$6.2*10^5$	99.5 %	153
35 °C 100% flue gas	11-10-2011	95.6 %	$2.2*10^6$	98.8 %	75.5

6-10-2011 inlet concentration was 100 mg/m^3 at standard settings T_{top} 40 °C

The BDU capture efficiency for aerosols is above 90% for all cases, except the 35°C standard conditions.

Measurements at the top of the washer after the demister showed such high concentration in droplets larger than 10 µm that the high voltage trap of the ELPI stopped to function within one minute after sampling commenced. This barely left time for the ELPI signal to stabilize and perform a proper measurement. To protect the ELPI and enable adequate sampling times stage 0 of an Anderson impactor (D50 of ca. 11 µm) was inserted in the sampling line as a pre-impactor plate.

During the measurements around the BDU on the 11-10-2011 no pre-impactor plate proved necessary because of the lower concentrations. Figure 5-1 shows the inlet concentration of the BDU with and without the pre-impactor plate. Based on the results the pre-impactor plate causes an underestimation of 20% when measuring with the ELPI.

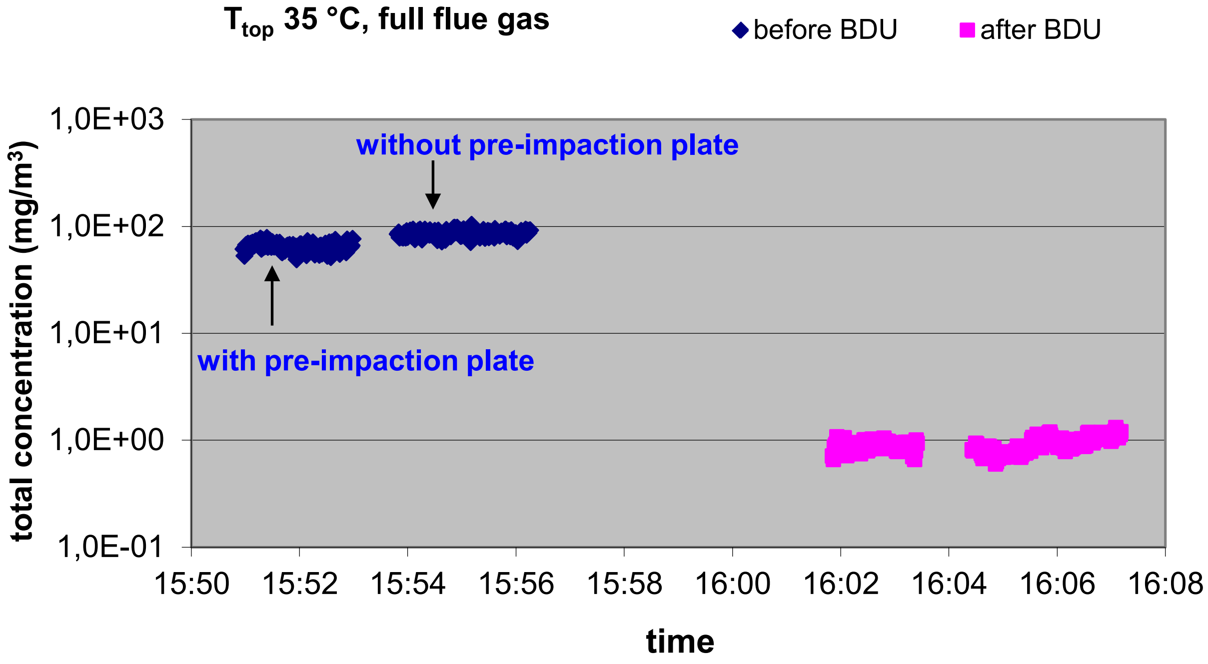


Figure 5-1: Typical overall concentration measurements and effect of pre-impaction plate. Data from 11-10-2011 full flue gas.

During the measurements on 11-10-2011 activities causing significant vibrations were taking place. This may influence the concentrations measured (notably negative currents in certain size classes in the outlet concentration measurements occurred). As a result the efficiency at 40 °C may be overestimated. Especially the smallest size class and the range around 2 µm were visibly influenced.

As a check, a duplicate measurement at 40°C was performed on 13-10-2011, results of this are shown in Figure 5-2. Here both the inlet and outlet concentrations were much higher than on 11-10-2011. Additional measurements at T_{top} 35 °C and 45 °C performed on 23-11-2011, gave much higher concentrations. Because the concentrations measured with the APS on 23-11-2011 were also much higher than on 11-10-2011 it is not likely that the error is caused by dis-functioning of the ELPI due to the vibrations.

It is hypothesized that a sampling error occurred during the 11-10-2011 measurements such as partial blockage of the sampling tube or a setting in the plant was inadvertently varied.

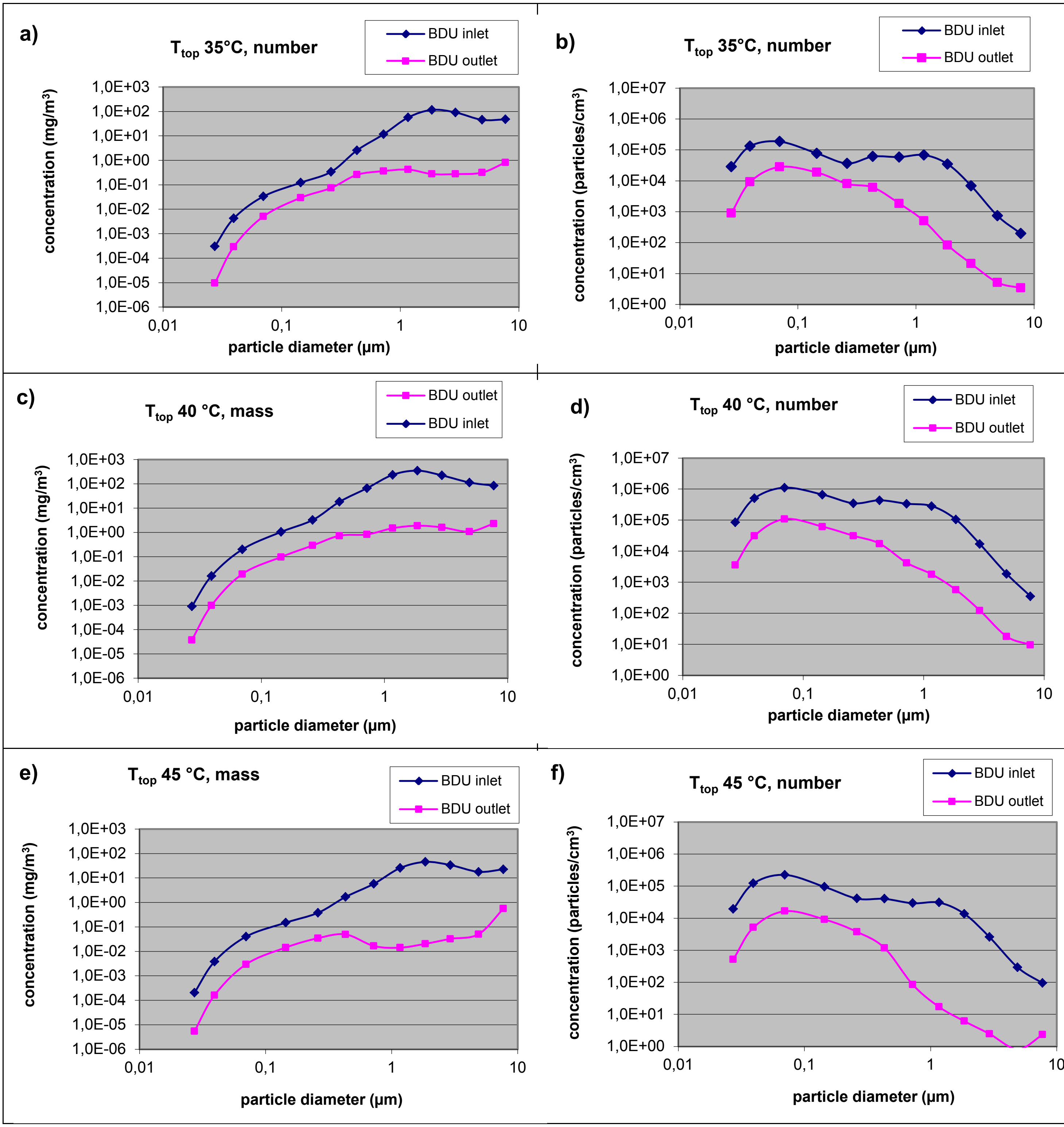


Figure 5-2: BDU inlet and outlet concentrations measured with the ELPI at standard settings, mass concentrations on the left, number concentrations on the right. (a+b) T_{top} 35°C 23-11-2011; (c+d) T_{top} 40 °C13-10-2011; (e+f) T_{top} 45 °C 23-10-2011

5.2.2 APS measurements

The results from the APS (Aerodynamic particle sizer) are summarized in Table 5-8.

Table 5-8: Overview APS results

MEA absorber inlet temperature	Date	Efficiency	Inlet concentration BDU [particles/cm^3]	Inlet concentration BDU [mg/m^3]
35 °C standard settings	11-10-2011	97.6 %	$1.6*10^4$	1.65
35 °C standard settings	23-11-2011	99.7 %	$7.2*10^4$	20.9
40 °C standard settings	11-10-2011	98.8 %	$9.1*10^4$	16.5
40 °C standard settings	05-10-2011	99.4 %	$1.4*10^4$	2.9
45 °C standard settings	11-10-2011	99.5 %	$3.4*10^4$	3.1*
45 °C standard settings	23-11-2011	99.7 %	$5.5*10^4$	15.1
35 °C standard settings	1-10-2011	96.9 %	$6.3*10^4$	2.11

*inlet and outlet concentration measured with pre-impactor. Duplicate without pre-impactor 23-11-2011.

Efficiencies measured with the APS tend to be comparable to those measured with the ELPI.

However, due to the necessary use of diluters larger particles, over 2.5 µm, are barely measured. Volckens and Peters (J. Aerosol Science, 2005) already showed that the APS is less sensitive to larger liquid particles even without diluters and evaporation, whereas solid particle concentrations are measured fairly accurately. The counting efficiency for oil droplets in their research is 25% at 10 µm if no diluters are used. This is attributed to deposition in the APS entrance nozzle. The diluter nozzles are smaller than the APS nozzle, further enhancing deposition losses. Also the diluters lengthen the pathway to the measurement instrument and thus also the evaporation time.

As a result the APS can be used for comparisons, and measurements of non-volatiles. But especially the mass concentrations are heavily underestimated and not representative if diluters are necessary. However, tests without diluters were not representative due to high coincidence rates.

The concentrations measured on 23-11-2011 shown in Figure 5-3 are much higher than on 11-10-2011 and the trends in concentration are the same as was seen with the ELPI measurements.

The temperature of the sampling line has a significant influence on the concentration as can be seen by the difference between BDU outlet concentrations for Ttop 35 °C. When the temperature is too high a significant amount of the droplets evaporates which corresponds to a 1 or 2% difference in the BDU efficiency.

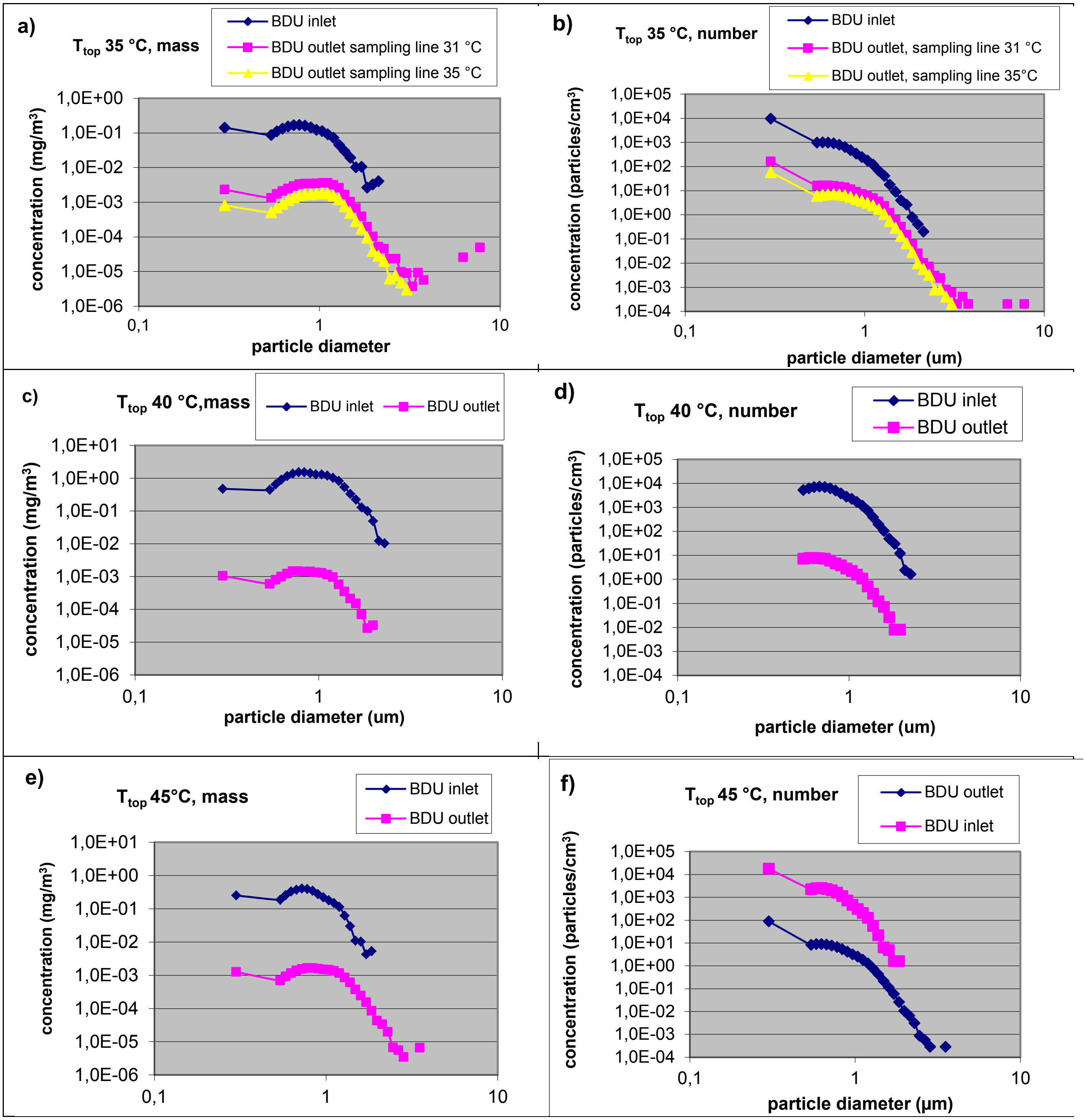


Figure 5-3: Typical results of APS-measurements around the BDU. Due to the necessary use of diluters, the larger particles are filtered out, hence the inlet concentration is underestimated in the larger size range.(a+b) T_{top} 35°C 23-11-2011; (c+d) T_{top} 40 °C13-10-2011; (e+f) T_{top} 45 °C 23-10-2011

5.3 FTIR results and plant operating parameters

The data obtained from pilot plant operation and FTIR are analysed and results are presented here as a) Process parameters, b) Emission results, c) BDU efficiency and d) Fog sensor results. The results obtained from 11th October are not taken into consideration since several operating conditions were changed on the same day for the APS measurements and the results obtained are not consistent with the results from previous days.

5.3.1 Process parameters

Capture rate

Figure 5-4 shows the CO_2 capture rate for the entire period of the campaign. It can be seen that the CO_2 capture rate varies between 88 and 92 % and on an average can be considered to be about 90 % capture.

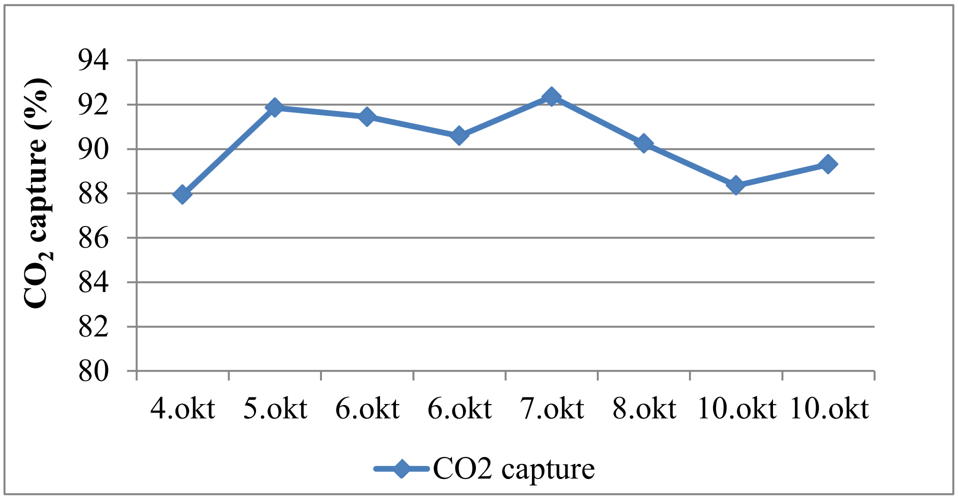


Figure 5-4: CO_2 capture rate for the entire period of the campaign

Pressure drop

Figure 5-5 shows the relation of pressure drop across the BDU with the gas flow rate. There is a clear linear relationship with the gas flow rate and the pressure drop across the BDU. The graph was plotted from data obtained for a particular day (7th October) by increasing the flue gas flow rate to the desired set-point.

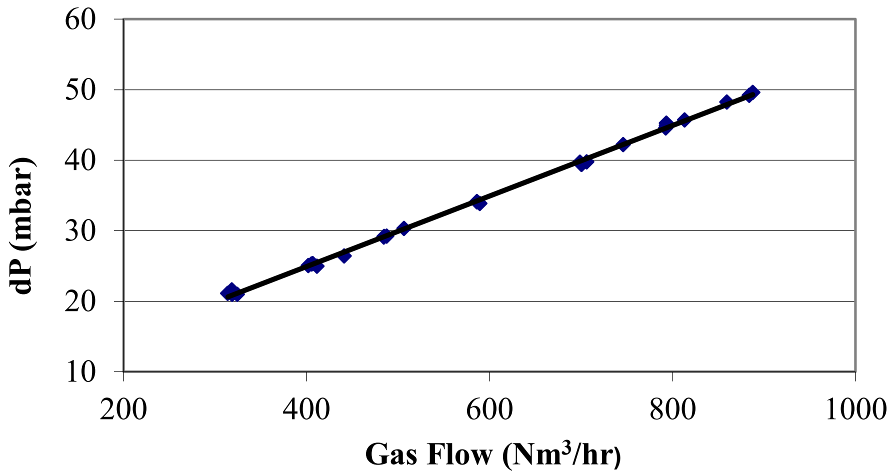


Figure 5-5: Pressure drop across BDU

Figure 5-6 shows the pressure drop across the BDU for the entire period of the campaign. The pressure drop across the BDU increases with time owing to the accumulation of trapped aerosol particles and finally reaches a value of 50 mbar. 50 mbar is the maximum pressure drop the fan can produce and is also the maximum limit for the operation of the BDU filter.

