

ION Final Project Report National Carbon Capture Center Pilot Testing

Project Award No. DE-FE0031727

"Validation of Transformational CO₂ Capture Solvent Technology with Revolutionary Stability"

SUBMITTED BY

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Acknowledgement:

This material is based upon work supported by the Department of Energy Award Number DE-FE0031727

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Executive Summary

As part of ION Clean Energy's (ION) continuous solvent development, ION has completed a six-month testing campaign with ICE-31, at the National Carbon Capture Center (NCCC) as part of U.S. Department of Energy project DE-FE0031727 "Validation of Transformational CO₂ Capture Solvent Technology with Revolutionary Stability". The objective for the project was to scale up ICE-31 from the bench scale to the pilot scale in an industrially relevant environment.

ICE-31 has previously been tested in lab and small-scale studies demonstrating its unique physical and chemical properties that result in low energy requirements and exceptional solvent stability. Looking to further validate those results, the ICE-31 campaign at NCCC operated for over 4,000 hours between March and October of 2021, where the program was designed to include parametric and long-term steady-state testing using natural gas combined-cycle (NGCC) surrogate flue gas (4.4% CO₂), real natural gas-fired boiler gas (7.8% CO₂), and real coal-fired flue gas (13% CO₂).

Using a simple stripper configuration at the PSTU, ION demonstrated at least 95% CO₂ capture on all three flue gases. With NGCC flue gas, ION increased to 98% capture and observed an increase in the Specific Reboiler Duty (SRD) by 2-3%. Using the PSTU heat-integrated stripper ("Advanced Flash Stripper" or AFS) configuration, ION demonstrated a minimum SRD of 2.6 GJ/tCO₂ at 91% CO₂ capture for NGCC flue gas with a slight increase to 2.7 GJ/tCO₂ at 97% capture. With the AFS configuration testing coal-fired flue gas, ION achieved an SRD of 2.5 GJ/tCO₂ at 91% CO₂ capture.

In addition to validation of key performance indicators, a key focus of the test campaign was validation of ICE-31 in Optimized Gas Treating ProTreat[®] process simulator. The model predicted SRDs with an average error of $0.4\% \pm 1.7\%$, utilizing default parameters for all heat and mass-transfer equipment, lending confidence to ION's ability to scale to a commercial facility with minimal process risk. ProTreat[®] modeling indicated that when utilizing ION's advanced heat-integrated stripper configuration and ICE-31, optimization of process design for typical U.S. facilities could expect SRDs of 2.6 GJ/tCO₂ for NGCC and 2.4 GJ/tCO₂ for coal-fired flue gas at 95% CO₂ capture. Increasing these designs to achieve 99% CO₂ capture in a path towards carbon neutrality has a minimal impact on capture cost relative to the 95% CO₂ capture case.

After more than 1,200 hours of parametric testing, ION executed a long-term test on NGCC gas at 95% CO_2 capture for 1,500 hours. PSTU operation was stable and reliable with over 99% uptime and no solvent addition. Extractive sampling after the water wash for NH₃ and solvent were below 1 ppm and 0.04 ppm, respectively, indicating very low emissions.

In conclusion, the campaign at NCCC further validated ION's expectations related to solvent stability, capture performance and suitability for post-combustion CO₂ capture from natural gas-fired facilities, including those with high oxygen environments.



Table of Contents

ION FINAL PROJECT REPORT NATIONAL CARBON CAPTURE CENTER PILOT TESTING Acknowledgement: Disclaimer:	1 2 2
EXECUTIVE SUMMARY	3
INTRODUCTION	6
TEST PLAN OVERVIEW	7
METHODOLOGY Overview of PSTU Standard Operations Testing and Results Methodology	8 8 9
RESULTS AND DISCUSSION	12
Parametric Testing with NGCC Flue Gas	12
Parametric Testing with Undiluted Natural Gas-Fired Boiler Flue Gas	15
Parametric Testing with Coal Flue Gas	17
ProTreat [®] Model Validation	18
Long-Term Steady State Testing	21
Heat Stable Salt Analysis	24
CONCLUSION	26
APPENDIX A: ABBREVIATIONS	27
APPENDIX B: CALCULATIONS FOR CAPTURE EFFICIENCY	28
CO ₂ Capture Method 1:	28
CO ₂ Capture Method 2:	29