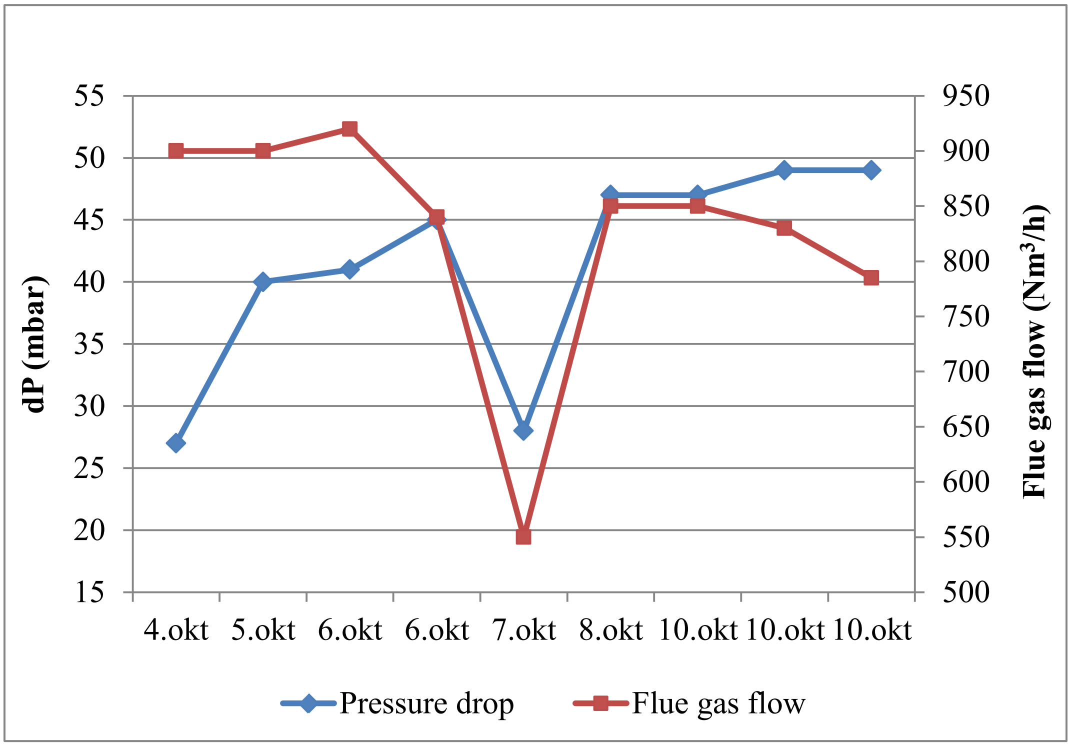


Figure 5-6: Pressure drop across BDU for campaign

It is also observed that for a particular operating point, the pressure drop across the BDU remains constant over the time duration of the experiment as shown in Figure 5-7 .

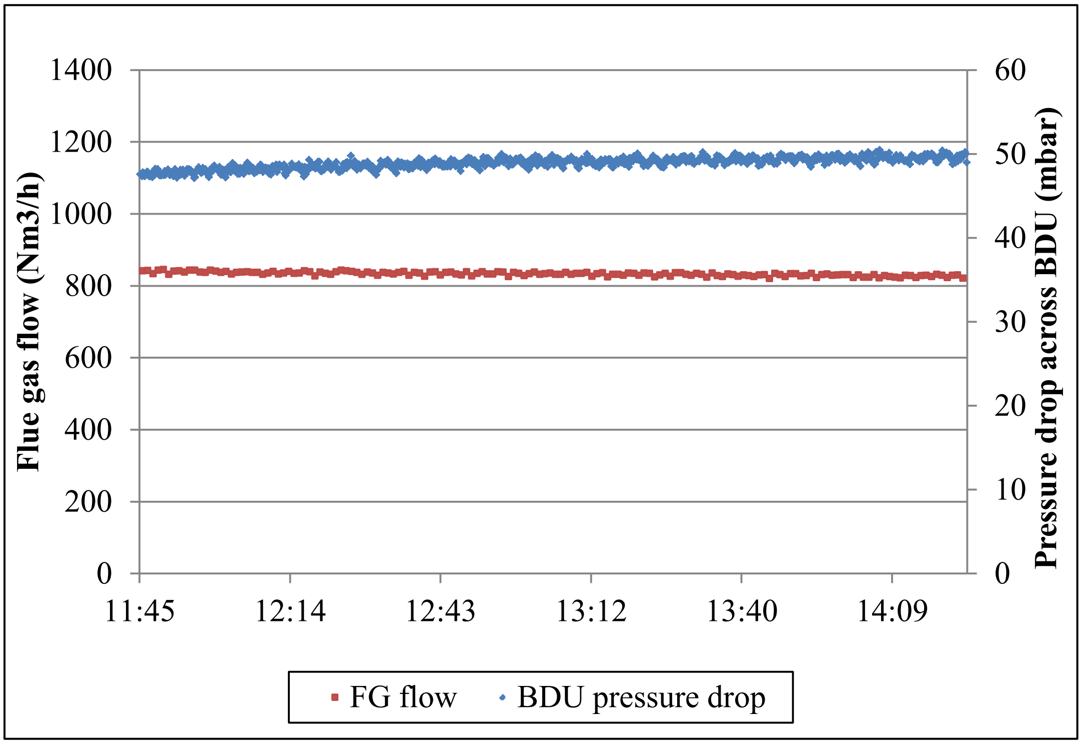


Figure 5-7: Pressure drop across BDU for one particular experiment (**10-10-2011**)

Overall mass balance

A check of the mass balance can be done for MEA, since its concentration is measured at various locations in the system. The gas phase concentrations are known from the FTIR results as well as SINTEF gas sampling analysis. The liquid phase concentrations are obtained from SINTEF measurements. The mass balance is done over the absorber, wash section and the BDU. The analysis presented here is for the 6th October experiment A of the campaign period.

Absorber

Consider the absorber section as shown in Figure 5-8.

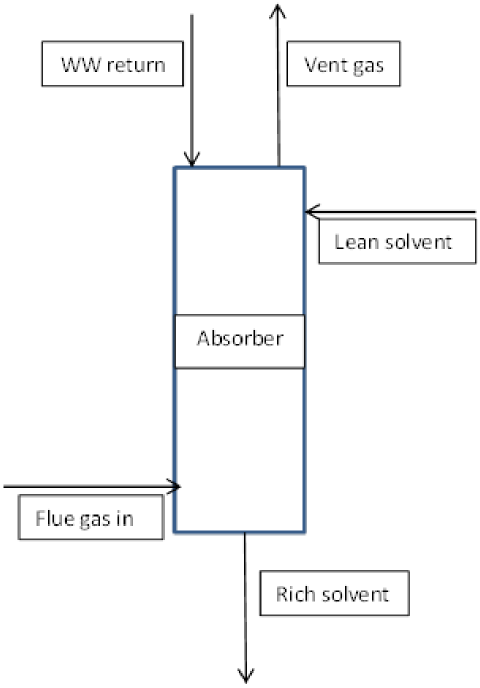


Figure 5-8: Process streams across the absorber

Mass balance streams across absorber

The inlet terms are as shown in Equation 5-1. All the gas and the liquid streams entering the absorber are taken into account, which are Flue gas in: flue gas to absorber, Lean solvent: MEA solvent to absorber and Water wash return: backflow of wash liquid from absorber section which is calculated based on amount of water condensed in the wash section.

$$\boldsymbol{Inlet = Lean\ solvent + Flue\ gas\ in + Water\ wash\ return} \tag{5.4}$$

Table 5-9: Absorber inlet process streams

Component	**MEA concentration (liquid: mmol/L, gas : mmol/Nm^3 wet)**	**Flow rate (liquid:m^3/h, gas: Nm^3/h wet)**
Lean solvent	4190	3,10
Flue gas in	0	884,16
Water wash return	88,3	0,025

Using the values as mentioned in Table 5-9, the inlet term for MEA $(\boldsymbol{Inlet})$ is 12991 mol/h.

The outlet terms are shown in Equation 5-2.The streams leaving the absorber are *Rich solvent*: MEA solvent absorber outlet and *Vent gas*: flue gas leaving absorber.

$$\boldsymbol{Outlet = Rich\ solvent + Vent\ gas} \tag{5.5}$$

Table 5-10: Absorber outlet process streams

Component	MEA concentration (liquid: mmol/L, gas : mmol/Nm^3 wet)	Flow rate (liquid:m^3/h, gas: Nm^3/h wet)
Rich solvent	3760	2.81
Vent gas	6.94	848

Using the values as mentioned in
Table 5-10, the outlet term for absorber is 10571.5 mol/h.

This analyses shows that 81.4 % of the inlet amount of MEA can be accounted for. The difference is due to the error in the measurement values, especially the flow measurement for the rich solvent stream.

Water wash

Similarly, for wash section MEA mass balance can be done over the streams shown in Figure 5-9.

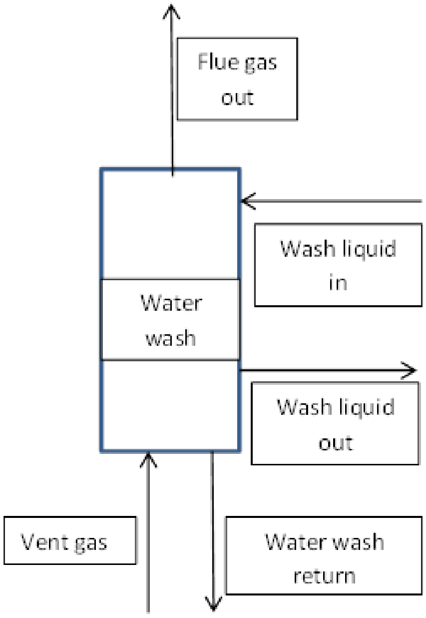


Figure 5-9: Process streams across wash section

The inlet stream to the wash section is the Vent gas from the absorber (Equation 5.6). The wash liquid stream is a closed loop and at steady state thus is not taken into consideration for mass balance.

$$\boldsymbol{Inlet = Vent\ gas} \tag{5.6}$$

Table 5-11: Wash section inlet process stream

Component	MEA concentration (liquid: mmol/L, gas : mmol/Nm^3 wet)	Flow rate (liquid:m^3/h, gas: Nm^3/h wet)

Vent gas	6.94	848

Using the values from Table 5-11 table, the *inlet* term for Wash section is 5.89 mol/h.

The outlet terms are as shown in Equation 5.7. The outlet streams are Flue gas out: flue gas leaving wash section, wash liquid out: wash liquid out in the closed wash liquid loop and the water wash return. The flow rate of the flue gas leaving the water wash is calculated on the basis that 25 kg/h of water is condensed from the gas phase.

$$\boldsymbol{Outlet = Flue\ gas\ out + Water\ wash\ return} \tag{5.7}$$

Table 5-12: Wash section outlet process streams

Component	**MEA concentration (liquid: mmol/L, gas : mmol/Nm^3 wet)**	**Flow rate (liquid:m^3/h, gas: Nm^3/h wet)**
Flue gas out	4.11	848
Water wash return	88.3	0.025

Using the values from Table 5-12 table, the *outlet* term is 5.69 mol/h.

In the wash section, 96.6 % of the inlet amount of MEA can be accounted for, thus giving a closed mass balance considering the errors associated with the measurements.

BDU

A mass balance is done over the BDU, also taking into account the aerosol concentration and the MEA concentration in the aerosol as shown in Figure 5-10.

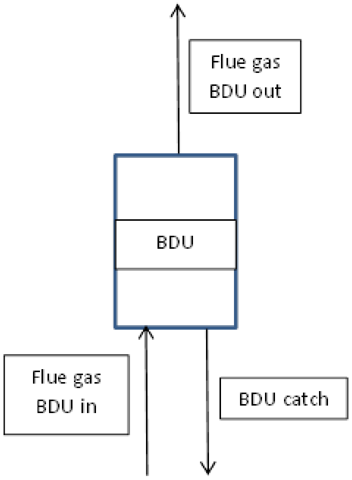


Figure 5-10: Process streams across BDU

The inlet stream to the BDU consists of the flue gas leaving the wash section and entering the BDU:

$$\boldsymbol{Inlet = Flue\ gas\ BDU\ in} \tag{5.8}$$

Table 5-13: BDU inlet process stream

Component	MEA concentration (liquid: mmol/L, gas : mmol/Nm^3 wet)	Flow rate (liquid:m^3/h, gas: Nm^3/h wet)
Flue gas BDU in	2.6	848

Using the values of the flue gas leaving the wash section from the

Table 5-13 *Inlet* =2.21 mol/h. The outlet stream consists of the flue gas leaving the BDU and the aerosol condensate collected in the BDU as BDU catch as shown in Equation 5.9:

$$\boldsymbol{Outlet = Flue\ gas\ BDU\ out + BDU\ catch} \qquad (5.9)$$

Table 5-14: BDU outlet process streams

Component	MEA concentration (liquid: mmol/L, gas : mmol/Nm^3 wet)	Flow rate (liquid:m^3/h, gas: Nm^3/h wet)
Flue gas BDU out	0.2	848
BDU catch	2.1	848

Using the values from the Table 5-14, the *outlet* term for the BDU is 1.91 mol/h.

86.4 % of the inlet MEA to the BDU can be accounted for. A complete mass balance is thus obtained also for the BDU, considering the small numbers associated with the measured values.

Conclusion

A mass balance recovery of 96.6 % is achieved for the wash section while for the BDU it's about 86 %. Thus, a reasonable balance of MEA is achieved for all the sections: absorber, washing and BDU. This implies that the measured values for the liquid phase, gas phase and aerosol phase are in agreement indicating the robustness of the methods.

Temperature profiles

Absorber temperature profile

The temperature profile across the various locations of the system is an important parameter for correlation of emissions, aerosols and also for crosschecking the performance of the analytical instruments. Figure 5-11 shows the temperature profile in the absorber over the period of the campaign. The y-axis indicates the position in the absorber with 1 representing the 1st absorber bed counting from the top while 5 indicating the absorber sump (bottom). It is clear that the magnitude of the temperature bulge in the absorber is increasing with the lean solvent absorber inlet temperature.

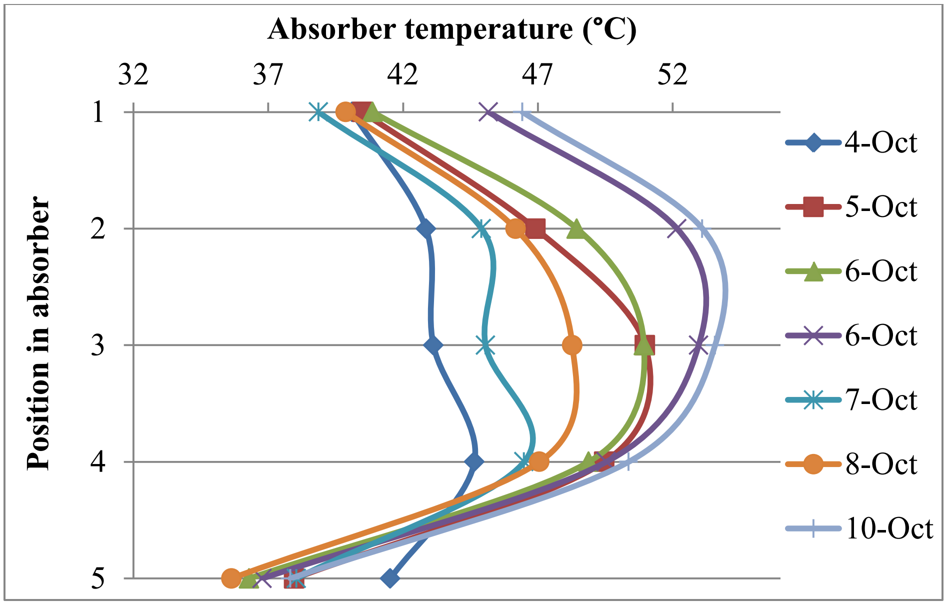


Figure 5-11: Absorber temperature profile

Temperature profile after absorber

Figure 5-12 shows the temperature profile of the flue gas at the 1st absorber bed, flue gas temperature in the down comer and the BDU inlet/outlet temperature. The temperature difference between the top of the 1st absorber bed and flue gas temperature in the down comer is due to the presence of the water wash system at the top of the absorber, which sufficiently cools the flue gas. The flue gas outlet temperature in the down comer is in agreement with the temperature at the BDU inlet, thus implying no significant condensation. Figure 5-13 shows the relation between water vapour (vol %) as measured by FTIR and the temperature across the BDU. It can be seen that the values for the inlet and outlet streams are very similar both with respect to water vapour and temperature.