Table of Figures

Figure 1: NCCC's PSTU (center and right) and the new gas-fired boiler (left); photo courtesy of NCCC	6
Figure 2: Process Flow Diagram for ICE-31 operation at the PSTO	8
Figure 3: Capture efficiency variation during heat-loss testing due solely to ambient condition changes	.10
Figure 4: Optimal SRD at PSTU: 95% CO ₂ capture with 4.4% inlet CO ₂ and 18 m of MP252.Y absorbe	۶r
packing	13
Figure 5: SRD at optimal L/G for varying capture efficiencies with 4.4% inlet CO2	14
Figure 6: SRD at optimal process conditions for varying capture efficiencies utilizing both the Simple	
Stripper (squares) and Advanced Flash Stripper (triangles) with 4.4% inlet CO ₂	15
Figure 7: Optimal SRD at PSTU: 95% CO ₂ capture with 7.2% inlet CO ₂ , 18 m of MP252.Y absorber	
packing	16
Figure 8: SRD at optimal L/G for varying capture efficiencies with 7.5% inlet CO ₂	
Figure 9: Optimal SRD at PSTU: 95% CO ₂ capture with 11% inlet CO ₂ and 18 m of absorber packing	17
Figure 10: ProTreat® process model for simple stripper validation	19
Figure 11: ProTreat® process model validation versus empirical results for SRD	20
Figure 12: DroTreat® process model validation for other empirical results	20
Figure 12: Figure 12: Tetal CO. contured during lang targe stoody state energies.	2 1
Figure 13: Total CO_2 captured during long-term steady state operation	22
Figure 14: Comparison of Total Captured CO ₂ over Expected Captured CO ₂	22
Figure 15: SRD during long-term operations; spikes in SRD correspond to suspected steam flowmeter	
miscalibration	23
Figure 16: Capture efficiency for long-term operation (6-hr average)	23
Figure 17: Total solvent component mass balance over the 1500 hour long-term campaign with no	
reclaiming or solvent addition. Mass balance includes active components, water, and CO2	24
Figure 18: HSS accumulation throughout test campaign. Note: the dips in concentration on 7/28 are du	Je
to replacement of solvent with fresh solvent upon change from natural gas-focused to coal-focused flu	е
gas testing	25
5 5	-



Introduction

Carbon Capture Utilization and Storage (CCUS) technologies continue to be of great interest to pointsource emitters as mechansims to reduce their carbon footprint. Post-combustion CO₂ capture (PCCC) is an affordable decarbonization technology today compared to other carbon removal technologies such as direct air capture. However, the effective and widespread use of CCUS today is limited almost exclusively because of economic considerations. There has been increased interest in the United States over the past few years with the announcement of tax credits for CCUS deployment which help to offset costs but, a key focus of technology developers is to reduce capital costs (CAPEX) and operating costs (OPEX) to create a successful business case to support deployment on a large-scale, commercial level.

ION Clean Energy, Inc. (ION) is developing and deploying solvent-based CO₂ capture technologies with aims of capturing 1 billion tonnes of CO₂ by 2050 to decarbonize the electrical grid and carbon-intensive industries. As part of its continuous development of solvent-based capture systems, ION completed a sixmonth testing campaign for its third-generation solvent technology, ICE-31, at the National Carbon Capture Center (NCCC) (Figure 1) as part of U.S. Department of Energy project DE-FE0031727 "Validation of Transformational CO₂ Capture Solvent Technology with Revolutionary Stability." The objective for the project was to scale up ICE-31 from the bench scale to the pilot scale in an industrially relevant environment.

This report covers the results and conclusions of ION's test campaign at the NCCC's Pilot Solvent Test Unit (PSTU) using ICE-31. ION is the first technology developer to test at the PSTU using the test facility's newly configured natural gas-fired boiler as part of its "Apollo" project. The test results from this Apollo pilot test program demonstrate a viable reduction in both capital and operating expenses to support large-scale carbon capture deployment within the next decade.



Figure 1: NCCC's PSTU (center and right) and the new gas-fired boiler (left); photo courtesy of NCCC



Test Plan Overview

ION tested ICE-31 at NCCC from March 2021 through October 2021. Prior to testing, a detailed test plan was provided by ION to the National Carbon Capture Center with the following primary objectives for technological development:

- Validate the Optimized Gas Treating (OGT) Process Model for all key performance indicators; including utility requirements and process paramaters required for commercial scale plant designs
- Confirm expected solvent stability as a function of tonnes of CO₂ captured at ideal operations with NGCC or coal flue gas
- Verify chemical reaction kinetics of the solvent at various liquid flow rates and packing heights as a function of partial CO₂ pressure
- Determine the effect of the water wash sections on the emissions profile
- Determine process stability to CO₂ emitter base plant upsets such as high contaminants, high flue gas temperature, low flue gas temperature, etc.
- Investigate system response to transient and ramped operation to determine suitability for commercial applications such as power facilities who are commercially dispatched
- Assess benefits of advanced stripper heat integration such as the cold-rich bypass or the advanced flash stripper (AFS)
- Identify optimum operating profile to determine key performance indicators for use in technoeconomic analysis

The test plan consisted of a series of work packages aimed at addressing each of these objectives. Due to COVID-19 travel restrictions, all operations and on-site monitoring was performed by the NCCC team. Remote monitoring and operational setpoints were provided by ION to complete the test plan.



Methodology

Overview of PSTU Standard Operations

The Pilot Solvent Test Unit (PSTU) is a 0.5 MWe CO₂ capture pilot unit at NCCC. The primary flue gas utilized for the test campaign was provided from a natural gas packaged boiler and has a concentration of roughly 7-8% CO₂ which can then be cooled and diluted with air to NGCC flue gas CO₂ content (4.4% CO₂) prior to introduction to the PSTU absorber. Pre-treated coal-derived flue gas was also used for tests at about 12% CO₂ supplied by the host site E.C. Gaston coal-fired power station. The absorber contains three 6-meter beds of Sulzer MellapakTM 252Y structured packing. The lean solvent travels down the column, absorbing CO₂ while the flue gas flows in a counter-current direction up the column. Due to the exothermic reaction, the flue gas at the top of the column is significantly warmer than the inlet flue gas. Thus, a water wash vessel cools the flue gas to within a few degrees of inlet flue gas temperatures and restores water balance via recirculating, cooled wash water. The CO₂-lean flue gas exits the PSTU through an NCCC or Gaston stack for release to atmosphere, depending on sources of flue gas used.