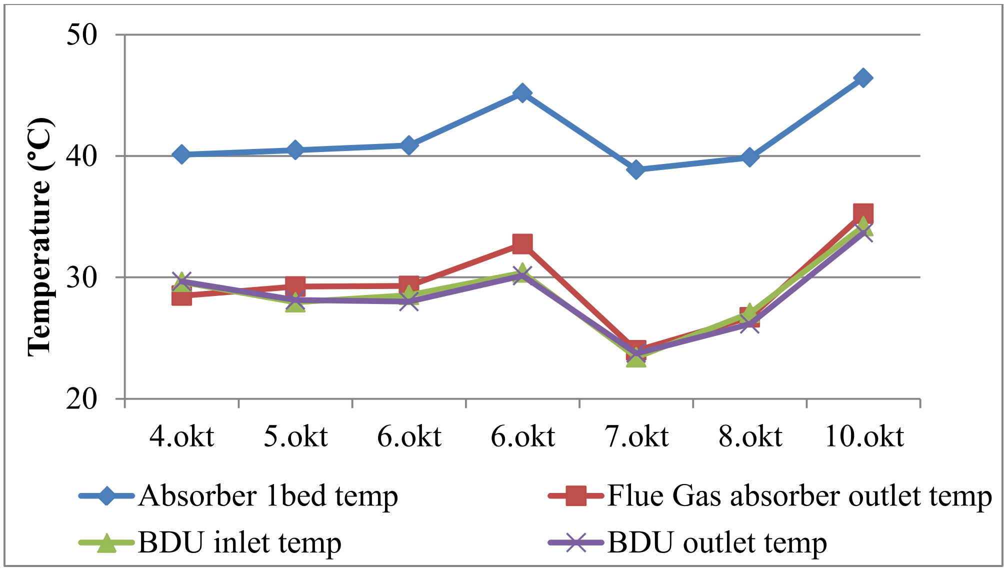


Figure 5-12: Temperature profile of the flue gas at the 1st Absorber bed, flue gas temperature in the down comer and BDU inlet/outlet temperature

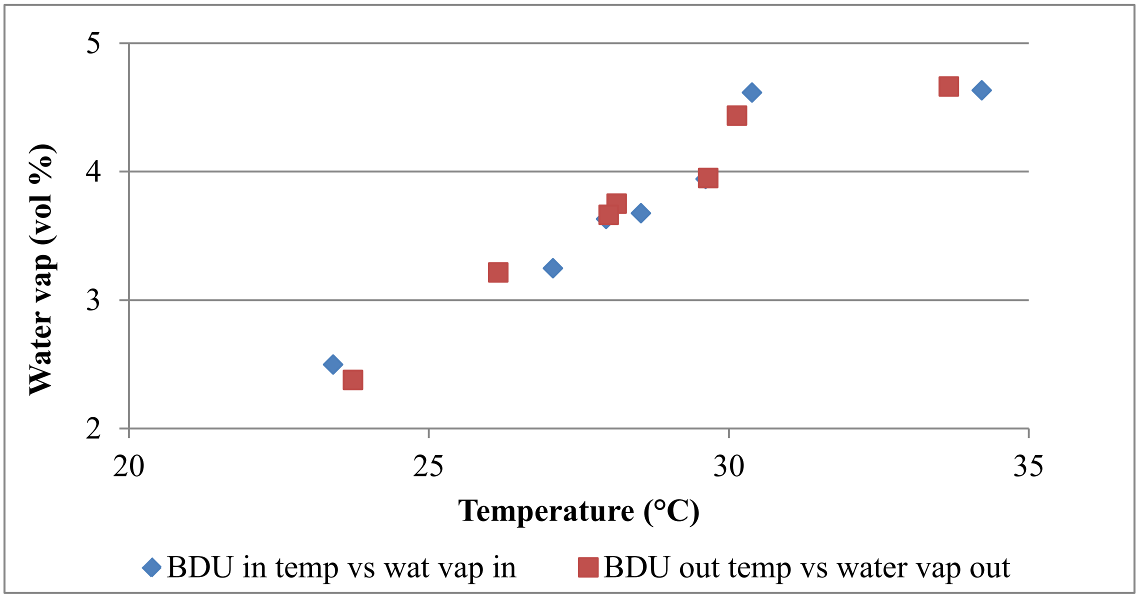


Figure 5-13: Water vapour measured by FTIR plotted with temperatures over BDU

Figure 5-14 shows the BDU inlet temperature with the saturation temperature calculated on the basis of the water vapour content at the BDU inlet. It can be seen that there is strong direct correlation between the two temperatures.

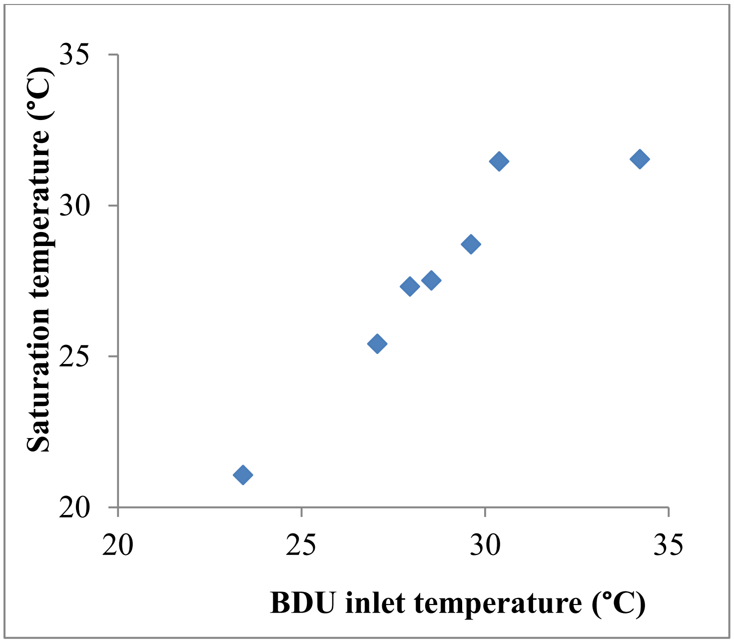


Figure 5-14: Saturation temperature calculated from water vapour content versus measured temperature

5.3.2 Emission results

Emission across BDU

Figure 5-15 shows the average MEA and ammonia emission over each day of the campaign at the BDU inlet. The MEA emission remains more or less constant initially but shows an increasing trend from the 7th October. Ammonia emission is high initially at about 70 mg/Nm^3 but reduces over a period of time to an average value of 25 mg/Nm^3. The ammonia emission are higher than normal emission levels due to high oxygen content in the flue gas, which leads to oxidative degradation in the solvent. The magnitude of the temperature bulge is plotted in the same graph. As seen there is no clear correlation between ammonia emission and the magnitude of the temperature bulge, while there is a slight correlation for MEA and the magnitude of the temperature bulge.

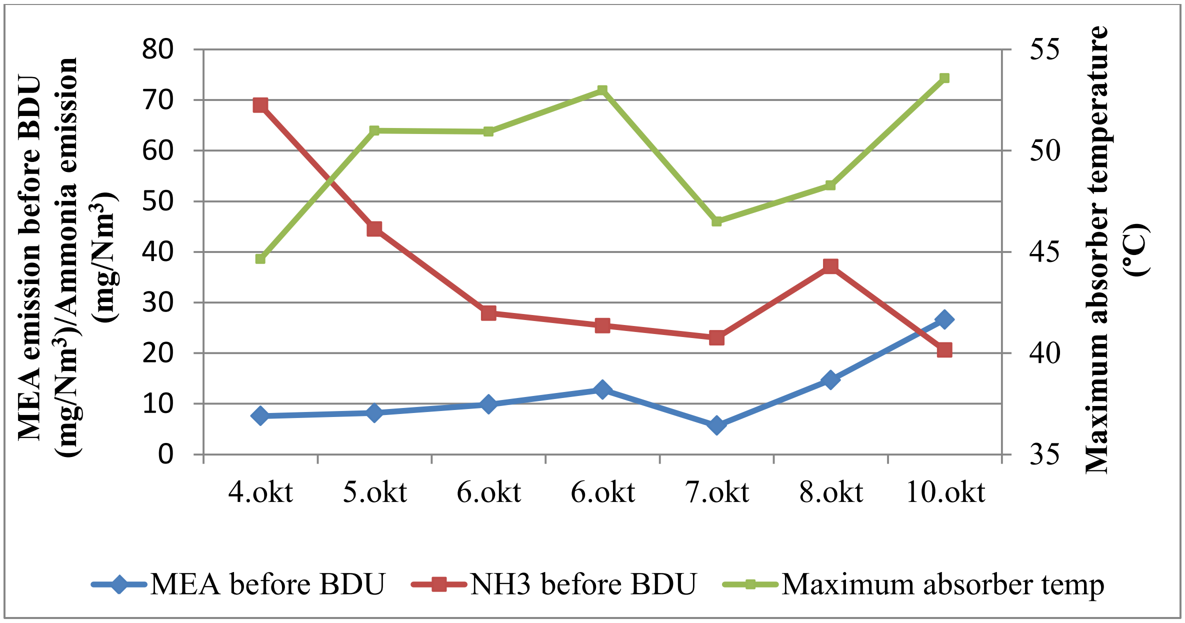


Figure 5-15: Emissions at BDU inlet

Figure 5-16 shows the MEA and NH_3 concentration at the inlet and outlet of the BDU. The MEA concentrations at the outlet are in the range 1 to 4 mg/ Nm^3 thus indicating removal by the BDU. The MEA removal is owing to its presence in the aerosol phase, which is effectively removed by the BDU. There is no change in the ammonia levels across the BDU indicating that the BDU is not effective in ammonia removal. This result is as expected since all the ammonia is present in the vapour phase and not in the aerosols.

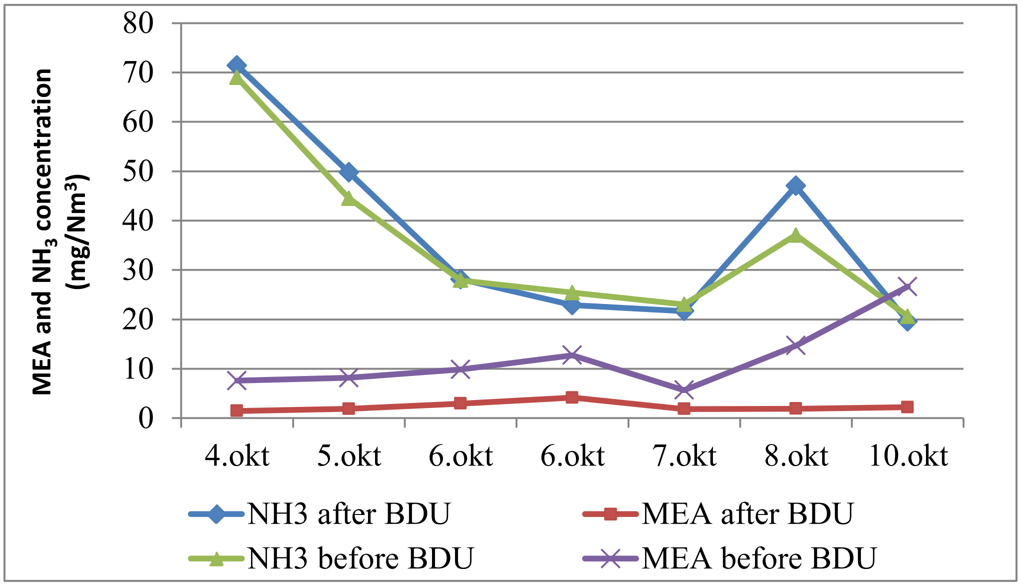


Figure 5-16: Effect of BDU on MEA and NH_3 emissions

In Figure 5-17 the ammonia emissions versus the temperature of the flue gas in the down comer after the water wash section are show. As seen there is no clear correlation between the ammonia emissions and this temperature.

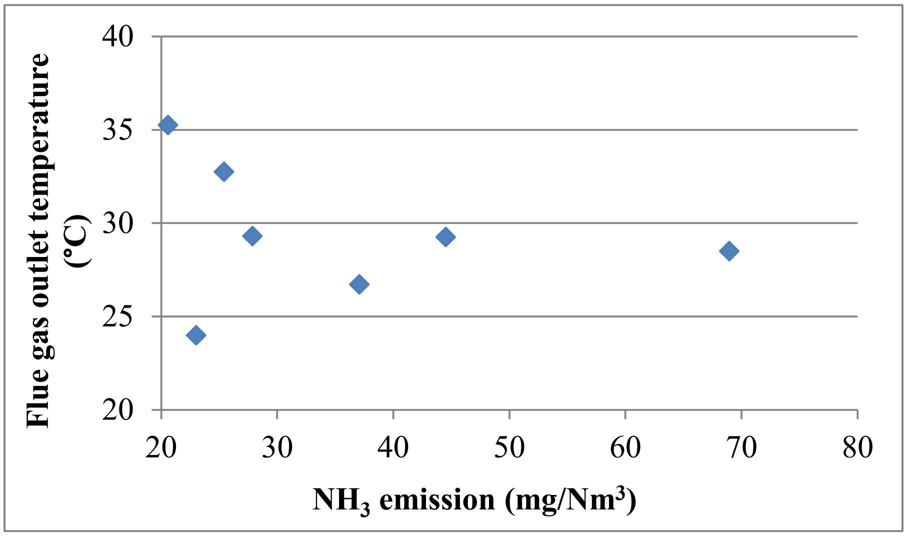


Figure 5-17: NH_3 emission plotted against flue gas outlet temperature

BDU efficiency

Figure 5-18 shows the percentage efficiency of the BDU in MEA removal over the entire period of the campaign. Also the MEA absorber inlet temperature and maximum absorber temperature is plotted to see any correlation. As can be seen the BDU is effective in reducing the MEA emission over all the operating conditions with its efficiency varying from 67 to 92 %. MEA removal efficiency of the BDU follows neither the magnitude of the temperature bulge nor the lean solvent absorber inlet temperature thus implying that the BDU efficiency is not dependent on these two factors.

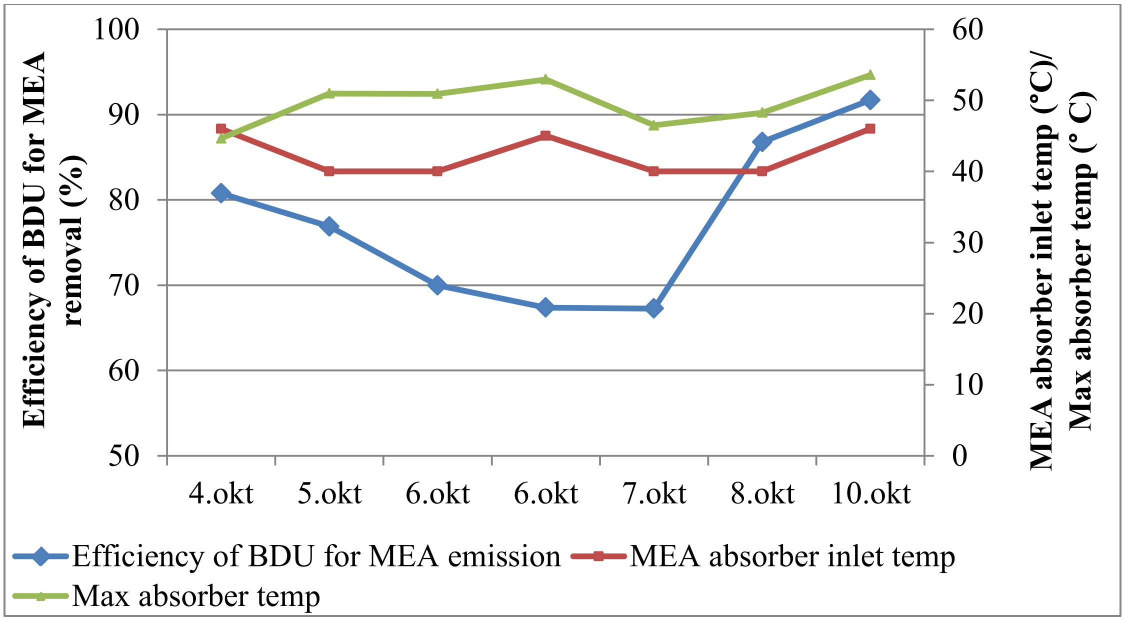


Figure 5-18: Efficiency of BDU for reducing MEA emission calculated with FTIR

Figure 5-19 shows that the BDU efficiency for MEA removal is neither directly nor inversely proportional to the inlet MEA concentration (as determined by FTIR), thus implying no correlation.

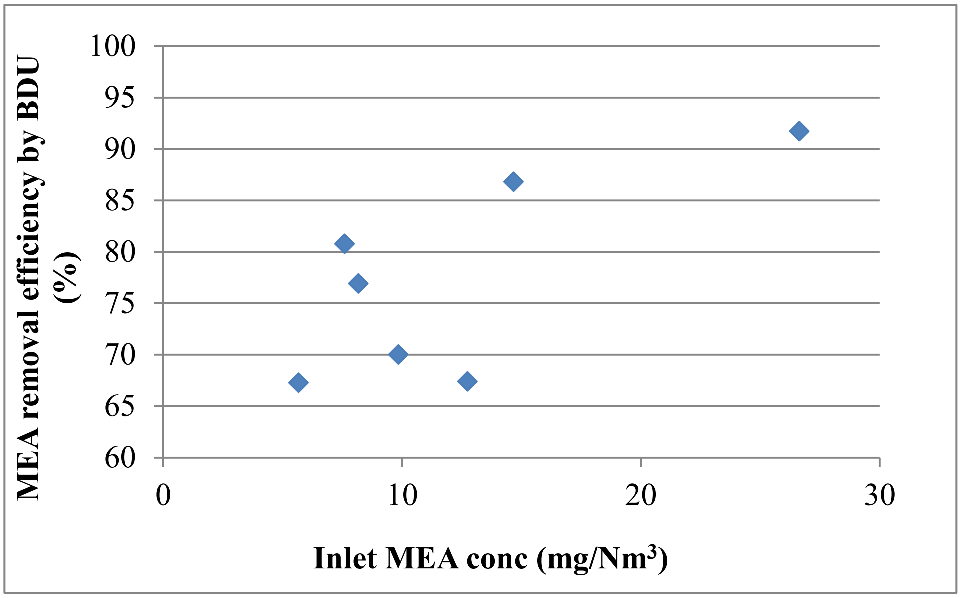


Figure 5-19: Efficiency of BDU for reducing MEA emission plotted against inlet MEA concentration (determined by FTIR) in gas before BDU

5.4 Comparison of MEA emission by FTIR and SINTEF gas sampling method

The MEA emission levels mentioned here were obtained from online monitoring by an FTIR. During this campaign, MEA emission levels were also measured by SINTEF using a gas sampling method as mentioned in Chapter 4. These measurements were done during a specific time interval for each day of the campaign. The results from these measurements along with the FTIR emission results are compared for the same time duration in

Table 5-15. All the data presented here are time averaged and on a dry basis. For both the methods, measurements were taken before and after the BDU.

Table 5-15: Comparison of FTIR results with SINTEF offline analysis

Date and Time	FTIR MEA BDU IN (mg/Nm^3 dry)	SINTEF MEA BDU IN (mg/Nm^3 dry)	FTIR MEA BDU OUT (mg/Nm^3 dry)	SINTEF MEA BDU OUT (mg/Nm^3 dry)
4th Oct 1004-1330h	3.57	87	1.08	1.8
5th Oct 1127-1512h	8.24	129	1.83	0.97
6th Oct 1330-1710h	8.73	157	2.73	1.15
6th Oct 1905-2115h	12.28	173	3.28	2.10
7th Oct 1652-1846h	4.97	86	1.06	4.37
8th Oct 1602-1832h	10.07	159	0.79	1.27
10th Oct 1208-1605h	-	146	-	1.89
10th Oct 1804-2105h	20.72	177	2.11	3.95

The MEA emission levels at the BDU inlet measured by the FTIR are significantly less than the emission measured by gas sampling method of SINTEF. The MEA emission levels measured at the BDU outlet are in reasonable agreement for both the methods. Figure 5-20 shows a plot comparing the analysis methods. There is some qualitative agreement between the methods, but not a very good overall correlation. At low emission levels the methods give similar results, but at higher emission levels the methods give different results.