Exiting the bottom of the absorber, the CO_2 -rich solvent gravity-flows into a buffer tank. The rich solvent is then pumped through the lean-rich cross exchanger where it exchanges heat with the lean solvent and enters the top of the regenerator. The rich solvent flows down the regenerator, releasing CO_2 and absorbing water vapor while CO_2 and water vapor from the reboiler flow up the column. The semi-lean solvent falls to the stripper sump and recirculates through a forced-convection reboiler, which utilizes steam to heat the solvent and removes CO_2 to achieve lean loading. The lean solvent is then recirculated back to the absorber through the lean-rich heat exchanger for further CO_2 capture. The CO_2 out of the top of the stripper is cooled and then released to the atmosphere via the stack (Figure 2).



Figure 2: Process Flow Diagram for ICE-31 operation at the PSTU



PSTU Modifications for ION Testing

Before testing, ION requested several modifications to the PSTU:

- Installation of a Sulzer distributor and packing system designed specifically for solvent absorption in carbon capture water washes. This bed allowed for countercurrent gas-liquid mass transfer of solvent vapors into the water wash solution which significantly reduces solvent emissions.
- A bypass line feeding the regenerator from the cold-rich solvent upstream of the lean/rich heat exchanger. This cold rich bypass (CRB) was split fed to the regenerator with a portion entering above the top bed and with the main flow entering above the bottom bed.
- ION's Multi-Component Liquid Analyzer (MLA) was installed on the cold lean solvent feed (immediately upstream of the absorber) to provide continuous, real-time analysis of all major solvent components including water and CO₂.

PSTU Advanced Flash Stripper Operations

In some of the work packages, ION opted to use the existing Advanced Flash Stripper (AFS) configuration which is similar to the simple regenerator but with an incoproated heat exchange system. In the AFS, the rich solvent from the buffer tank is first split so a portion goes to a gas/liquid heat exchanger to exchange heat from the hot CO₂ out of the stripper. Similarly, after the first lean-rich heat exchanger, the other portion of warm-rich solvent bypasses the heat exchanger and enters directly in the top of the stripper. Lean solvent exchanges heat with the remaining portion of rich solvent. Finally, the hot-rich solvent passes through a once-through forced convection steam heater and then flashes into the bottom of the stripper. When operating the AFS on the PSTU, the absorber and water wash operations are identical to the simple regenerator operation.

Testing and Results Methodology

Measuring Specific Reboiler Duty (SRD)

Over the first two months of testing, ION focused on parametric testing where 70 steady state-test points were measured. Each data point was recorded once both the SRD and capture efficiency were constant within 1% over the course of 30 minutes. The most important output from the parametric data was the Specific Reboiler Duty (SRD), defined by Equation 1.

The reboiler heat duty was calculated from the overall flow of steam (\dot{m}_{steam}) into the reboiler multiplied by the enthalpy differential between the steam conditions into the reboiler and the condensate conditions coming out (Equation 2).

Reboiler Heat
$$Duty[=] \frac{GJ}{hr} = \dot{m}_{Steam}(H_{steam} - H_{condensate})$$
 Eq 2

The captured CO_2 was measured on the absorber side of the process as the difference between CO_2 going in and coming out of the absorber (Equation 3), where C represents CO_2 concentrations.

$$CO_2 \ Captured[=]\frac{tonnes}{hr} = \dot{m}_{Flue \ Gas \ in}C_{CO_2 \ in} - \dot{m}_{Flue \ Gas \ Out}C_{CO_2 \ out}$$
 Eq 3

During the campaign ION measured the heat loss for the simple stripper configuration to estimate the ambient heat losses for the PSTU. For this measurement, the simple stripper was operated at standard gas and liquid flows but at a very low steam input. Under these operating conditions, most of the heat is



lost to the atmosphere and only a small portion of the reboiler heat goes towards regenerating the solvent. To isolate the effects of temperature swings throughout the day, the data was analyzed and averaged over two days. Ambient heat loss was calculated at 60 MJ/hr under heat loss conditions, which extrapolates to 80 ± 10 MJ/hr at the standard reboiler temperature. Ambient heat loss accounts for a significant amount of overall heat duty for NGCC flue gases and must be properly considered in Equation 1 above to improve the accuracy of the modeling results. During heat-loss testing, the steam rate was set at a low setpoint and the capture efficiency was therefore lower. With a set steam rate, the capture efficiency was directly observed to fluctuate due to the ambient heat losses without any other process changes for two day/night cycles (Figure 3). Instead of attempting to account for changing ambient conditions, ION utilized an average heat loss term for all SRD calculations knowing this could add up to 1% deviation in SRD depending on the ambient losses.