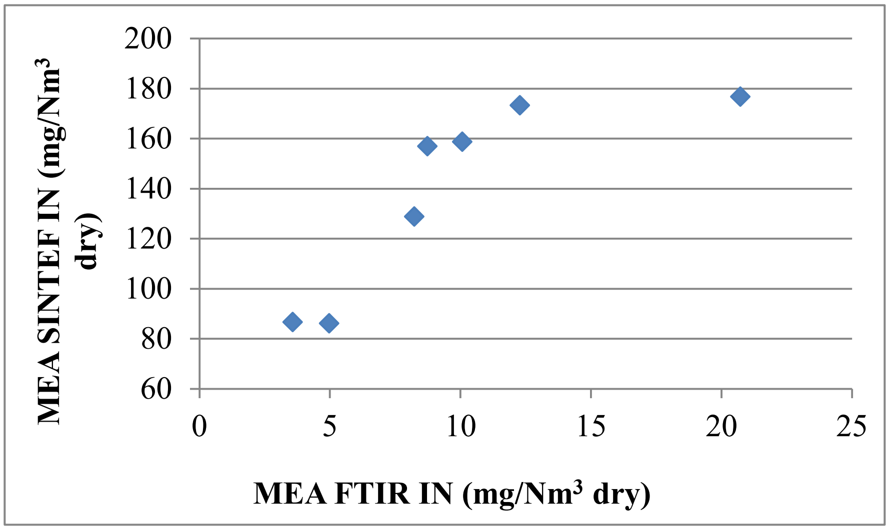


Figure 5-20: Plot of MEA emissions as measured by offline analysis and FTIR

One possible explanation for the difference in the two methods for MEA emission could be that the FTIR sucks in gas at a velocity of approximately 1 m/s, which may not be sufficient to keep the droplets in the gas phase at bends of the sampling pipe due to inertia. The gas velocity in the pipe to the BDU is about 19 m/s. Moreover, the FTIR probe is almost perpendicular to the direction of the flow. The gas sampling done by SINTEF is at a much higher velocity as well as the sampling probe was in the direction of the flow implying collection of larger droplets with sufficient inertia to escape sampling into the FTIR. Thus the samples collected by SINTEF could be more representative. This hypothesis needs to be further investigated.

6 Emission of degradation products

The results of the analysis and subsequent processing and summary of data related to nitrosamines and other degradation products for some of the samples are presented in this Chapter.

6.1 Introduction

It has been observed in prior test campaigns at the Esbjerg pilot plant as part of the CESAR project, that nitrosamines (NA) can be formed in the amine absorption liquid. This is most likely due to reaction of nitrite (resulting from NO_2 absorption) with secondary amines[1] (nitrosation). This is a minor pathway and, therefore, should not consume large amounts of amine or substantially change the amine solution composition. The exact reaction mechanism for the formation of NA is not known at this stage. However, the reaction pathway starts with nitrite present in the absorption liquid.

Due to the fact that nitrosation is only possible for secondary amines, MEA (monoethanolamine), as a pure substance, will not react with nitrite to form NA. However, the MEA used in typical commercial absorption processes and in other test facilities is of technical grade. This means that impurities of secondary amines (e.g. DEA, diethanolamine) can be present. DEA reacts readily with nitrite, forming NAs. Though still subject to research investigation, aldehydes[2] could also catalyse the formation of NAs. In addition, MEA may degrade into secondary amines which can form NAs.

The most relevant degradation product is DEA. Nitrosation of this can lead to N-nitrosodiethanolamine (NDELA). In this case, NAs are second generation degradation products (degradation products of degradation products), thus in small quantities. Next to NDELA, other NA such as nitrosodimethylamine (NDMA) can theoretically occur. NDMA can be formed from the nitrosation of dimethylamine. Dimethylamine (DMA) can be formed as a degradation product. However, in prior pilot plant campaigns (as e.g. the Cesar campaign at the Esbjerg plant) the level of DMA emissions were below detection limit (<0.02 mg/Nm^3). It is of importance to note that DMA is a very volatile degradation product. In theory DMA can react with nitrite to from NDMA (nitrosodimethylamine). However, as indicated by the very low emissions of dimethylamine, this component would not be present to a great extent. It can be concluded that for the presence of NA, NDELA is much more indicative than NDMA.

N-MOR can be formed based on the reaction of nitrite with morpholine (MOR). MOR is a secondary degradation product from MEA. The secondary degradation product MOR originates to a great extent from the degradation products N-(2-hydroxyethyl)-ethylenediamine and diethanol-amine.

6.2 Results

It can be noted that the three nitrosamines were all detected during the emission campaign. All NDELA concentrations analysed in rich solvent were in the range 2.0 – 2.1 mg/l. However, though this content is extremely low it was possible to quantify the content of nitrosodiethanolamine (NDELA), nitrosodimethylamine (NDMA), and morpholine (MOR) in the gas from the absorber. This was successfully achieved by enhancing concentration and mass sensitivities using liquid-liquid extraction (LLE). The results are shown in Tables 6-1 and 6-2, respectively.

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[1] The origin of the secondary amines can be that these amines were present as impurities in the starting material or due to degradation of MEA during operation.

[2] Typical degradation product of MEA during operation

Table 6-1 shows that the average BDU inlet gas concentration of NDELA was 25 ng/Nm^3 dry gas during the measurement campaign, while table 6-2 shows a much higher average gas concentrations of 53 ng NDELA /Nm^3 dry gas based on the BDU catch. This might be caused by NO_x reactions with liquid amines covering the tiny fibres within the BDU depth filter, which expose an extremely large surface to such heterogeneous reactions.

The BDU outlet NDELA concentration in table 6-1 is for one case larger than the inlet concentrations and for another case smaller, showing no significant efficiency of the BDU with regard to collecting NDELA. The same holds for NDMA, while for NMOR collection efficiency seems fairly good. The average inlet and outlet concentrations of NDMA were respectively 8.5- and 9 ng/Nm^3 dry gas. The average inlet and outlet concentrations of NMOR were respectively 18- and 3 ng/Nm^3 dry gas.

Table 6-1: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Concentrations of degradation products in and out of BDU measured for some samples + MEA in the gas flow at different sampling points

Time of gas sampling by impingers		Date	2011-10-04	2011-10-05	2011-10-06	2011-10-06	2011-10-07	2011-10-08	2011-10-10	2011-10-10	2011-10-11
		Time Start Stop	10:04 13:30	11:27 15:12	13:30 17:10	19:05 21:15	16:52 18:46	16:02 18:32	12:08 16:05	18:04 21:05	16:10 18:10
Test number same day			1	1	1	2	1	1	1	2	1
Average flue gas flow during impinger sampling Nm^3/h			701	881	884	833	560	880	839	812	857
Temperatures °C		Flue gas outlet	32	29	29	32	22	27	33	37	25
		SP-BDU	35	31	31	31	31	31	35	35	33
Before WW	**MEA Gas conc.**	**mg/Nm^3 dry gas**			407,4	466,0	212,8	337,5	384,5	460,3	336,4
After WW											206,4
After demister					241,0	251,6	111,5				271,6
In BDU Gas conc.	**MEA**		**86,6**	**128,8**	**156,9**	**173,2**	**86,1**	**158,6**	**145,7**	**176,7**	**255,7**
	NDELA	**ng/Nm^3 dry gas**		**13,5**				**11,0**	**28,1**	**47,0**	
	NDMA			**7,3**					**9,9**	**8,3**	
	NMOR			**4,8**				**6,9**	**36,3**	**22,0**	
Out BDU Gas conc.	**MEA**	**mg/Nm^3 dry gas**	**1,2**	**1,0**	**1,2**	**2,1**	**4,4**	**1,3**	**1,9**	**4,0**	
	NDELA	**ng/Nm^3 dry gas**		**16,3**						**20,5**	
	NDMA			**6,5**						**11,1**	
	NMOR			**2,4**						**4,0**	

Table 6-2: Gas and aerosol sampling analysis results from the campaign at Maasvlakte. Concentrations of degradation products in condensate collection in and out of BDU and in BDU catch measured for some samples + correspondingly for MEA

Time of gas sampling by impingers		**Date**	**2011-10-04**	**2011-10-05**	**2011-10-06**	**2011-10-06**	**2011-10-07**	**2011-10-08**	**2011-10-10**	**2011-10-10**	**2011-10-11**
		Time Start	**10:04**	**11:27**	**13:30**	**19:05**	**16:52**	**16:02**	**12:08**	**18:04**	**16:10**
		Stop	**13:30**	**15:12**	**17:10**	**21:15**	**18:46**	**18:32**	**16:05**	**21:05**	**18:10**
Test number same day			**1**	**1**	**1**	**2**	**1**	**1**	**1**	**2**	**1**
Condensate collection before BDU Gas conc.	MEA	mg/Nm^3 dry gas	8,4	16,6	23,9	20,9	7,9	12,7	11,3	14,8	8,8
	NDELA	ng/Nm^3 dry gas			1,45						0,52
BDU catch Gas conc.	**MEA**	**mg/Nm^3 dry gas**	**58,9**	**112,0**	**134,4**	**245,5**	**34,4**	**147,4**	**140,8**	**188,1**	**161,0**
	NDELA	**ng/Nm^3 dry gas**	**140,3**	**82,6**	**40,8**	**49,3**	**9,4**	**39,1**	**43,6**	**46,9**	**26,0**
	MEA-nitramine		**200,5**	**89,5**	**43,1**	**35,1**	**8,1**	**40,0**	**71,5**	**38,4**	**26,8**
	Liquid collected in BDU	**g/Nm^3 dry gas**	1,15	2,40	2,79	5,04	0,72	3,10	3,30	5,02	3,77
Condensate collection before BDU Liquid conc.	MEA	g/l	4,39	4,39	6,47	7,88	5,11	2,89	3,34	3,00	6,90
	NDELA	**µg/l**									
					0,50						**0,40**
BDU catch	MEA	g/l	51,37	46,76	48,19	48,71	47,64	47,61	42,60	37,47	42,73

7 Overall assessment of the campaign and conclusions

7.1 Overall assessment of the campaign

Based on the aerosol measurements with the ELPI the following conclusions can be drawn:

The concentration at the top of the tower is much higher than at the BDU entrance due to condensation on the wall of the down comer. In the ELPI range (0.024-10 µm particles) the concentration is 3 g/l and more moisture is contained in larger droplets.

Aerosol concentration increases with the flue gas concentration in the gas. If more air is mixed in, the aerosol concentration drops notably.

De BDU unit is very effective in removing aerosols from the gas flow. With an efficiency of around 95% for aerosols the BDU unit is more efficient for aerosol removal than for MEA removal for which the efficiency is approximately 85% with values as low as 65% measured.

No dependency of the BDU efficiency on operating temperature was observed. Only in one instance was a lower efficiency noted. This coincides with a low inlet concentration and low MEA removal efficiency.

Temperature of the sampling lines must not exceed the temperature at the sampling point in the reactor to prevent condensation losses.

The ELPI seems to give representative particle size distributions up to 10 µm. Particles larger than 10 µm cannot be measured. Measurements with a pre-impactor plate indicate that such large droplets are present and the difference between ELPI mass concentration and aerosol mass trapped in the BDU is further confirmation.

The APS with diluters proved only useful for measuring water droplets up to 2-3 µm. Unfortunately, the concentrations in the pilot plant are too high to measure without diluters. Conclusions can be based on relative measures.

7.2 CONCLUSIONS

The objective of this campaign was to evaluate the emission levels based on the different settings, efficiency of the BDU and the use of optical fog sensor as an analytical instrument for indirectly measuring aerosol formation. Thus the conclusions are presented on this basis,

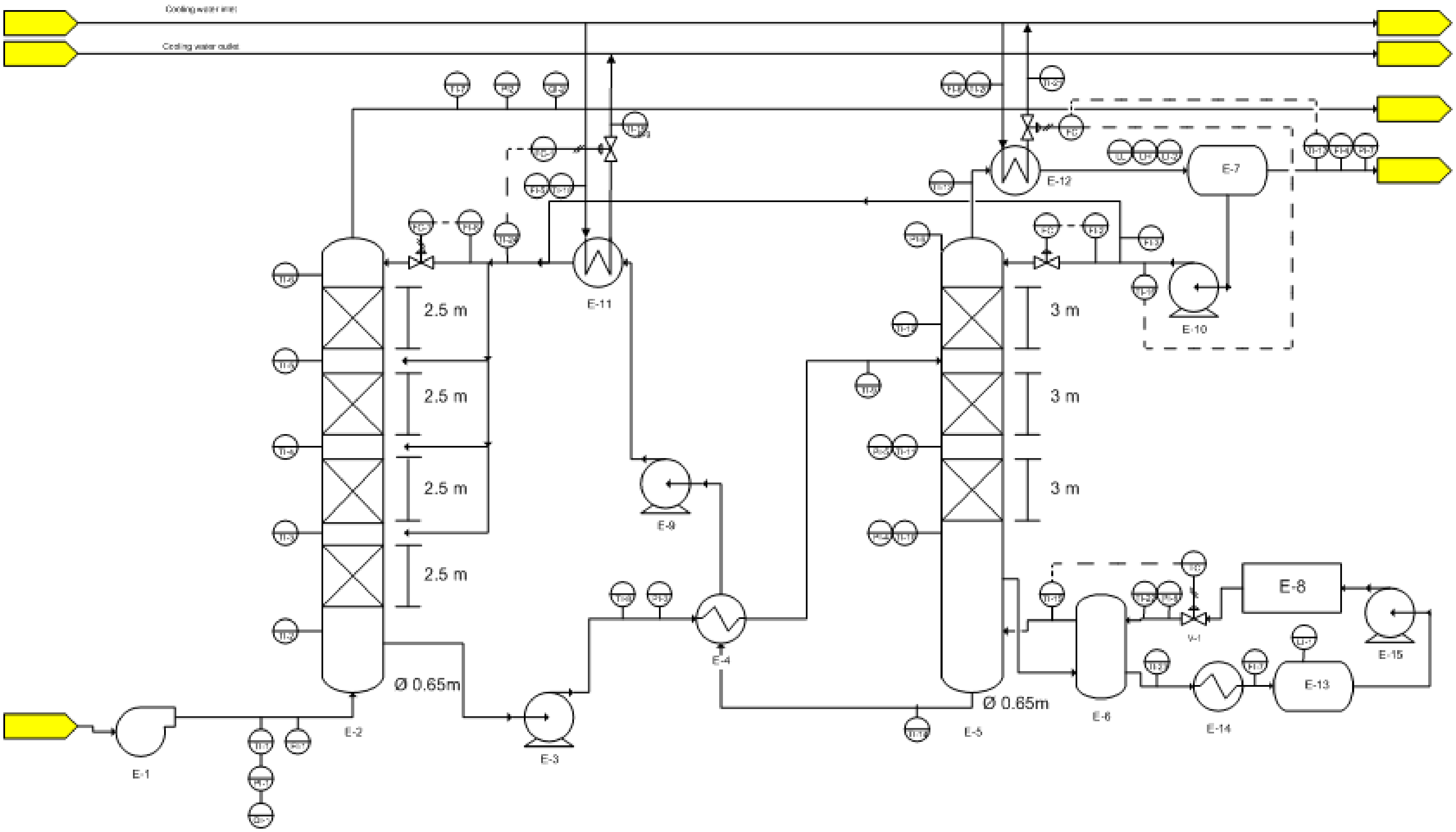
- The BDU is effective in removing MEA emissions, in the range of 67-92 %. Thus, the MEA emission levels at the BDU outlet are in the order of 1-4 mg/Nm^3.
- The BDU is (as expected) not effective in reducing the ammonia emissions, since all the ammonia is present in the vapour phase.
- The efficiency of the BDU is not correlated to the inlet MEA concentration. Thus, higher MEA emission does not imply lower efficiency of the BDU.
- The efficiency of the BDU is also not correlated to either the MEA absorber inlet temperature or the maximum temperature in absorber.
- The BDU is effective in removal of aerosols as measured by the optical fog sensor.

- The maximum temperature in the absorber relates to the temperature of MEA entering the absorber.
- There is no significant temperature change in the pipeline after the absorber indicating that there is no or very little condensation of vapour.
- Ammonia emissions from the absorber are high, in the range of 20-70 mg/Nm^3 due to the higher amount of oxygen present in the flue gas.
- MEA emissions as measured by FTIR are in the order of 10-20 mg/Nm^3 (dry) while the gas sampling method measures much higher MEA emissions in the order of 85-180 mg/Nm^3 (dry).
- The FTIR MEA emissions follow the trend of MEA emissions measured by gas sampling except for the difference in magnitude. The low MEA emissions measured by FTIR could be due to low gas velocity in the sampling which does not allow isokinetic conditions.
- MEA emissions do not correlate to the maximum absorber temperature.