Figure 3: Capture efficiency variation during heat-loss testing due solely to ambient condition changes

Solvent Analysis

Solvent samples on both the lean and rich side were taken during each test condition throughout the parametric testing and at least three times a week during the long-term, steady-state testing. These samples were analyzed at NCCC's laboratories as well as in ION's laboratory in Boulder, CO.

The major organic components were quantified using an Agilent 7890B gas chromatograph (GC) with a thermal conductivity detector. An Agilent capillary column was used for separation with helium as the carrier gas. A standard calibration curve was constructed using five different concentrations of known standards encompassing the full expected concentration range of components in the samples. All analyses were performed at a minimum in triplicate. Analysis of trace components and decomposition products were performed using an HP 6890 GC coupled with a 5973 mass spectrometer using electron impact ionization. The compounds were separated using a capillary column with helium as the carrier gas. For qualitative analysis, a mass range of 33 to 250 amu was scanned and for quantitative analysis the instrument was set to scan for the highest abundant fragment ion of each species using single ion monitoring. Heat-stable salts (HSS) were qualitatively and quantitatively analyzed using a Dionex ICS-5000+ ion chromatography system with a potassium hydroxide eluent generator and conductivity detector. The anions were separated using a Dionex analytical column and an AERS 500 electrolytically regenerated suppressor was used to eliminate background conductivity. A standard calibration curve was constructed using five different concentrations of known standards encompassing the full expected concentration range of anions in the samples. All analyses were performed in triplicate. The CO₂ loading



of the solvent was determined using a UIC, Inc. CM-5015 coulometer in conjunction with a CM5200 acidification module. The water concentration was determined using a Mettler-Toledo V30 volumetric Karl-Fischer titrator.

Emissions Analysis

The flue gas outlet of the PSTU was equipped with a Gasmet DX4000 hot-gas FTIR. The sample was pulled through a heated line into the FTIR where the spectra was then analyzed for main solvent components as well as degradation products. Both the outlet of the lower water wash and the upper water wash (UWW) were tested during the campaign. Table 1 provides the spectra range for the solvent-specific components analyzed along with the approximate limit of detection in the process gas. Water, CO₂, and NO were also analyzed using their standard wavenumbers. Residuals for the spectra were very low, ranging from 0.001 to 0.003 for the analyzed species.

Component	Wavenumber [cm ⁻¹]	Lowest Calibration Standard [ppm]	Limit of Detection [ppm]
Solvent Component(s)	2700 – 3150	30	~ 3
Ammonia	1650 – 1550	10	~ 2
Formaldehyde		25	~ 2

Table 1: Emissions Analysis on Outlet Flue Gas

Three extractive samples were taken during long-term testing at the UWW outlet and sent to RJ Lee Group for analysis. Solvent components were analyzed via OSHA PV21xx based on the actual compound; the NH₃ was quantified with OSHA ID 188 while formaldehyde was quantified following EPA TO-11A. Extractive samples for NH₃ were about four times lower than the reported FTIR results, showing that NH₃ was typically below the limit of detection throughout long-term testing.



Results and Discussion

Parametric Testing with NGCC Flue Gas

ION analyzed ICE-31 performance over a range of operating conditions with surrogate NGCC flue gas by diluting the on-site boiler flue gas with air by approximately 50%. Table 2 gives the range of conditions and baseline condition for select operating parameters.

Condition	Range	Baseline
Inlet CO ₂ (vol%)	4.3 – 4.5	4.4
Capture Efficiency (%)	78 – 98	95
Absorber Packing Height (m)	12 or 18	18

Table 2: Operating conditions for parametric testing with NGCC flue gas

Optimal L/G at 95% Capture

ION varied the L/G ratio at 95% ± 1% CO₂ capture to determine optimal performance of the solvent at the PSTU using an absorber packing height of 18 m. CO₂ outlet pressure was maintained constant while the lean and rich loading varied with L/G; as the L/G increased, the loading range decreased to maintain the same CO₂ removal. For each setpoint, ION modeled the steam duty needed for 95% capture prior to changing operating conditions. The steam load was then set at a determined flow rate, and the capture efficiency was allowed to reach steady-state. Due to the high accuracy of the ProTreat[®] model, the capture efficiency was almost always within the range of the target capture rate, allowing relatively smooth and quick transitions between setpoints. The optimal lean loading is a balance between increasing carrying capacity of the solvent to limit sensible heat loss in the lean/rich cross exchanger and increasing lean loading in the stripper bottoms to better utilize stripping steam (Figure 4). The wide range for optimal performance allows a more robust design of a large-scale facility without concern that solvent energy performance will be significantly different than the guarantees. The ProTreat[®] model predicted the same optimal range of performance based directly on the equipment at the PSTU.





Figure 4: Optimal SRD at PSTU: 95% CO₂ capture with 4.4% inlet CO₂ and 18 m of MP252. Y absorber packing

SRD versus Capture Efficiency

Another work package was focused on varying the capture efficiency of the PSTU while simultaneously choosing optimal L/G ranges to determine minimum SRD performance at each capture efficiency. Additionally, these tests were completed at two packing heights in the absorber (Figure 5). With 18 m of packing, the CO₂ mass transfer into the solvent was fast enough to reach a similar rich loading across the entire capture efficiency range. The overall impact of the fast kinetics and favorable thermodynamics of ION's ICE-31 solvent demonstrate that there is only a 4% SRD penalty when increasing capture from 90% to 98%. Further optimization with cold-rich bypass would allow even leaner optimal lean loadings and a smaller penalty when increasing capture efficiency. When utilizing 12 m of packing in the absorber, the SRD penalty increases by 14% when increasing capture from 87% to 95%.