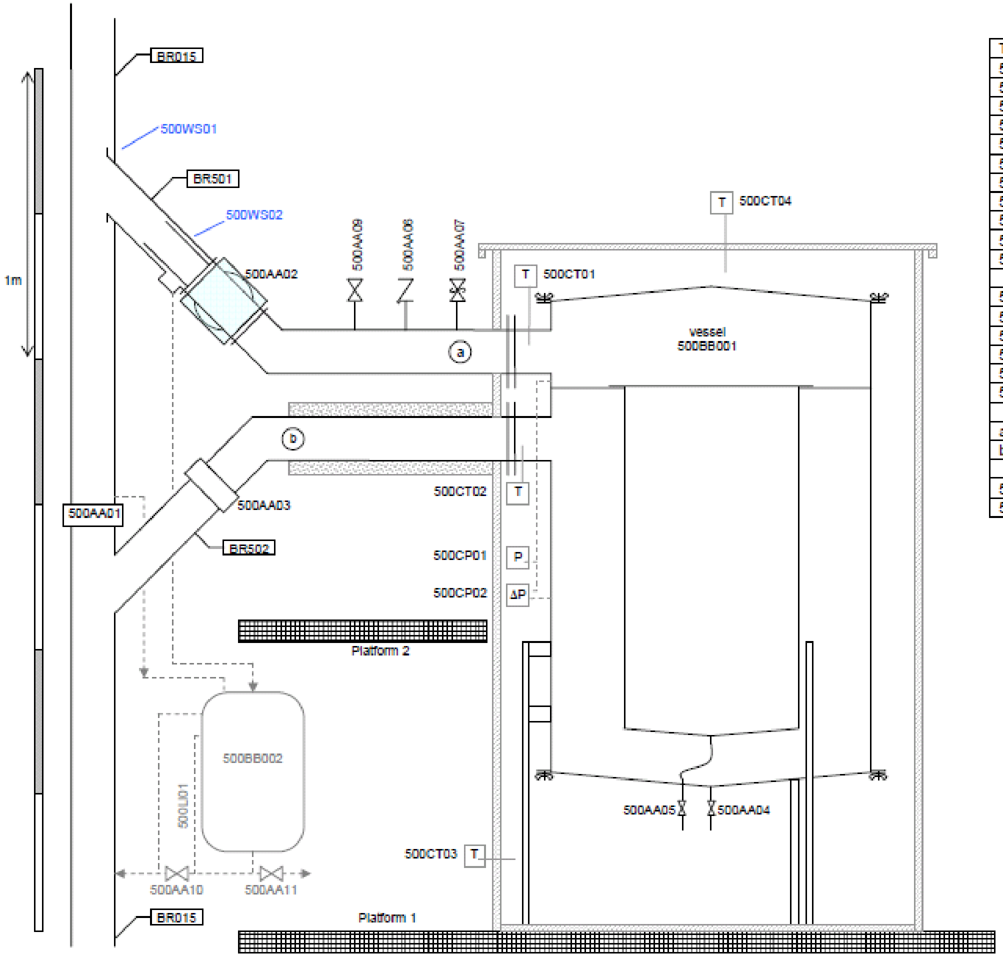
REFERENCE

Mejdell, T., Haugen, G., Tobiesen, A., and Khakharia, P.M., WP 2 in the project: CCM TQP amine 6 – Validation of simulation models, SINTEF report F21934

Appendix 1: Simplified PID Maasvlakte pilot plant prior to modifications



Appendix 2: PID BDU



Tag no.	Description	Line size	Function	Brand - Type
500AA01	Butterfly valve	6"	shut main FG	
500AA02	Ball valve	6"	gate to BD	
500AA03	Butterfly valve	6"	gate from BD	
500AA04	Ball valve	½"	sample from BD sump	
500AA05	Ball valve	½"	sample from BD	
500AA06	Vacuum breaker	3/4"	safety	Swagelok SS-12C2-1
500AA07	Savety valve	½"	safety	Swagelok
500AA08	Ball valve	½"	sample from BD top	Swagelok
500AA09	Ball valve	½"	BD to atmospheric	Swagelok
500AA10	Ball valve	3/8"	condensate to FG out	Swagelok
500AA11	Ball valve	3/8"	condensate sample	Swagelok
500CT01	T fluegas in	1/8"	reg. T in	
500CT02	T fluegas out	1/8"	reg. T out	
500CT03	T box bottom	1/8"	contr. T box	
500CT04	T box top	1/8"	reg. T top box	
500CP01	P	3/8"	reg. total P	
500CP02	DP	3/8"	reg. DP BD	
a	sample spot	3"	sample mist in	-
b	sample spot	3"	sample mist out	-
500WS01	flange on 45° line	6"	remove wall condens.	-
500WS01	5" tube in 6" tube	5"/ 6"	remove large drops	-

Appendix 3: Memo: Test plan for the campaign

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NO 948007029 MVA

Memo

Experimental test plan for CCM amine 6 at Maasvlakte Pilot plant

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Summary

This document describes the execution of the experimental test campaign at the Maasvlakte Pilot plant.

The test campaign is scheduled to start in week 39 and continue for two weeks.

The main focus of the test campaign is to measure droplets formation and transport in the pilot plant absorber and to verify the use of a Brownian diffusion demister as a tool for droplet removal and a subsequent emission reduction.

Introduction

Aerosols

There has in the last year been an increasing awareness that aerosols formed in the CO_2 capture plant may be a significant contributor to total emissions.

While the details of such aerosol emissions are not well-understood and the contribution to total emissions has not been quantified there are some likely key parameters affecting the emission levels:

1. Nature and quantity of potential condensation nuclei entering the CO_2 capture plant.
2. Temperature gradients in the CO_2 capture plant
3. Nature of solvent and degradation products

SO_3 is an example of a component that is known to contribute to very strong aerosol formation. It reacts to form H_2SO_4.

Rapid cooling in the absorber or water-wash system may lead to growth of aerosols, once formed these may be stable and will not be captured in water-wash or demisters. It should be noted that the temperature gradients also depend on the CO_2 content in the exhaust gas.

Aerosol emissions should have the greatest effect for components that have significant vapour pressure and are also hydrophilic. Components that do not have vapour pressure, such as organic acids, will not enter the gas phase at any stage and will not be available for condensation. Components such as ammonia are less hydrophilic and will to a lesser extent condensate.

Importance for CCM project

In the present project the focus should be on extrapolating the findings at the Maasvlakte to a CO_2 capture plant at Mongstad. In order to do this we should attempt to understand how the conditions at Maasvlakte affect the emissions.

When doing emission measurements we would also aim to know all relevant parameters in the plant. At present it seems that impurities in the exhaust gas entering the plant (in other words potential condensation nuclei) is an unknown parameter. It would be very good to have at least some indication of this variable by taking gas and droplet/particle measurements at standard operational condition on the flue gas inlet.

Since the Maasvlakte pilot plant is running using flue gas from a coal fired power plant there is a need to change this composition to simulate the flue gas coming from a gas fired power plant like Mongstad.

This will be done by diluting the flue gas using ambient (outside) air to bring the CO_2 concentration down to 3.4%. In this case the oxygen percentage in the combined flue gas will be 17%, which is slightly higher than the 14% in the Gasnova specification (as given in appendix B). This might have some effect on the degradation rate of the MEA solution.

Operational requirements

To be allowed to work in the E.ON premises, people must have a VCA certificate. VCA stands for “Veiligheid, Gezondheid en Milieu Checklist Aannemers” (Safety, Health and Environment Checklist for constructors). For the present project this concerns personnel from SINTEF (Kai Hjarbo and Herman Kolderup) who will conduct samplings at the Maasvlakte pilot during the campaign. The course has been followed over the internet, and an exam has ben taken in The Netherlands and both have passed (appendix D). Besides that a short, E.ON specific, HSE test has to be passed at the pilot plant site prior to the experimental work.

The candle filter shall be installed, checked and removed prior to the experimental start up. The Brownian Demister setup should be run without the candle filter unit inserted prior to the test campaign to establish the condensation rate in the demister casing as a function of the demister casing temperature. This will ensure the best possible determination of the demister efficiency during the experimental campaign.

Additional experimental equipment to be used by TNO and SINTEF should also be present and tested prior to the campaign. As mentioned before the pilot plant is relaying on the operation of the EON Maasvlakte power plant.

The initial plan was to perform droplet measurements before and after the actual gas sampling period to have a reasonable good indication of the mist characteristics during the sampling period. Due to EON safety requirements, the flue gas flow has to be shut down when the gas and droplet sampling equipment is either inserted or removed from the various sampling points. This will disrupt the operation of the pilot plant, but indications from TNO are that the pilot will be back to normal flows and temperatures within 30 to 60 minutes. This means that it will take around 1 hr. to change and wait for stable conditions before the measurements can be performed whenever a change of sampling points or sampling equipment has to be performed.

Some spare time in the experimental schedule is added to be able to cope with unexpected delays due to the factors mentioned above.

Sampling points

The following sample points (see Figure 1) have been identified for the various tasks

Droplet measurement points:

1. Absorber inlet (after SO2 washer)
2. Absorber wash section outlet (before demister)
3. Absorber wash section outlet (after demister, 300 cm)
4. Before Brownian Demister Unit
5. After Brownian Demister Unit

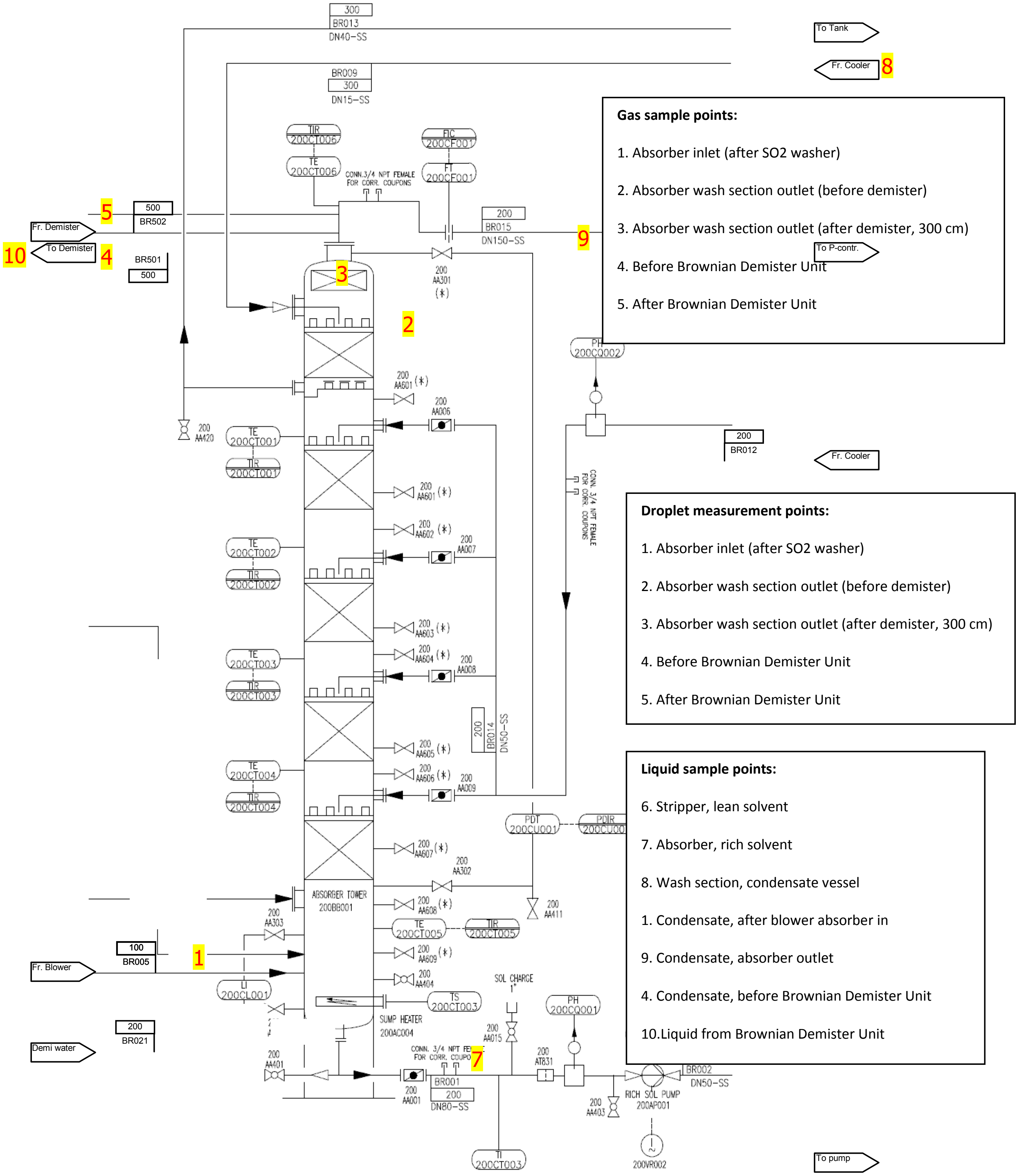
Gas sample points:

1. Absorber inlet (after SO2 washer)
2. Absorber wash section outlet (before demister)
3. Absorber wash section outlet (after demister, 300 cm)
4. Before Brownian Demister Unit

5. After Brownian Demister Unit

Liquid sample points:

6. Stripper, lean solvent
7. Absorber, rich solvent
8. Wash section, condensate vessel
1. Condensate, after blower absorber in
9. Condensate, absorber outlet
4. Condensate, before Brownian Demister Unit
10. Liquid from Brownian Demister Unit



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5. After Brownian Demister Unit

Liquid sample points:

6. Stripper, lean solvent
7. Absorber, rich solvent
8. Wash section, condensate vessel
1. Condensate, after blower absorber in
9. Condensate, absorber outlet
4. Condensate, before Brownian Demister Unit
10.Liquid from Brownian Demister Unit

Figure 1: Absorber PID and sampling points

Experimental schedule

The intention of this campaign is that the experimental tests will be performed in two different phases, the first phase will be conducted without any tracer components added and the focus will be on finding operational settings that will give an increased formation of mist. The second phase will focus on running standard and high mist settings with tracer components added. This will ensure that any influence on the system by the selected tracers is detected and hence accounted for. The total experimental schedule with indicated sampling points and methods is given in appendix A.

Note that the following schedule indicates only days with experiments. Basically, no experiments will be conducted in the weekends, but if required due to any operational delays it is planned to do some extra experiments on Saturdays (not Sundays). During the weekends the pilot plant will be run at standard conditions preferably with the BDU in-line.

The indicated abbreviations concern those persons who will be involved (AH=Arjen Huizinga, AW=?, KH= Kai Hjarbo, HK=Herman Kolderup).

Experimental schedule period 1

Day 1: <u>Pilot plant runs at standard conditions using empty Brownian demister unit</u>

<u>(as specified in app. A)</u>

(AH and AW)

The first day will be used to start up the flow through the empty Brownian demister unit (BDU) at the chosen casing temperature (38 +/-1 ^{0}C). Drainage of free liquid from the empty filter unit and condensate collection vessel will be performed at the end of the day. The filter unit will then be installed and ready for use.

A thorough check of the droplet distribution in the pilot plant will also be conducted. This will be done to check for the influence of possible fluctuations (time dependent), the cross sectional position, and the actual gas sampling position. The droplet measurements will be performed using the APS instrument on measuring position 3 and 4. If time permits, sampling using the APS/ELPI should also be done in position 1 (flue gas inlet).

This will give the baseline droplet information for standard operating conditions. No gas and liquid sampling will be performed during this day due to the EON regulations that will prolong the experimental duration.

Day 2: <u>Dependency of temperature conditions (as specified in app. A)</u>

AH+AW+KH+HK

The BD filter must be installed and ready to use before the tests can start.

This test will emphasize on creating a high delta T in the top of the absorber to create favorable conditions preferable to mist formation.

- Change (lower) the lean liquid temperature entering the absorber to create more mist.
- Optional change (lowering) the wash water flow rate entering the absorber. The variation possible of the cooling water temperature to create a change in delta T in the top of the absorber is considered to be too small to create conditions that should give variation in the mist formation. This will only be tested if time allows.
- Change the liquid flow to make sure that the temperature bulge is in the top of the packed section

Gas sampling will be performed at the predefined positions 3, 4 and 5. The droplet measurements will be performed using the APS instrument

Day 3: Dependency of gas velocity (as specified in app. A)

AH+AW+KH+HK

The effect of high gas velocity on droplet sizes and distribution will also be evaluated. Gas sampling will be performed at the predefined positions 3, 4 and 5. The droplet measurements will be performed using the APS instrument.

- Increase flue gas flow, or if pressure drop too high in BDU, increase the absorption liquid flow to obtaina lower gas liquid ratio. Possibly a combination of the two may be used.

Gas sampling will be performed at the predefined positions 3, 4 and 5. The droplet measurements will be performed using the APS instrument

Day 4: Variation of capture rate on gas emission and droplet measurements (as specified in app. A)

(AH+AW+KH+HK)

- The pilot plant will be run at high capture level (95% = 0.17% CO_2 in outlet) to evaluate the effect of this factor. The droplet measurements will be performed using the APS instrument on measuring position 3.
- The pilot plant will then be run at low capture level (85%= 0.51% CO_2 in outlet) to evaluate the effect of this factor. The droplet measurements will be performed using the APS on measuring position 3.

Based on the raw data we will decide on which capture level to use for the gas sampling period. High capture level may be the best candidate since a higher amine concentration in the mist may be expected.

Gas sampling will be performed at the predefined positions 3, 4 and 5.

Droplet measurements will be performed using the APS on measuring position 3, 4 and 5.

Drainage of free liquid from the Brownian demister unit and condensate collection vessel will be performed at the end of the day.

Day 5: Standard conditions with gas emission sampling and droplet measurements (as specified in app. A)

(AH+AW+KH+HK)

This will give a repetition of the baseline droplet information for standard operating conditions. In addition gas sampling with respect to chemical analysis of amine and degradation products will be performed.

Gas sampling will be performed at the predefined positions 3, 4 and 5.

Droplet measurements will be performed using the APS on measuring position 3, 4 and 5.

Drainage of free liquid from the filter unit and condensate collection vessel will be performed at the end of the day.

Thorough inspection and possible rinsing and cleaning of the Brownian demister unit will be performed.

The preliminary results from the experiments performed will be used to establish a set of parameters that will create a “high mist” and possibly “no/low mist” condition in the pilot plant.

Experimental schedule period 2

Day 6: Pilot plant runs at standard conditions (as specified in app. A)

(AH+AW+KH+HK)

Addition of a pair of the selected non-volatile tracer components to both the amine solution and the wash water system will be performed at this point

The droplet measurements will be performed possibly using both the APS and ELPI instrument.

Drainage of free liquid from the filter unit and condensate collection vessel will be performed at the end of the day. Additional samples of wash water and amine solution will be taken for tracer analysis purposes.

This will give the standard droplet information for the specific operating conditions.

Gas sampling will be performed at the predefined positions 3, 4 and 5.

Day 7: Pilot plant runs at "high mist" conditions with gas emission sampling and droplet measurements (as specified in app. A)

(AH+AW+KH+HK)

The droplet measurements will be performed possibly using both the APS and ELPI instrument.

In addition gas sampling with respect to chemical analysis of amine, degradation products and tracer components will be performed at the predefined positions 3, 4 and 5.

Drainage of free liquid from the filter unit and condensate collection vessel will be performed at the end of the day. Additional samples of wash water and amine solution will be taken for tracer analysis purposes.

This will give the “high mist” droplet information for the specific operating conditions.

Day 8: Pilot plant runs at “high mist” conditions with gas emission sampling and droplet measurements (as specified in app. A)

AH+AW+KH+HK

Addition of second a pair of the selected non-volatile tracer components to both the amine solution and the wash water system will be performed at this point

The droplet measurements will be performed possibly using both the APS and ELPI instrument.

In addition gas sampling with respect to chemical analysis of amine, degradation products and tracer components will be performed at the predefined positions 3, 4 and 5.

Drainage of free liquid from the filter unit and condensate collection vessel will be performed at the end of the day. Additional samples of wash water and amine solution will be taken for tracer analysis purposes.

This will give the “high mist” droplet information for the specific operating conditions.

Day 9: Standard conditions with gas emission sampling and droplet measurements (as specified in app. A)

(AH+AW+KH+HK)

This will give a repetition of the baseline droplet information for standard operating conditions. In addition gas sampling with respect to chemical analysis of amine, degradation products and tracers will be performed at the predefined positions 3, 4 and 5.

Drainage of free liquid from the filter unit and condensate collection vessel will be performed at the end of the day.

Day 10: Spare day to allow for practical problems that may have resulted in a shift in the experimental plan.

AH+AW+KH+HK

Thorough rinsing and cleaning of the Brownian demister unit (BDU) will be performed.

In addition equipment and samples has to be prepared for transport/shipment to SINTEF/TNO

Experimental procedures

The following experimental procedures will be used to obtain sound and reliable experimental samples and results for the determination of possible emission related problems in MEA operated CO_2 capture plants.

APS measurements

1. Measure the local temperature of the process at the sample point. This should not be above 40°C.
2. Make sure the three-way valve on the APS is set to deliver dry air to the analyser.
3. Attach the sample line to the sample point.
4. Trace and insulate the sample point. The set-points should be set to the process temperature at the sample point.
5. Wait until the equipment is on temperature. When the thermostated box starts at room temperature, this takes about an hour.
6. After this, the three-way valve can be set to the sample position. The measurement should take about half-an-hour.

To prevent condensation in APS-diluter nozzle flush for 10 minutes with dry air every 30/60 minutes as needed. A 3-way valve will be installed to this end, possibly with silica gel or similar to further dry the flush air for the APS.

Sampling time will be 60 seconds.

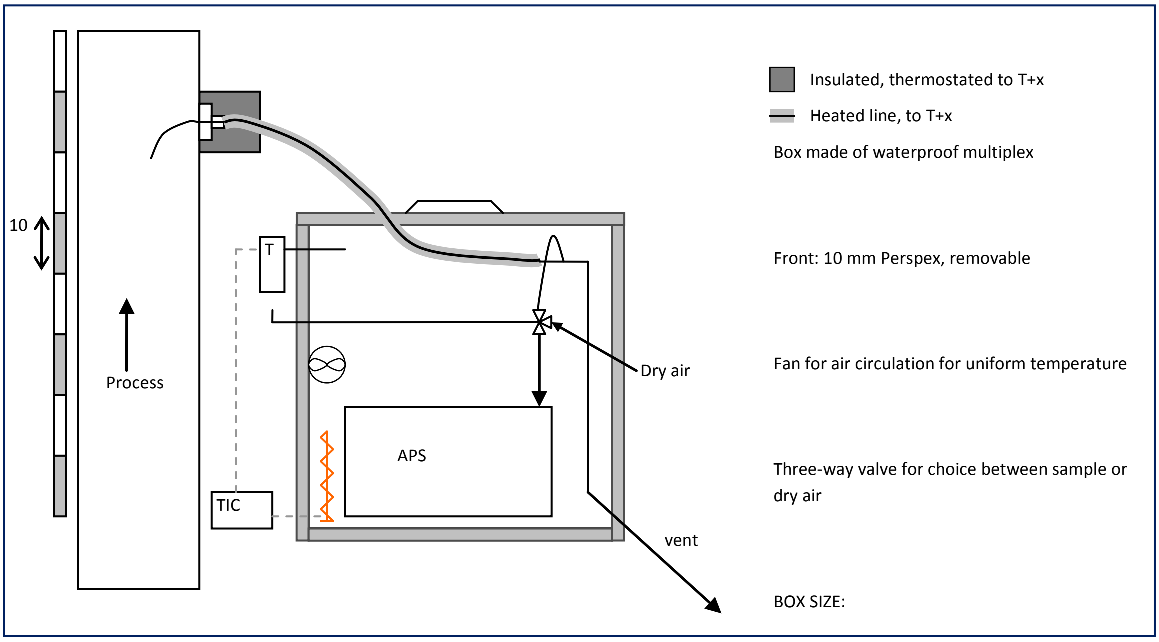


Figure 2: APS box.

ELPI measurements

Connection and thermostating procedure is the same as that of the APS.

Flush with dry air after 30/60 minutes. A 3-way valve (with filter) will be installed to this end

Sampling time 10/20/30 seconds. Clean ELPI at the end of every week.

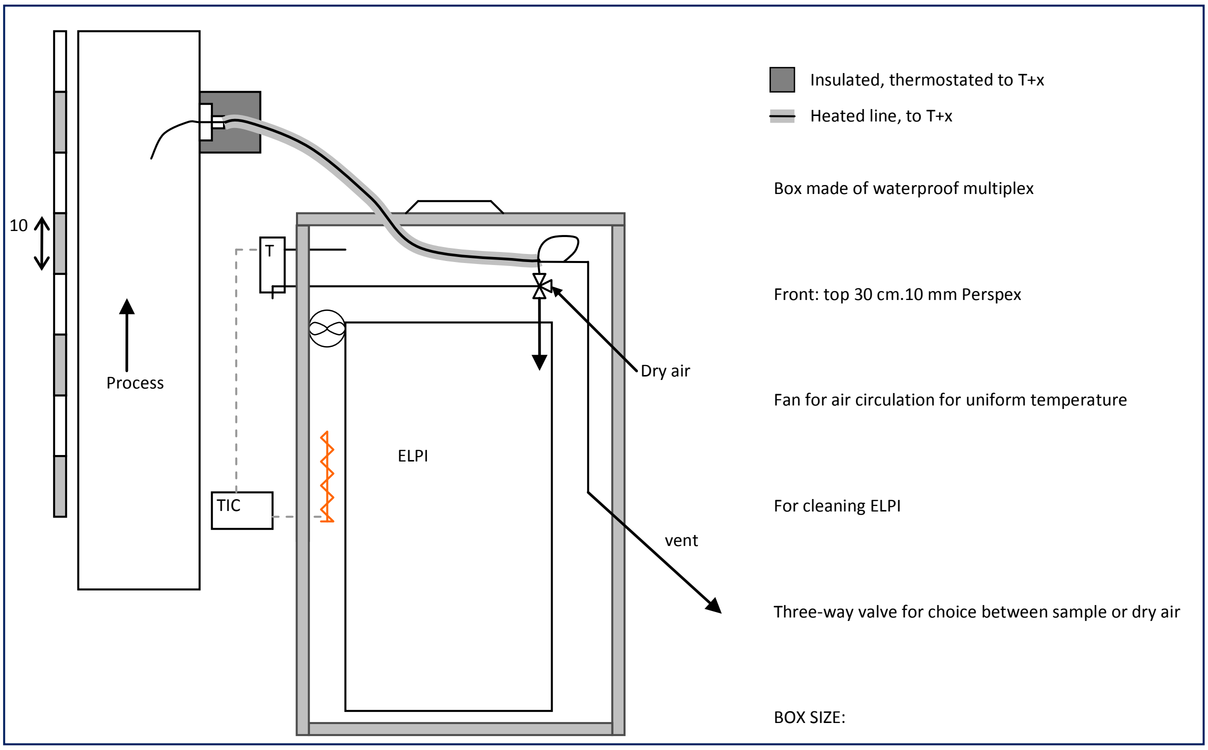


Figure 3: ELPI box

Gas component sampling

The gas component sampling will be conducted using both iso-kinetic and non iso-kinetic sampling methods depending on the objective of the test. The gas sample will be collected using a “sampling train” to capture both mist particles and volatile components from the gas phase.

The sampling train will consist of the following items. First the gas will be collected through a sample tube to be passed through three glass bottles followed by a silica gel drier before the sampling pump and a gas meter. Two impingement bottles will be used to ensure maximal removal of mist particles in the gas. The third bottle will be an empty one to collect any liquid spill over from the last impingement bottle. All bottles will be sitting in an ice-water bath to ensure minimal re-evaporation of volatile gas components. The two first impingement bottles will contain sulphonamide acid (≈100 ml, 0.1N). The sampled gas volume will be approximately 5-6 m^3 in a period of 3-4 hrs. in order for the impingement bottles to operate efficiently. It would be of great value to sample the three proposed sampling locations simultaneously for comparison of results, thus at lest three sampling trains should be prepared. If possible this sampling should be performed together with the droplet size measurements in order to have the best possible data basis for later interpretation.

1. Absorber inlet (after SO2 washer) *(once for baseline, non iso-kinetic)*
2. Absorber wash section outlet (before demister) *(iso-kinetic)*

3. Absorber wash section outlet (after demister, 300 cm) *(iso-kinetic)*
4. Before Brownian Demister Unit (*non iso-kinetic)*
5. After Brownian Demister Unit *(non iso-kinetic)*

The samples will be stored dark and in a cooler until the chemical analysis is performed.

Liquid sampling

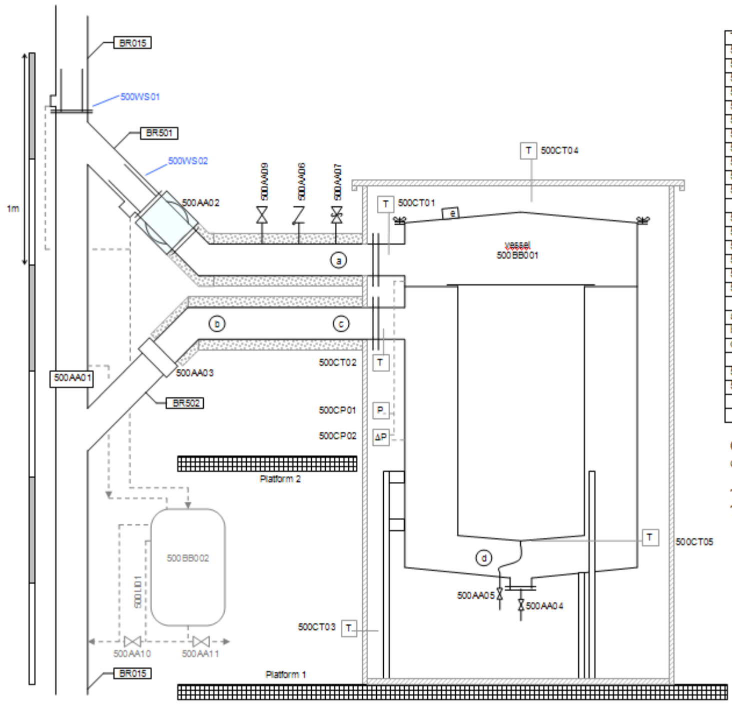
The necessary volumes of liquid will be collected from sampling valves at the sampling points. The first 100 ml (or an otherwise found necessary volume) of liquid will be discarded and then the required sample volume will be collected in order to obtain a representative sample. The samples taken should be cooled and stored in dark containers. (Any chemicals deemed necessary for sample “protection” may be added.)

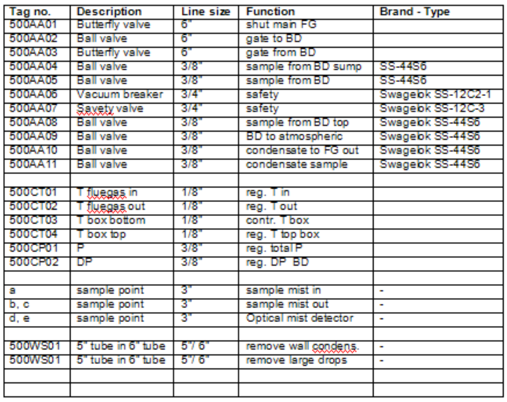
6. Stripper, lean solvent (500ml)
7. Absorber, rich solvent (500 ml)
8. Wash section, condensate vessel (500 ml)
1. Condensate, after blower absorber in (500 ml)
9. Condensate, absorber outlet (500 ml)
4. Condensate, before Brownian Demister Unit (500 ml)
10. Liquid from Brownian Demister Unit (500 ml)

The total volume of condensate from the collection vessel before the BDU is always registered together with actual time of collection if this has to be done regularly in order to avoid over flow situations. Possibly only part of the condensate collected is stored for analytical purposes.

Brownian Demister operation

In the following paragraphs the operation of the Brownian demister is explained.





Tag no.	Description	Line size	Function	Brand - Type
500AA01	Butterfly valve	6"	shut main FG	
500AA02	Ball valve	6"	gate to BD	
500AA03	Butterfly valve	6"	gate from BD	
500AA04	Ball valve	3/8"	sample from BD sump	SS-44S6
500AA05	Ball valve	3/8"	sample from BD	SS-44S6
500AA06	Vacuum breaker	3/4"	safety	Swagelok SS-12C2-1
500AA07	Savety valve	3/4"	safety	Swagelok SS-12C-3
500AA08	Ball valve	3/8"	sample from BD top	Swagelok SS-44S6
500AA09	Ball valve	3/8"	BD to atmospheric	Swagelok SS-44S6
500AA10	Ball valve	3/8"	condensate to FG out	Swagelok SS-44S6
500AA11	Ball valve	3/8"	condensate sample	Swagelok SS-44S6
500CT01	T fluegas in	1/8"	reg. T in	
500CT02	T fluegas out	1/8"	reg. T out	
500CT03	T box bottom	1/8"	contr. T box	
500CT04	T box top	1/8"	reg. T top box	
500CP01	P	3/8"	reg. total P	
500CP02	DP	3/8"	reg. DP BD	
a	sample point	3"	sample mist in	-
b, c	sample point	3"	sample mist out	-
d, e	sample point	3"	Optical mist detector	-
500WS01	5" tube in 6" tube	5"/ 6"	remove wall condens.	-
500WS01	5" tube in 6" tube	5"/ 6"	remove large drops	-

Connection of drain in candle bottom: 3/8" O.D. nipple for flexible hose to 3/8" nipple connected to tube of valve 500AA05. (facilitates removal of bottom lid)

Thermostated box: 20 mm waterproof multipex, partly removable, see drawing 110806 Thermostated box.doc

Attach lids

The stepwise procedure to attach the lids is as follows:

1. Open valve 500AA09
2. Make sure the demister is clean and dry
3. Work from Platform 2. Lift the lid onto the demister with two persons.
4. Fix the lid with the wingnuts.
5. For the bottom lid, make sure, the hose connection to the bottom of the filter-candle is fixed.
6. Lift the lid into the holding clamps, then close the seal using the wingnuts.
7. Make sure valves AA02, AA03, AA04 and AA05 are closed and all other holes (T, P, etc.) are closed too.
8. Pressurise the vessel to 1.04 bara via valve 500AA09 using compressed air.
9. Leak-test all seals and valves with leak detection spray.
10. Release pressure with valve 500AA09.

Assemble the thermostated box

The stepwise procedure to assemble the thermostated box is as follows:

1. Work with two persons.
2. Make sure all T and P measurements are properly installed and valves AA04 and AA05 are closed.

3. Place the heater and connect it through the cable slot. Lead all other cables through the cable slot.
4. Attach the two removable side walls to the fixed walls using the clamps.
5. Lift the roof onto the box and fix it with the clamps.

Starting a measurement session

The stepwise procedure to start a measurement session is as follows:

1. Make sure valve AA09 is open. All other valves except AA01 closed.
2. Start the circulation fans and set the box temperature to the required value.
3. Start at a box temperature ~5^{0}C higher than the FG temperature, check the demister core temperature (TC05). If the demister is warmer than the flue gas, no condensation will occur.
4. Meanwhile, make sure the condensate receiving system (500BB02) is prepared (see section condensate receiver).
5. On the benchlink computer, start a new measurement, starting with the date (yymmdd – name).
6. If the demister is on temperature, open valves AA02, then open valve AA03, then close valve AA01.
7. After this the thermostated box can be set to the flue-gas temperature plus xx°C. (the xx has to be determined in an experiment without filter-candle.)

Condensate receiver

1. Condensate from the three condensate collection points (500WS01, 500WS02 and above valve 500AA01) flows into vessel 500BB02 via 3/8” PTFE lines. A water-lock for 0.03 bar DP is foreseen.
2. Before starting a measurement BB002 can be drained to line BD015 with valve AA10.
3. Upon closing valves AA10 and AA11, liquid is collected in the vessel BB002. Its level can be monitored using level indication tube (PTFE) 500LI01.
4. The amount of condense formed is considerable, so check the level regularly. BE SURE TO OPEN VALVE AA10 AT THE END OF THE DAY, SO THE VESSEL DOES NOT OVERFLOW. To be safe, an overflow line to the flue-gas line is installed.
5. Liquid samples can be taken from valve AA11. Be sure to rinse the line with ~100 ml sample. The rinsing liquid should be disposed as chemical waste (waste barrel available).
6. (Rinsing of vessel BB002 is not foreseen.)

Operation of the BDU during operation

1. Monitor the inlet temperature (500TC01) and adapt the set-point of the thermostated box if necessary.
2. (monitor the level in vessel BB002)
3. Check the temperatures of the box (CT03 and CT04) to check on the performance of the Thermostating (temperature level and difference between top and bottom of box).

4. (If the temperatures are too high, a visual alarm will be shown on the benchlink screen)

1.1.1 Brownian Demister sampling

The Brownian Demister Unit will before the sampling of collected mist droplets be set in off-line modus, which is, disengaged from the absorber exit gas by turning valves 500AA01, 500AA02 and 500AA03 into bypass positions. When this is done the demister housing will be checked for poisonous/toxic gases or the lack of oxygen before any drainage operations are started. If there are any indications of risk, the demister unit housing will be flushed by air until safe values are obtained before it is opened to get access to the BDU. At the same time power to the heating elements should be turned off to ensure that no hot surfaces will be present.

The BDU is equipped with two liquid drainage valves 500AA04 and 500AA05 and those will be opened in order to collect any free and easily drainable liquids. A small overpressure (100 mbar) may be applied to the BDU through 500AA09 to facilitate this operation. Based on the results from this several options on how to proceed may be considered.

If no liquid or only a very small amount of liquid is collected a visual inspection and rinsing/washing of the candle filter should be performed to ensure that even minute amounts of collected mist can be analysed. It may also be necessary to have longer sampling intervals to collect enough sample material.

If larger amounts of liquids are collected and no particulate material is observed and the pressure drop has not exceeded some expected value, there may be no need to actually do any intervention on the BDU.

In order to inspect and rinse the candle filter unit inside the BDU it is necessary to open the top lid on the BDU (see 2.1.5.1 and 2.1.5.2 for details). But before this can be done it is necessary to turn the fog sensors off by shutting of power to the two units.

Then the top of the demister housing may be opened. The top lid of the BDU is then unscrewed and removed. The candle filter unit can then be hoisted out of the BDU tank either manually or by means of a tackle or some other suitable equipment. This will facilitate a thorough inspection of both the inside and outside surfaces of the candle filter. For rinsing/washing, both the inside and outside surfaces of the candle filter is thoroughly wetted and flushed by a known amount of distilled water. All drained liquid is then collected from the outlet sample valves. The candle filter is then lowered back into the BDU and the top lid is the replaced and the housing reassembled (see 2.1.5.1 and 2.1.5.2 for details).

The BDU is then put back into operation by moving the valves back into normal operating positions (see 2.1.5.3 and 2.1.5.5 for details).

All volumes both collected before rinsing/washing and the reclaimed rinsing/washing fluid are measured before storage for later chemical analyses.

The condensate collected in the condensate receiver is measured and a sample is taken for later chemical analysis (see 2.1.5.4 for details).

1.1.2 Tracer addition

The selected tracer components will be added to the amine solution at a suitable place in the pilot plant. The tracer will be inserted in the absorber through a (gas) sampling point. The most likely is valve 200AA608. This valve is situated just above the sump of the absorber and is easy reachable. We will use some rich sample at sampling point 7 (200AA404) to dilute the tracer.

The tracers will be dissolved in fresh/spent amine solution or distilled water in such a concentration that it gives the desired total tracer concentrations. Two inorganic tracer components (X1, Y1) will be added to the amine solution, and two inorganic tracer components (X2, Y2) will be added to the wash water system. See separate memo about tracer selection for further details.