Figure 5: SRD at optimal L/G for varying capture efficiencies with 4.4% inlet CO2

Optimal SRD Using the Advanced Flash Stripper (AFS)

Similar to the simple stripper tests, ION ran a series of tests utilizing the Advanced Flash Stripper with varying capture efficiencies at optimal solvent flow and ratios for both the cold-rich and warm-rich bypasses. The AFS significantly outperformed the Simple Stripper across all capture efficiencies with respect to SRD (Figure 6). The AFS operation empirically demonstrated an SRD of 2.7 GJ/tonne CO₂ at 95% CO₂ capture from an NGCC flue gas. With optimized process design including ION's substantially similar heat-integrated regeneration process, ICE-31 can provide an SRD of 2.6 GJ/tonne CO₂ at 95% CO₂ capture from an NGCC flue gas.





Figure 6: SRD at optimal process conditions for varying capture efficiencies utilizing both the Simple Stripper (squares) and Advanced Flash Stripper (triangles) with 4.4% inlet CO₂

Parametric Testing with Undiluted Natural Gas-Fired Boiler Flue Gas

ION analyzed ICE-31 performance over a range of operating conditions with undiluted boiler flue gas, containing a CO_2 content of ~7%, using the PSTU simple stripper to determine optimum performance both on PSTU equipment and for large-scale applications. Table 3 gives the range of conditions and baseline condition for select operating parameters.

Condition	Range	Baseline
Inlet CO ₂ (vol%)	7.2 – 7.4	7.2
Capture Efficiency (%)	74 – 98	95
Absorber Packing Height (m)	12 or 18	18

Table 3: Operating conditions for parametric testing with undiluted natural gas-fired boiler flue gas

Optimal L/G at 95% Capture

Similar to the NGCC flue gas, ION varied the L/G ratio at $95\% \pm 1\%$ CO₂ capture to determine optimal performance with boiler flue gas using the simple stripper and an absorber packing height of 18 m. CO₂ outlet pressure was maintained at 1.9 bara while the lean and rich loading varied with L/G; as the L/G increased, the loading range decreased to maintain the same CO₂ removal. Under these conditions, there was a wide optimal L/G range where the SRD was within 1% of the minimum (Figure 7).





L/G (kg/kg)



SRD versus Capture Efficiency

Similar to the NGCC flue gas testing, ION also varied the capture efficiency for the undiluted boiler flue gas while simultaneously choosing optimal L/G ranges for minimum SRDs. Both 12 m and 18 m absorber heights were tested for each capture efficiency (Figure 8). The overall impact of the fast kinetics and favorable thermodynamics demonstrate that there is only a minor SRD penalty when increasing capture from 90% to 98%. When utilizing 12 m of packing in the absorber, the penalty increases significantly.



Figure 8: SRD at optimal L/G for varying capture efficiencies with 7.5% inlet CO2



Parametric Testing with Coal Flue Gas

ION analyzed ICE-31 performance over a range of operating conditions with coal-derived flue gas from Plant E.C. Gaston using the PSTU simple stripper. Table 4 gives the range of conditions and baseline condition for select operating parameters.

Condition	Range	Baseline
Inlet CO ₂ (vol%)	10.2 – 11.7	11
Capture Efficiency (%)	94 – 96	95
Absorber Packing Height (m)	18	18

Table 4:	Operating	conditions	for	parametric	testing	with	undiluted	boiler	flue	qas
										3

Optimal L/G at 95% Capture

ION again varied the L/G ratio at $95\% \pm 1\%$ CO₂ capture to determine optimal performance with coalderived flue gas using the simple stripper and an absorber packing height of 18 m. CO₂ outlet pressure was constant with L/G (Figure 9). As expected, the coal-derived flue gas had the lowest empirical SRD of all flue gases tested at 2.9 GJ/tCO₂ due to its high CO₂ partial pressure at the bottom of the absorber. However, there was not a significant decrease in SRD over the NGCC and boiler flue gas cases because the solvent achieves a high rich loading with all three flue gas types.



L/G (Kg/Kg)

Figure 9: Optimal SRD at PSTU: 95% CO2 capture with 11% inlet CO2 and 18 m of absorber packing



ProTreat[®] Model Validation

A two-part approach was taken for determining optimum performance of the ICE-31 solvent for technoeconomic analysis. First, the results for the simple stripper cases were thermodynamically and kinetically validated in the OGT's ProTreat[®] process modeling tool.

For this process validation, the empirical data from the simple stripper configuration was compared to a ProTreat[®] simulation that matches the PSTU mass and heat transfer equipment (Figure 10). Packing heights and packing types used the default values built into the ProTreat[®] model, which have been validated directly with Sulzer's test facilities. Table 5 gives the main inputs and outputs from the ProTreat[®] validation models. All parametric test conditions reported above were validated and reported in the ProTreat[®] process models.

Stream #	Input	Output
101 (Dilution Air In)	Flow, Pressure, Temperature, Composition	
100 (Flue Gas In)	Flow, Pressure, Temperature, Composition	
301 (Absorber Rich Outlet)	Flow	Temperature, CO ₂ Loading
205 (Water Wash Recirculation)	Flow	
208 (Water Wash Recirculation)	Temperature	
104 (CO ₂ Out)	CO ₂ Mole Fraction	Solvent Mole Fraction, Temperature
501 (Stripper CO ₂ Outlet)	Pressure	Temperature, Water Content
Absorber		Max Temperature
Stripper		Specific Reboiler Duty (SRD)
403 (Stripper Lean Outlet)		Temperature, CO ₂ Loading

Table 5: Select inputs and outputs for ProTreat® process model validation





Figure 10: ProTreat[®] process model for simple stripper validation



Process Model Fit for SRD

ProTreat[®] models closely validated the NCCC results for SRD over the wide range of flue gas, capture efficiency, and flow rate parametric test conditions. The average error for SRD was 0.4% with a standard deviation of 1.7% (Figure 11). The very close fit proves the robust nature of the thermodynamic and kinetic framework with the ICE-31 proprietary module inside OGT's ProTreat[®]. This imparts strong confidence in ION's ability to model energy performance for large-scale systems and minimizes the risk in utility costs associated with those projects.



ProTreat[®] Process Model Fit for Other Key Performance Indicators

Empirical results from the campaign were also used to validate the process model for ability to accurately predict cooling and pumping loads for a large-scale facility. To determine this, model temperatures and solvent CO₂ capacity at key process points were compared to empirical results. ProTreat[®] was able to simulate process conditions at all points in the process model with extremely good fit. In all cases, the PSTU empirical results are not statistically different than the ProTreat[®] simulated results (Figure 11)





Long-Term Steady State Testing

A critical test during the campaign was long term steady-state testing to determine the overall stability of the ICE-31 solvent. For this test, ION maintained an optimal plant performance with NGCC flue gas for 1,500 hours. The conditions for the long-term operation are shown below (Table 6).

Condition	Value
Inlet CO ₂ (vol%)	4.4
Capture Efficiency (%)	95
Absorber Packing Height (m)	18

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I able	61	Operating	conditions	tor long-term	steady-state
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PSTU on time during the long-term operation was very high with only minor operational upsets. With almost a constant steam input, ION was able to maintain 100% of expected CO₂ capture output throughout the test. Minor shutdowns due to boiler performance were made up by operating at slightly higher than 95% average capture efficiency (Figure 13 & Figure 14).









Figure 14: Comparison of Total Captured CO₂ over Expected Captured CO₂



Capture Efficiency and SRD

Since the steam rate was almost entirely constant throughout the long-term test campaign, the overall SRD at the expected capture efficiency was close to constant over the 1,500 hours of operations (Figure 15). The capture efficiency was also maintained with only minor drops at a target of 95%. Each drop was attributed to an inadvertent change in the steam flowmeter calibration that tended to drift significantly during testing. After recorrecting the steam flowmeter, capture efficiency always returned to its previous point (Figure 16).



Figure 15: SRD during long-term operations; spikes in SRD correspond to suspected steam flowmeter miscalibration





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Long-Term Emissions for Ammonia and Solvent Components

During long-term testing, the emissions of key components were tracked through online monitoring; however, due to the sensitivity of emissions at or below the detection limits of the instrumentation, ION also had extractive sampling performed for more accurate data. This extractive sampling showed that NH₃ emissions were under 1 ppm. Additionally, solvent components in total were below the 40 ppb quantitation limit for the extractive sample.

Active Component Analysis

Lean samples from the long-term campaign were analyzed for all active components, CO_2 , and water to determine the degradation rate of active components. The loss rate for the sum of active components was statistically insignificant with total active concentration varying randomly at $99 \pm 1\%$ of the starting composition (Figure 17). It is important to note that during this test, no solvent make-up or reclamation was performed. While these initial results are quite promising, longer-term testing is necessary to confirm the expected low loss rates for active components via the liquid-side mass balance. ION plans to actively study this through long-term operation of its 10 tonne per day pilot that is currently in construction.



Figure 17: Total solvent component mass balance over the 1500 hour long-term campaign with no reclaiming or solvent addition. Mass balance includes active components, water, and CO₂

Heat Stable Salt Analysis

Heat stable salts (HSS) are typically a result of oxygen and impurities of the flue gas interacting with the solvent. The major HSS observed during this campaign were sulfate, Component A, nitrate, nitrite, and Component B (in order of abundance). Among them, only Component A and Component B are the result of solvent oxidation. Figure 18 shows the HSS content during the entire campaign; the dips in concentration on 7/28 are due to taking a significant sample after the long-term testing on natural gas-derived flue gas and replacement of that solvent with fresh solvent in preparation for testing on coalderived flue gas.





Figure 18: HSS accumulation throughout test campaign. Note: the dips in concentration on 7/28 are due to replacement of solvent with fresh solvent upon change from natural gas-focused to coal-focused flue gas testing

Sulfate (SO_4^{2-}) was the major HSS accumulating in the solvent. It derives from capture of SO_x (mostly SO₂) in the flue gas stream and subsequent oxidation of the intermediary sulfite (SO_3^{2-}) to sulfate.