Due to the condensation of the vapours in the washing section, a small amount of the wash water will be returned to the lean solvent and thus tracer migration from the wash section to the solvent will take place. This may be prevented by operational procedures, but may then influence the normal running of the pilot. By carefully monitoring the condensate stream it should be possible to account for this. It will therefore actually be useful to add the two tracers at separate times in order to have a "fresh" tracer distribution for a longer time during the experimental period.

This would then lead to addition of solvent tracer one and wash water tracer one at day one, and then solvent tracer two and wash water tracer two at say day three.

HSE activities

An assessment of fieldwork risks together with a Safe Job Analysis were conducted at TNO premises in Delft 21st September. The associated forms are given in Appendix C.

HSE for the operation of the pilot plant is covered by earlier documentation provided by TNO.

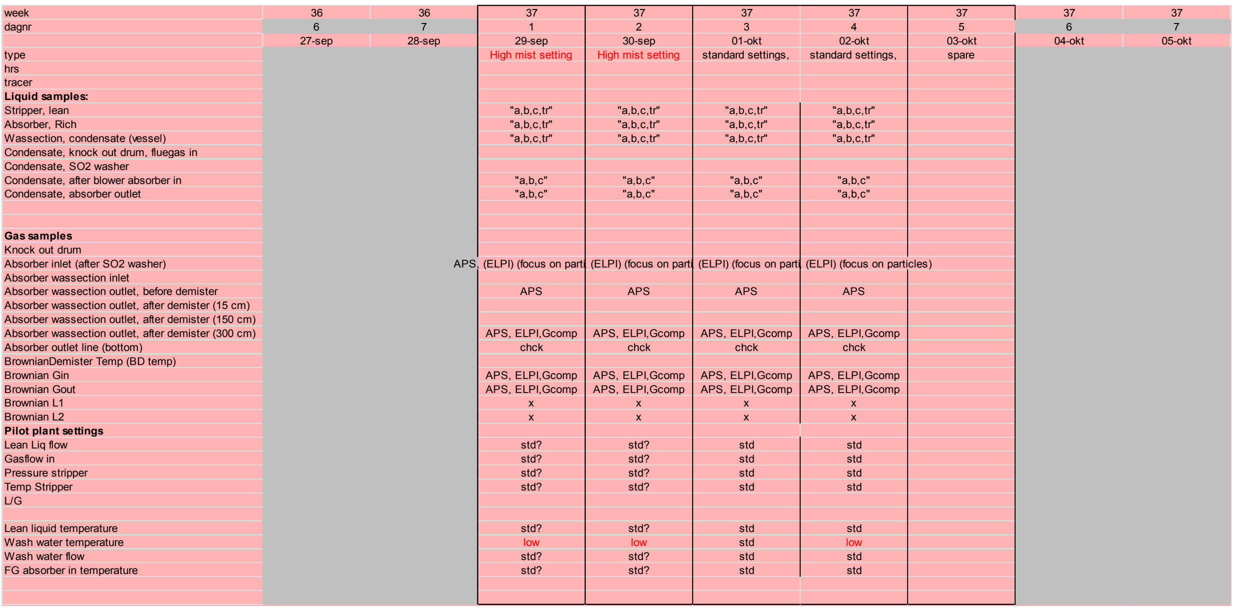
A Appendix: Plan for the experiments

As indicated earlier the following schedule indicates only days with experiments and the indicated dates are not valid as the campaign is 1-2 days delayed. Basically, no experiments will be conducted in the weekends, but if required due to any operational delays it is planned to do some extra experiments on Saturdays (not Sundays).

Preliminary plan for period 1 of the measurement campaign:

week													
dagnr		1	1	1	1	2	2	2	3	3	3	4	5
	#######	2011-09-21	2011-09-21	2011-09-21	2011-09-21	2011-09-22	2011-09-22	2011-09-22	2011-09-23	2011-09-23	2011-09-23	2011-09-24	2011-09-25
type		turn on BDU	check for flow fluctuations	area check	function other points	dependency Tprofile high DT top abs	dependency gas velocity high	dependency 95%cap	dependency 80%cap	dependency cooling water lowT	dependency cooling water: hithT	standard settings, chem analysis	spare
hrs		1	2	2	3	3	3	3	3	3	3		
tracer													add
Liquid samples:													
Stripper, lean						x	x	x	x	x	x	"a,b,c"	
Absorber, Rich						x	x	x	x	x	x	"a,b,c"	
Wassection, condensate (vessel)						x	x	x	x	x	x	"a,b,c"	
Condensate, knock out drum, fluegas in													
Condensate, SO2 washer													
Condensate, after blower absorber in												"a,b,c"	
Condensate, absorber outlet						x	x	x	x	x	x	"a,b,c"	
Gas samples													
Knock out drum													
Absorber inlet (after SO2 washer)					APS						APS, (ELPI) (focus on particles)		
Absorber wassection inlet					APS								
Absorber wassection outlet, before demister					APS	(APS)	APS			APS	APS	APS	
Absorber wassection outlet, after demister (15 cm)													
Absorber wassection outlet, after demister (150 cm)													
Absorber wassection outlet, after demister (300 cm)			APS	APS		APS	APS	APS	APS	APS	APS	PS, ELPI,Gcomp	
Absorber outlet line (bottom)					APS			chck	chck	chck	chck	chck	chck
BrownianDemister Temp (BD temp)		T+x before weeke	T+x	T+x	T+x			end: T-x			end: 'T+x'		
Brownian Gin						x	x	x	x	x	x	PS, ELPI,Gcomp	
Brownian Gout						x	x	x	x	x	x	PS, ELPI,Gcomp	
Brownian L1		chck			x	chck		x	Gcomp			Gcomp	
Brownian L2		chck			x	chck		x					
Lean Liq flow		std	std	std	std	low	90%C	std	std	std	std	std	std
Gasflow in		std	std	std	std	std	high	std	std	std	std	std	std
Pressure stripper		std	std	std	std	low	std	low	high	std	std	std	std
Temp Stripper		std	std	std	std	120	std	std	std	std	std	std	std
L/G													
Lean liquid temperature		std	std	std	std	low	std	std	std	std	std	std	std
Wash water temperature		std	std	std	std	std	std	std	std	low	high	std	std
Wash water flow		std	std	std	std	std	std	std	std	std	std	std	std
FG absorber in temperature		std	std	std	std	std	std	std	std	std	std	std	std

Preliminary plan for period 2 of the measurement campaign



week	36	36	37	37	37	37	37	37	37
dagnr	6	7	1	2	3	4	5	6	7
	27-sep	28-sep	29-sep	30-sep	01-okt	02-okt	03-okt	04-okt	05-okt
type			High mist setting	High mist setting	standard settings,	standard settings,	spare		
hrs									
tracer									
Liquid samples:									
Stripper, lean			"a,b,c,tr"	"a,b,c,tr"	"a,b,c,tr"	"a,b,c,tr"			
Absorber, Rich			"a,b,c,tr"	"a,b,c,tr"	"a,b,c,tr"	"a,b,c,tr"			
Wassection, condensate (vessel)			"a,b,c,tr"	"a,b,c,tr"	"a,b,c,tr"	"a,b,c,tr"			
Condensate, knock out drum, fluegas in									
Condensate, SO2 washer									
Condensate, after blower absorber in			"a,b,c"	"a,b,c"	"a,b,c"	"a,b,c"			
Condensate, absorber outlet			"a,b,c"	"a,b,c"	"a,b,c"	"a,b,c"			
Gas samples									
Knock out drum									
Absorber inlet (after SO2 washer)		APS,	(ELPI) (focus on parti	(ELPI) (focus on parti	(ELPI) (focus on parti	(ELPI) (focus on particles)			
Absorber wassection inlet									
Absorber wassection outlet, before demister			APS	APS	APS	APS			
Absorber wassection outlet, after demister (15 cm)									
Absorber wassection outlet, after demister (150 cm)									
Absorber wassection outlet, after demister (300 cm)			APS, ELPI,Gcomp	APS, ELPI,Gcomp	APS, ELPI,Gcomp	APS, ELPI,Gcomp			
Absorber outlet line (bottom)			chck	chck	chck	chck			
BrownianDemister Temp (BD temp)									
Brownian Gin			APS, ELPI,Gcomp	APS, ELPI,Gcomp	APS, ELPI,Gcomp	APS, ELPI,Gcomp			
Brownian Gout			APS, ELPI,Gcomp	APS, ELPI,Gcomp	APS, ELPI,Gcomp	APS, ELPI,Gcomp			
Brownian L1			x	x	x	x			
Brownian L2			x	x	x	x			
Pilot plant settings									
Lean Liq flow			std?	std?	std	std			
Gasflow in			std?	std?	std	std			
Pressure stripper			std?	std?	std	std			
Temp Stripper			std?	std?	std	std			
L/G									
Lean liquid temperature			std?	std?	std	std			
Wash water temperature			low	low	std	low			
Wash water flow			std?	std?	std	std			
FG absorber in temperature			std?	std?	std	std			

The high mist settings has to be decided based on the results from the first period where the test conditions has been varied to create favourable conditions for mist formation in order to test the performance of the BDU.

Possible variation of pilot plant parameters during the test:

Parameter	unit	low	standard	high
Lean liquid flow	ton/hr	3,5	4	5
Gasflow in	m3/hr	800	900	1000
Stripper pressure	barg	0,9	0,95	1
Stripper temp.	°C	119	120	121

B Appendix

	Flue gas property Gasnova						
	N2	79	mol%				
	CO2	3,4	mol %				
	H2O	6,8	mol%				
	O2	13,8	mol%				
	NOx	3	ppmv				
	NH3	2	ppmv				
	Fluegas EON Maasvlakte				Outside Air		
	N2	70,64	%		N2	78	%
	CO2	12,36	%		CO2	0,04	%
	H2O	12,75	%		H2O	0,7	%
	O2	7	%		O2	21	%
	Sox	60	vppm		NOx		
	NOx	40	vppm		NH3		
Ratio		1				2,666666	
		327,2728				872,7272	Nm3
	Flue gas property SIMULATED Gasnova						
	N2	75,99					
	CO2	3,40					
	H2O	3,99					
	O2	17,18					
	Sox	16,36					
	NOx	10,91					

Calculation showing the simulated Gasnova flue gas composition.

C Appendix

The fieldwork risk assessment form and Safe Job Analysis form are given in the following

SINTEF Materialer og kjemi
SINTEF Materials and Chemistry
Address:
Postboks 4760 Sluppen
NO-7465 Trondheim
NORWAY

Telephone: +47 73593000
Telefax:+47 73597043

info.mk@sintef.no
www.sintef.no/mk
Enterprise /VAT No:
NO 948007029 MVA

Department **Process Technology** **Project no.** 801862

Project implementation period: May-December 2011

Project Manager: Hanne M. Kvamsdal

Participants from SINTEF: Kai Hjarbo, Herman Kolderup, Hanne Kvamsdal

Participants from TNO: Arjen Huizinga, Peter van Os

Client: CCM

Risk assessment carried out by: all participants

Date completed: 2011-09-21

Activity	Potential unwanted incident	Prob-ability (1-5)	Consequence		Risk Value	Suggested mitigating action Remarks
			Environ-mental (A-E)	Personnel (A-E)		
Working at heights	Falling down	1	A	D	D1	Use of the fall protection equipment if necessary.
Dropping of equipment, tools and/or samples from the scaffolding floor	Hit others, spill of chemicals	2	B	C	C2	Wear helmet and PPE. Avoid the possibly exposed area when samples are taken
Flue gas leakage	Exposure of flue gas by leakage in flences and demister vessel etc.	2	A	B	B2	Working outside, quick dilution, possible closing of valves
Solvent leakage	Exposure of chemicals (up to 30 wt% MEA)	1	C	C	C1	Wear PPE. Possible closing of valves, spill tray will reduce the concequence

Probability	**Consequence**	**Risk value**
1. Very low	A. Very low	Consequence & Probablility (t.ex. “C4”, see risk matrix)
2. Low	B. Low	Individual index values in the red zone of the risk matrix

3. Moderate risk	C. Moderate	indicate that the project cannot be continued unless the
4. High	D. Serious	is reduced by taking mitigating actions.
5. Very high	E. Very serious	

SJA title: Emission measurements at the Maasvlakte pilot plant for CO_2 capture		
Date:2011-09-21		Place: TNO office in Delft
Enter cross on completion of checklist:	x	

Participants:		
Arjen Huizinga (TNO)	Kai Hjarbo (SINTEF)	
Peter van Os (TNO)	Hanne M. Kvamsdal (SINTEF)	
Herman Kolderup (SINTEF)		
Person responsible for completing SJA:	Hanne M. Kvamsdal (SINTEF)	

Description of work: (What and how?)

Sampling (gas/liquid) at the Maasvlakte pilot plant for CO_2 capture (owned by TNO). The plant is located at a coal power plant owned by EON. A slip stream of the flue gas from the power plant is treated in the pilot so there is a direct link. TNO personnel will also be present at site.

Risks associated with the work:

Some climbing in fixed ladders (height above platform up to 2m, height above ground is 20 m).

Dropping of equipment and tools from the scaffolding floor.

Flue gas leakage, solvent leakage.

Protection/safeguards: (safety measures plan, see following page)

PPE and rubber plastic gloves, fall protection set (one), oxygen and or CO detectors, no specific requirements related to the clothing, but the body needs to be covered, safety eye wash bottles available

Conclusion/remarks:

It is not regarded as very risky and there are very experienced persons involved. All necessary precautions are taken to do a safe job.

Recommendation/approval:	**Date/Signature:**	**Recommendation/approval:**	**Date/Signature:**
Person responsible for completing SJA:	2011-09-21	Area Officer:	
Person responsible for carrying out the work		Other (post):	

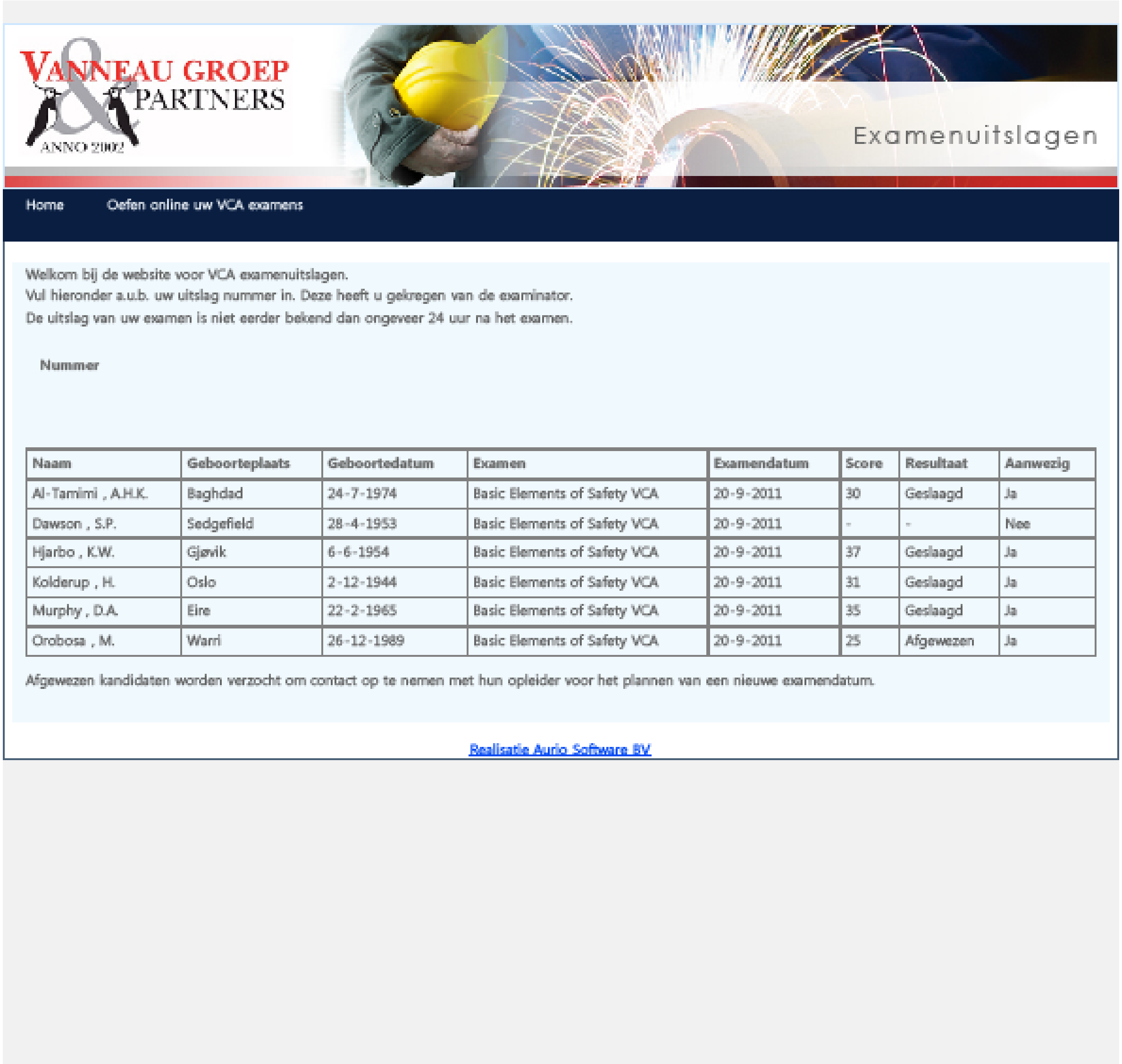
HSE factor	**Yes**	**No**	**N/A**	**Remarks / actions**	**Pers. resp.**
Documentation, experience, skills/expertise					
Familiar task/operation?	x			Done earlier at several similar plants	
Knowledge of experience/unwanted incidents in connection with similar tasks/operations?		x			
Requisite personnel?		x			

Communication and coordination					
Potential conflict with other tasks/operations?	x			Pilot plant is dependent on the power plant operation	
Handling of a possible incident (alarm, evacuation)?	x			Follow the EON procedures	
Requirement for additional supervision?		x			
The workplace					
Unaccustomed working postures?	x			For Herman and Kai	
Work in tanks, man-holes or similar?		x			
Work in trenches or pits?		x			
Clean and orderly?	x				
Protective equipment in addition to PPE?	x			Oxygen- and/or CO meter	
Weather, wind, visibility, illumination, ventilation?		x		Outside, may be windy and raining, but partly covered with a plastic cover	
Use of scaffolding/lifts/harnesses/slings?	x			The scaffolding has fences, one fall protection set is available at site.	
Work at height?	x			Scaffolding is used for the measurements	
Ionising radiation?		x			
Evacuation routes OK?	x			Provided by EON	
Chemical hazards					
Use of harmful/toxic/corrosive chemicals?	x			30 wt% MEA. Using gloves for the chemical handling	
Use of potentially inflammable or explosive chemicals?		x			
Should the chemicals be approved?	x			Some trace elements (Sr, Ba, etc.) may need to be approved, but MEA and sulfamic acid have already been approved	
Biological material?		x			
Dust/asbestos?		x			
Mechanical hazards					
Stability/strength/tension?		x		The scaffolding is tested and approved every month	

Risk from clamping/cutting/striking?		x			
Noise/pressure/temperature?		x		Outside at normal conditions. Some noise from pumps and fans, but ear protection is not required	
Treatment of waste?		x		Normal procedures	
Need for special tools?		x			
Electrical hazards					
Current/voltage/in excess of 1000V?		x			
Shock/stray current?		x			
Loss of current?		x			
The area					
Need for inspection?	x			The site is inspected regularly	
Labelling/signage/cordoning off?	x			EON procedure	
Environmental consequences?		x		Leakage bin provided under the pilot	
Key physical safety systems					
Work on safety systems?		x			
Disconnection of safety systems?		x			
Other					

D Appendix

Examen Uitslagen



Welkom bij de website voor VCA examenuitslagen.
Vul hieronder a.u.b. uw uitslag nummer in. Deze heeft u gekregen van de examinator.
De uitslag van uw examen is niet eerder bekend dan ongeveer 24 uur na het examen.