Nitrites (NO_2) and nitrates (NO_3) are the other major HSS introduced by flue gas impurities. NO_x is a mixture of gases primarily containing NO and NO₂. NO₂ is an acid gas which readily dissolves in alkaline solvents to form a mixture of nitrite and nitrate. The nitrite slowly oxidized to nitrate. The maximum combined nitrite/nitrate concentration was about 250 ppm at the end of the boiler gas testing (end of July) and the accumulation rate decreased thereafter.



Conclusion

ION completed a six-month testing campaign for its third-generation solvent technology, ICE-31, at the National Carbon Capture Center confirming the remarkable performance of the solvent technology. The ICE-31 campaign at NCCC operated for over 4,000 hours including parametric and long-term steady-state testing using natural gas combined-cycle (NGCC) surrogate flue gas (4.4% CO₂), real gas-fired boiler gas (7.8% CO₂), and real coal-fired flue gas (13% CO₂).

ION demonstrated 95% CO₂ capture on all three flue gases, achieved steady-state capture efficiencies of up to 98% capture with NGCC flue gas, and reached over 99% capture during dynamic operations. Using the PSTU heat-integrated stripper (AFS) configuration, ION demonstrated a minimum SRD of 2.6 GJ/tCO₂ at 91% CO₂ capture for NGCC flue gas with a slight increase to 2.7 GJ/tCO₂ at 97% capture.

Utilizing OGT's ProTreat[®], which was further validated with the empirical data from this test campaign, ION's process model predicted SRDs with an average error ± standard deviation of $0.4\% \pm 1.7\%$. The modeled performance indicates that typical U.S. facilities, where processes are optimized for capital costs due to relatively low fuel costs, ION's technology could provide SRDs of 2.6 GJ/tCO₂ for NGCC and 2.4 GJ/tCO₂ for coal-fired flue gas at 95% CO₂ capture. In high fuel cases, ION could further reduce SRD values.

During the long-term test on NGCC gas at 95% CO₂ capture for 1,500 hours without reclamation and without solvent make-up, the overall mass balance for original solvent components was 99 \pm 1%. These results further substantiate the environmentally advantageous characteristics of the ICE-31 solvent, including extremely low solvent replacement rates in high oxygen environments. Lastly, ICE-31's stability is further demonstrated as a result of the emissions monitoring and extractive sampling – with NH₃ emissions below 1 ppm and solvent below 40 ppb, indicating very low emissions.

The process performance results confirm ION's expectations that ICE-31 is an exceptional solvent for post-combustion carbon capture in general, but more specifically is extremely well suited for high oxygen environments such as NGCC facilities.



Appendix A: Abbreviations

AFS	Advanced Flash Stripper
CH ₂ O	Formaldehyde
CO ₂	Carbon Dioxide
DCC	Direct Contact Cooler
FTIR	Fourier Transform Infrared spectroscopy
GC	Gas Chromatography
LRXC	Lean-Rich Heat Exchanger
HSS	Heat Stable Salts
IC	Ion Chromatography
KF	Karl Fischer water content measurement
L/G	Liquid-Gas Ratio
LLOQ	Lower Limit of Quantification
MLA	Multi-component Liquid Analyzer
NCCC	National Carbon Capture Center
PSTU	Pilot Solvent Test Unit
SRD	Specific Reboiler Duty
TIC	Total Inorganic Carbon
UWW	Upper Water Wash
VLE	Vapor Liquid Equilibrium



Appendix B: Calculations for Capture Efficiency

CO₂ Capture Method 1:

CO_2 Capture Efficiency = (1 ·	$-\frac{\dot{m}_{CO2,abs\ Out}}{\dot{m}_{CO2,abs\ In}}) \times 100$
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Tags Tgas, C, abs IN	8610-TT-2041	Qabs,IN	8610-FT-0150
Tgas, C, abs OUT	8610-TT-2035	Qabs,OUT	8610-FT-2431A
Pgas, barg, abs IN	8610-PT-2040	%CO ₂ IR,high,dry,abs,IN	8610-AI-2004A
$P_{gas, barg, abs OUT}$	8610-PT-2430	%CO2 IR,high,dry,abs,OUT	8610-AI-2030A
Pambient, absolute, mbar	8630-ORA-0051		

 H_2O vapor pressure of both absorber in and out gas streams (P_{H_2O}) are calculated from the "water97_v13.xla" Excel plugin:

P_{H2O} = pSatW(T_{gas, C}+273.15)

The mole fraction of water (f_{H2O}) is taken as:

 $f_{H2O} = P_{H2O}/(P_{gas, barg} + P_{ambient, absolute, mbar}/1000)$

The mole fraction of CO₂, corrected for water content ($f_{CO2,wet}$), is taken as:

 $f_{CO2,wet} = (1-f_{H2O}) * \% CO_2 IR,high,dry/100$

The CO₂ mass flow (\dot{m}_{CO2}) is calculated as:

 $\dot{m}_{CO2,abs} = f_{CO2,wet} * Q * MW_{CO2} * P/RT$

Where Q is in Sm³/h, T_{STD}=15°C, P_{STD}=1 bar and m_{CO2} is calculated for both inlet and outlet gas flows.



CO₂ Capture Method 2:

CO ₂ Capture Efficiency = $\left(\frac{m_{CO2, reg Out}}{m_{CO2, abs In}}\right) \times 100$					
Tags T _{gas, C,}	abs IN 8610-TT-2041	$Q_{abs,IN}$	8610-FT-0150		
Pgas, barg, abs IN	8610-PT-2040	%CO ₂ IR,high,dry,abs,IN	8610-AI-2004A		
Tgas, C, reg, OUT	8615-TT-2210	Pgas, barg, reg, OUT	8615-PT-2213		
Pambient, absolute, n	_{bar} 8630-ORA-0051	\dot{m} reg Out	8615-FT-2215		

 H_2O vapor pressure of both absorber in and regenerator out gas streams ($P_{H_{2O}}$) are calculated from the "water97_v13.xla" Excel plugin:

P_{H2O} = pSatW(T_{gas, C}+273.15)

The mole fraction of water (f_{H2O}) is taken as:

 $f_{H2O,abs} = P_{H2O}/(P_{gas,barg,abs,OUT} + P_{ambient, absolute, mbar}/1000)$

f_{H2O,reg} = P_{H2O}/(P_{gas,barg,reg,OUT} + P_{ambient, absolute, mbar}/1000)

The mole fraction of CO₂, corrected for water content ($f_{CO2,wet}$), is taken as:

 $f_{CO2,wet,Abs In} = (1-f_{H2O}) * \%CO_2 IR high,dry, abs In/100$

The CO₂ mass flow (\dot{m}_{CO2}) is calculated as:

mco2,abs In = fco2,wet,Abs In * Q * MWco2 * P/RT

Where Q is in Sm³/h, T_{STD}=15°C, P_{STD}=1 bar and m_{CO2} is calculated for both inlet and outlet gas flows.

The mass fraction of CO_2 in the product gas (w_{CO2}) is calculated as and is needed because the CO_2 product flow is measured in mass flow (kg/hr):

WCO2 = (1 - fH2O,reg) * MWCO2 /[fH2O,reg * MWH2O + (1 - fH2O,reg) * MWCO2]

CO₂ product mass flow (*m*CO2,REG) is calculated as:

 $\dot{m}_{CO2,reg Out} = W_{CO2,reg Out} * \dot{m}_{Reg Out}$

CO2 capture method 3 (inert pass-thru):

CO₂ Capture Efficiency = $(1 - \frac{\dot{m}_{CO2,abs\,Out}}{\dot{m}_{CO2,abs\,In}}) \times 100$ 8610-TT-2041 Qabs,IN 8610-FT-0150 <u>Tags</u> Tgas, C, abs IN $T_{gas, C, abs OUT}$ 8610-TT-2035 %CO₂ IR,high,dry,abs,IN 8610-AI-2004A Pgas, barg, abs IN 8610-PT-2040 %CO2 IR,high,dry,abs,OUT 8610-AI-2030A Pgas, barg, abs OUT 8610-PT-2430 8630-ORA-0051 Pambient, absolute, mbar

 H_2O vapor pressure of both absorber in and out gas streams (P_{H2O}) are calculated from the "water97_v13.xla" Excel plugin:

P_{H2O} = pSatW(T_{gas, C}+273.15)



The mole fraction of water (f_{H2O}) is taken as: $f_{H2O} = P_{H2O}/(P_{gas, barg} + P_{ambient, absolute, mbar}/1000)$

The mole fraction of CO₂, corrected for water content ($f_{CO2,wet}$), is taken as: $f_{CO2,wet} = (1-f_{H2O}) * %CO_{2 IR,high,dry}/100$

The CO₂ mass flow ($\dot{m}_{CO2, Abs ln}$) into the absorber is calculated as: $\dot{m}_{CO2,abs} = f_{CO2,wet} * Q * MW_{CO2} * P/RT$ Where Q is in Sm³/h, T_{STD}=15°C, P_{STD}=1 bar and m_{CO2} is calculated for both inlet and outlet gas flows.

To calculate the mass flow ($\dot{m}_{CO2, Abs Out}$) out of the absorber, assume inert content fraction is: finert = 1 - f_{H2O} - f_{CO2,wet}

Then the volumetric flow out of the absorber is equal to: $Q_{abs,OUT} = Q_{abs,IN} * (T_{gas, C, abs OUT} / T_{gas, C, abs IN}) * (P_{gas, barg, abs IN} / P_{gas, barg, abs OUT}) * (1 - f_{H2O,Abs In} - f_{CO2,wet,abs In}) / (1 - f_{H2O,abs Out} - f_{CO2,wet,abs Out})$

The CO₂ absorber out mass flow ($\dot{m}_{CO2,abs\ Out}$) is calculated as: $\dot{m}_{CO2,Abs\ Out} = f_{CO2,wet} * Q_{abs\ Out} * MW_{CO2} * P/RT$ Where Q is in Sm³/h, T_{STD}=15°C, P_{STD}=1 bar