Nummer

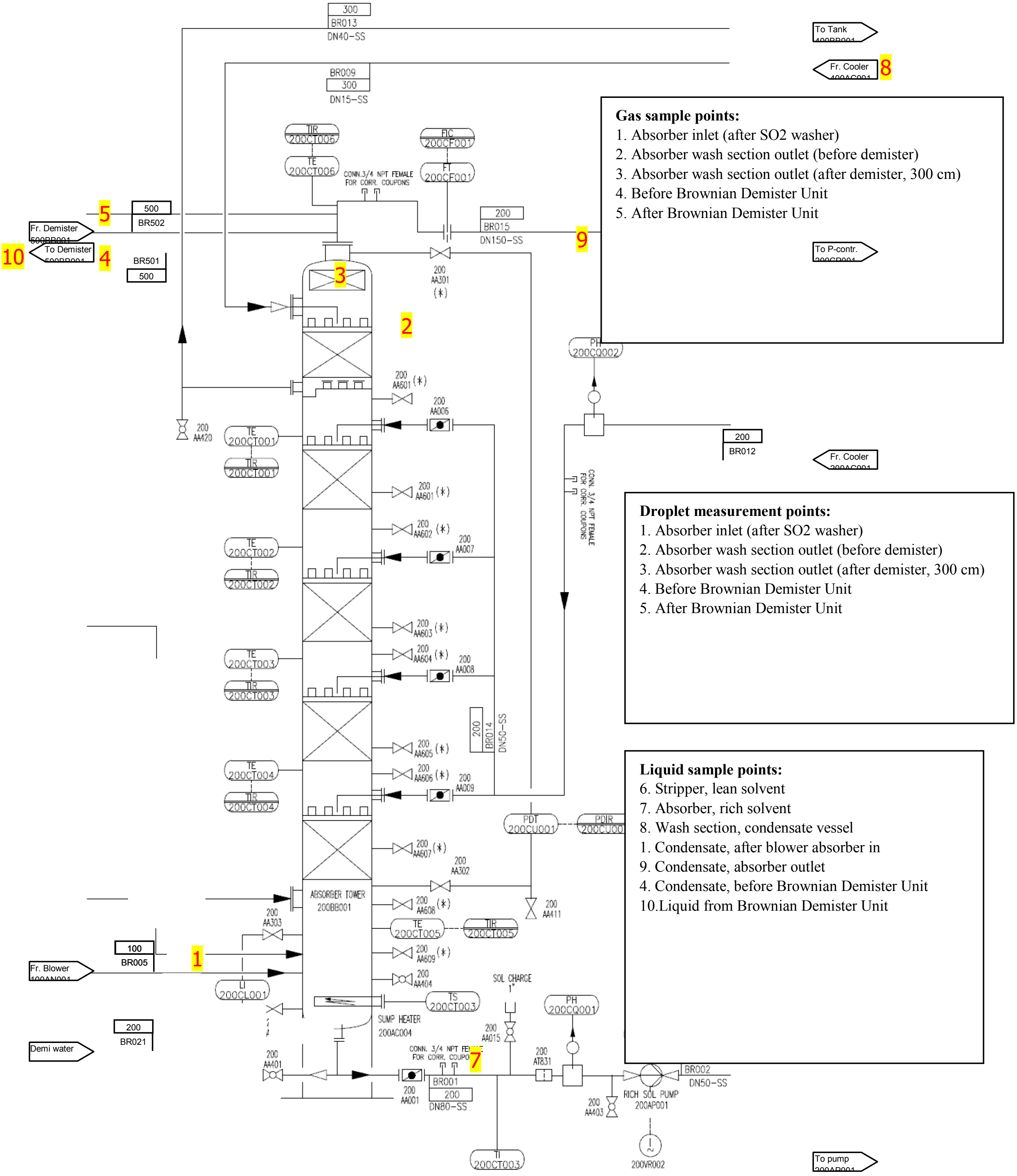
Naam	**Geboorteplaats**	**Geboortedatum**	**Examen**	**Examendatum**	**Score**	**Resultaat**	**Aanwezig**
Al-Tamimi , A.H.K.	Baghdad	24-7-1974	Basic Elements of Safety VCA	20-9-2011	30	Geslaagd	Ja
Dawson , S.P.	Sedgefield	28-4-1953	Basic Elements of Safety VCA	20-9-2011	-	-	Nee
Hjarbo , K.W.	Gjøvik	6-6-1954	Basic Elements of Safety VCA	20-9-2011	37	Geslaagd	Ja
Kolderup , H.	Oslo	2-12-1944	Basic Elements of Safety VCA	20-9-2011	31	Geslaagd	Ja
Murphy , D.A.	Eire	22-2-1965	Basic Elements of Safety VCA	20-9-2011	35	Geslaagd	Ja
Orobosa , M.	Warri	26-12-1989	Basic Elements of Safety VCA	20-9-2011	25	Afgewezen	Ja

Afgewezen kandidaten worden verzocht om contact op te nemen met hun opleider voor het plannen van een nieuwe examendatum.

Realisatie Aurio Software BV

http://www.vcauitslag.nl/[2011-09-21 16:24:26]

Appendix 4: Measurement points



Gas sample points:

1. Absorber inlet (after SO2 washer)
2. Absorber wash section outlet (before demister)
3. Absorber wash section outlet (after demister, 300 cm)
4. Before Brownian Demister Unit
5. After Brownian Demister Unit

Droplet measurement points:

1. Absorber inlet (after SO2 washer)
2. Absorber wash section outlet (before demister)
3. Absorber wash section outlet (after demister, 300 cm)
4. Before Brownian Demister Unit
5. After Brownian Demister Unit

Liquid sample points:

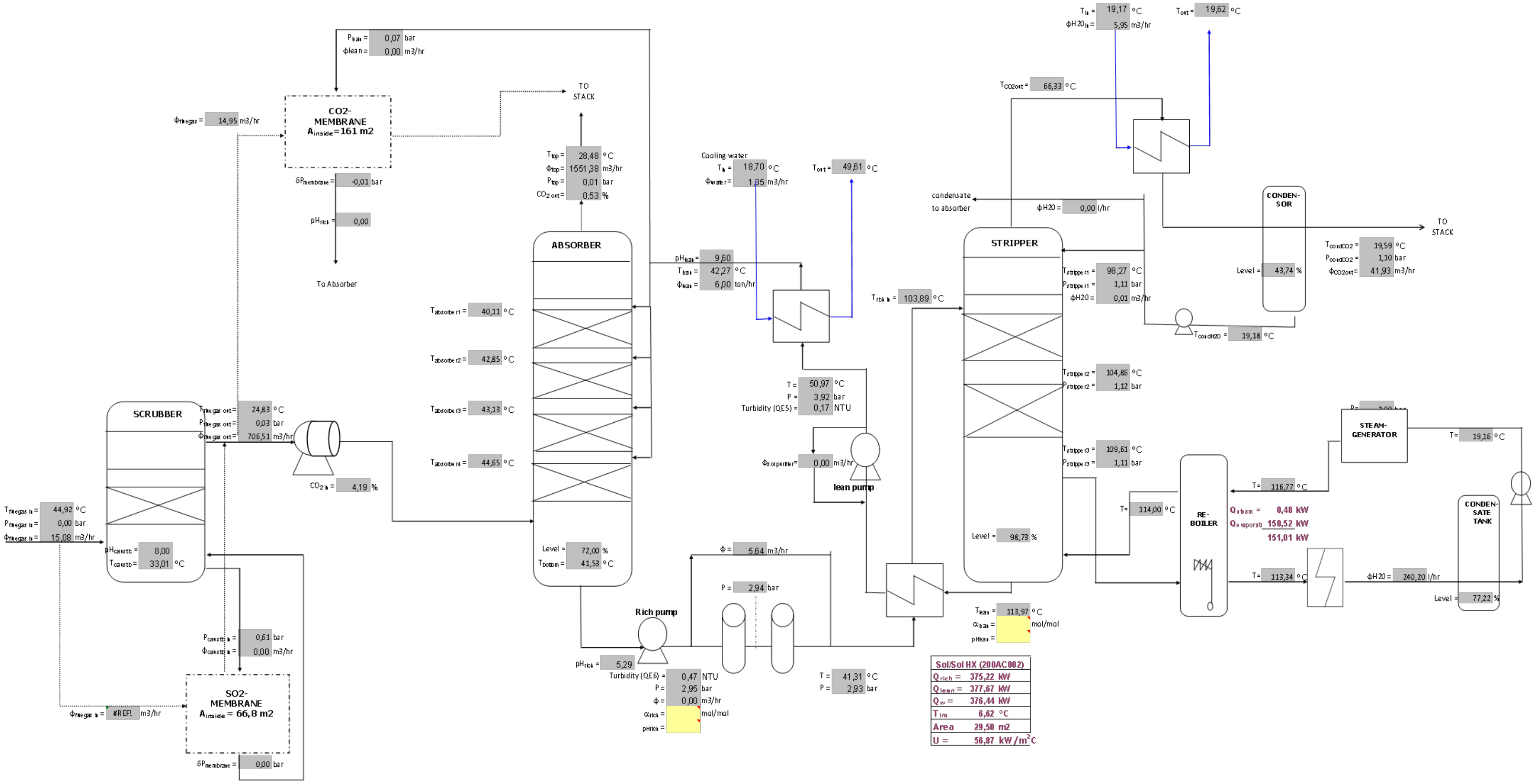
6. Stripper, lean solvent
7. Absorber, rich solvent
8. Wash section, condensate vessel
1. Condensate, after blower absorber in
9. Condensate, absorber outlet
4. Condensate, before Brownian Demister Unit
10.Liquid from Brownian Demister Unit

Appendix 5: Description of tags in PID

Tag	Name
GENERIC\KWT_MAIN_LINE_MEAS_REAL	
SEC100\FT100CF001_MEAS_REAL	FLUE GAS FLOW SCRUBBER INLET
SEC100\FT100CF002_MEAS_REAL	FLUE GAS FLOW SO2 MEMBRANE INLET
SEC100\FT100CF004_MEAS_REAL	FLUE GAS FLOW ABSORBER INLET
SEC100\FT100CF005_MEAS_REAL	CAUSTIC FLOW SO2 MEMBRANE INLET
SEC100\FT100CF006_MEAS_REAL	FLUE GAS FLOW CO2 MEMBRANE INLET
SEC100\PDT100CP001_MEAS_REAL	FLUE GAS DELTA P ON THE SO2 MEMBRANE
SEC100\PDT100CP002_MEAS_REAL	FLUE GAS DELTA P ON THE CO2 MEMBRANE
SEC100\PH100CQ001_MEAS_REAL	PH SCRUBBER SOLUTION
SEC100\PT100CP001_MEAS_REAL	FLUE GAS INLET PRESSURE
SEC100\PT100CP002_MEAS_REAL	CAUSTIC SO2 MEMBRANE INLET PRESSURE
SEC100\PT100CP003_MEAS_REAL	FLUE GAS SCRUBBER OUTLET PRESSURE
SEC100\QE100CQ001_MEAS_REAL	FLUE GAS ABSORBER INLET CO2 ANALYZER
SEC100\TE100CT001_MEAS_REAL	FLUE GAS INLET TEMPERATURE
SEC100\TE100CT002_MEAS_REAL	SCRUBBER SOLUTION TEMPERATURE
SEC100\TE100CT003_MEAS_REAL	FLUE GAS SCRUBBER OUTLET TEMPERATURE
SEC200\FT200CF001_MEAS_REAL	FLUE GAS FLOW ABSORBER OUTLET
SEC200\FT200CF002_MEAS_REAL	RICH SOLUTION FLOW
SEC200\FT200CF003_MEAS_REAL	FILTER BYPASS FLOW
SEC200\FT200CF004_MEAS_REAL	LEAN SOLUTION FLOW
SEC200\FT200CF005_MEAS_REAL	LEAN SOLUTION FLOW (FILETR BYPASS)
SEC200\FT200CF006_MEAS_REAL	COOLING WATER OUTLET FLOW HX SOL/H20
SEC200\FT200CF007_MEAS_REAL	LEAN SOLUTION LMC INLET FLOW
SEC200\LT200CL001_MEAS_REAL	ABSORBER SUMP LEVEL
SEC200\PDT200CU001_MEAS_REAL	ABSORBER DELTA P
SEC200\PH200CQ001_MEAS_REAL	PH AFTER THE ABSORBER (RICH SOL.)
SEC200\PH200CQ002_MEAS_REAL	PH BEFORE ABSORBER AND CO2 MEMBRANE (LEAN SOL.)
SEC200\PH200CQ003_MEAS_REAL	PH AFTER THE CO2 MEMBRANE (RICH SOL.)
SEC200\PT200CP001_MEAS_REAL	FLUE GAS ABSORBER OUTLET PRESSURE
SEC200\PT200CP002_MEAS_REAL	LEAN SOLUTION CO2 MEMBRANE INLET PRESSURE
SEC200\PT200CP003_MEAS_REAL	PRESSURE BEFORE 1.st FILTER
SEC200\PT200CP004_MEAS_REAL	PRESSURE AFTER LEAN PUMP
SEC200\PT200CP006_MEAS_REAL	PRESSURE AFTER 2.nd FILTER
SEC200\PT200CP007_MEAS_REAL	PRESSURE AFTER 1.st FILTER
SEC200\QE200CQ001_MEAS_REAL	FLUE GAS ABSORBER OUTLET CO2 ANALYZER
SEC200\QE200CQ005_MEAS_REAL	LEAN SOLUTION TURBIDITY METER
SEC200\QE200CQ006_MEAS_REAL	RICH SOLUTION TURBIDITY METER
SEC200\QE200CQ007_MEAS_REAL	CO2 MEMBRANE OUTLET CO2 ANALYZER

SEC200\TE200CT001_MEAS_REAL	ABSORBER 1st BED TEMPERATURE
SEC200\TE200CT002_MEAS_REAL	ABSORBER 2nd BED TEMPERATURE
SEC200\TE200CT003_MEAS_REAL	ABSORBER 3rd BED TEMPERATURE
SEC200\TE200CT004_MEAS_REAL	ABSORBER 4th BED TEMPERATURE
SEC200\TE200CT005_MEAS_REAL	ABSORBER SUMP TEMPERATURE
SEC200\TE200CT006_MEAS_REAL	FLUE GAS ABSORBER OUTLET TEMPERATURE
SEC200\TE200CT007_MEAS_REAL	COOLING WATER OUTLET TEMPERATURE HX SOL/H20
SEC200\TE200CT008_MEAS_REAL	COOLING WATER INLET TEMPERATURE HX SOL/H20
SEC200\TE200CT009_MEAS_REAL	RICH SOLUTION TEMPERATURE BEFORE HX
SEC200\TE200CT010_MEAS_REAL	RICH SOLUTION TEMPERATURE STRIPPER INLET
SEC200\TE200CT011_MEAS_REAL	LEAN SOLUTION TEMPERATURE BEFORE HX
SEC200\TE200CT012_MEAS_REAL	LEAN SOLUTION TEMPERATURE BEFORE COOLER
SEC200\TE200CT013_MEAS_REAL	LEAN SOLUTION TEMPERATURE ABSORBER INLET
SEC300\FT300CF001_MEAS_REAL	COOLED CO2 FLOW
-	Cooled CO2 flow, corrected for pressure
SEC300\FT300CF002_MEAS_REAL	CONDENSATE FLOW (TO STRIPPER)
SEC300\FT300CF003_MEAS_REAL	CONDENSATE FLOW (TO ABSORBER)
SEC300\FT300CF004_MEAS_REAL	COOLING WATER INLET FLOW HX CO2
SEC300\FT300CF005_MEAS_REAL	REBOILER CONDENSATE FLOW
SEC300\LIT300CL002_MEAS_REAL	KO DRUM LEVEL
SEC300\LT300CL001_MEAS_REAL	STRIPPER SUMP LEVEL
SEC300\LT300CL003_MEAS_REAL	CONDENSATE TANK LEVEL
SEC300\PT300CP001_MEAS_REAL	STRIPPER OUTLET PRESSURE
SEC300\PT300CP002_MEAS_REAL	STRIPPER 1.st BED PRESSURE
SEC300\PT300CP003_MEAS_REAL	STRIPPER 2.nd BED PRESSURE
SEC300\PT300CP004_MEAS_REAL	COOLED CO2 PRESSURE
SEC300\PT300CP005_MEAS_REAL	STEAM GENERATOR PRESSURE
SEC300\TE300CT001_MEAS_REAL	STRIPPER WASHING SECTION TEMPERATURE
SEC300\TE300CT002_MEAS_REAL	STRIPPER 1.st BED TEMPERATURE
SEC300\TE300CT003_MEAS_REAL	STRIPPER 2.nd BED TEMPERATURE
SEC300\TE300CT004_MEAS_REAL	REBOILER OUTLET TEMPERATURE
SEC300\TE300CT005_MEAS_REAL	REBOILER CONDENSATE OUTLET TEMPERATURE
SEC300\TE300CT006_MEAS_REAL	REBOILER STEAM INLET TEMPERATURE
SEC300\TE300CT007_MEAS_REAL	CONDENSATE TANK TEMPERATURE
SEC300\TE300CT008_MEAS_REAL	CO2 OUTLET TEMPERATURE
SEC300\TE300CT009_MEAS_REAL	COOLED CO2 TEMPERATURE
SEC300\TE300CT010_MEAS_REAL	COOLING WATER INLET TEMPERATURE HX CO2
SEC300\TE300CT011_MEAS_REAL	COOLING WATER OUTLET TEMPERATURE HX CO2
SEC300\TE300CT012_MEAS_REAL	CONDENSATE TEMPERATURE

Appendix 6: Flowsheet with measurement points



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