

Renewable methanol production for the shipping industry



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Document Approval

Name	Organisation	Role	Action	Date
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Acronym/Abbreviations

HazOp	Hazard and Operability Study
LOPA	Layers of Protection Analysis
PSSR	Pre-Startup Safety Review
SAT	Site Acceptance Test

Contents

Executive Summary	5
1. Introduction	6
Purpose	6
Scope	6
Existing Methanol Synthesis Pilot Plant	6
2. System Description	8
Pilot Distillation Unit Overview	8
Design Basis	9
3. Prerequisites and Readiness	10
Mechanical and Electrical Installation	10
Documentation Availability	10
4. Inspection and Verification	11
5. Functional Testing	12
6. Conclusion	13

Executive Summary

This report demonstrates the successful installation and site acceptance of a new distillation pilot unit. The pilot unit is directly linked to an existing methanol synthesis unit and used to separate the crude synthesized methanol into a methanol rich distillate and waste water. The distillation column was delivered to ALFE as a package unit on April 2nd, 2025. Following the installation and inspections the final water run test took place on November 17th, 2025. During the Site Acceptance Test, the distillation column demonstrated a safe and reliable operation, allowing for operation of the first test campaign.

1. Introduction

Purpose

This report documents the results of the Site Acceptance Test (SAT) conducted on the newly installed pilot distillation unit, located at Air Liquide Innovation Campus Frankfurt. The new pilot unit is directly coupled to an existing methanol synthesis pilot plant. The primary purpose of the SAT is to verify that the equipment, as installed and integrated at the site and the existing infrastructure, meets the specifications, functional requirements, and safety standards before start-up under operational conditions. The SAT serves as a critical step to ensure the unit has been correctly installed, configured, and is ready for the subsequent operation.

Scope

The scope of this SAT encompasses the complete pilot distillation unit, including the distillation column, reboiler, condenser, cooling system, and feed and product system, including all pumps, piping, instrumentation, control systems, and safety devices. The testing consists of mechanical and electrical inspection, approval by authorities as well as cold-commissioning (operation with water)

Existing Methanol Synthesis Pilot Plant

The existing methanol pilot plant situated at ALFE, as illustrated in Figure 1.1, was initially constructed in 2020 as part of the EU-funded "i3upgrade" project¹. Its purpose was to valorise steel off-gases through conversion into methanol. The plant employs a novel multi-stage methanol synthesis process depicted in Figure 1.2, where each reactor stage is followed by inter-stage condensation and product separation. This configuration shifts the reaction equilibrium towards the product side in subsequent reactor stages by separation of the reaction products from the unreacted gases. The reactors are housed in a water jacket for cooling, the reaction being exothermic.

Each of the four reactors contains 2.4 L of a commercial methanol catalyst provided by Clariant, a member of the advisory board. As part of the M²ARE project the pilot plant was extended by incorporating a recycle compressor, enabling the recycling of unreacted gases back into the feed gas. Previously, the plant operated without recycle in once-through operation, a mode advantageous for high inert content feed gases as it prevents inert accumulation. Moreover, a once-through operation allows for quicker stabilization after operational or feed condition changes, as the reactor outlet stream doesn't affect the inlet stream.

While i3upgrade tests demonstrated complete carbon conversion for high CO containing feed gases, CO₂ to methanol conversion rates are limited. To address this, a low recycle was integrated into the multistage reactor concept, facilitating high feedstock conversion while preserving operational flexibility.

¹ The i3upgrade project has received funding from the Research Fund for Coal and Steel under Grant Agreement No 800659.



Figure 1.1: Multi-stage methanol pilot plant at Air Liquide Innovation Campus Frankfurt

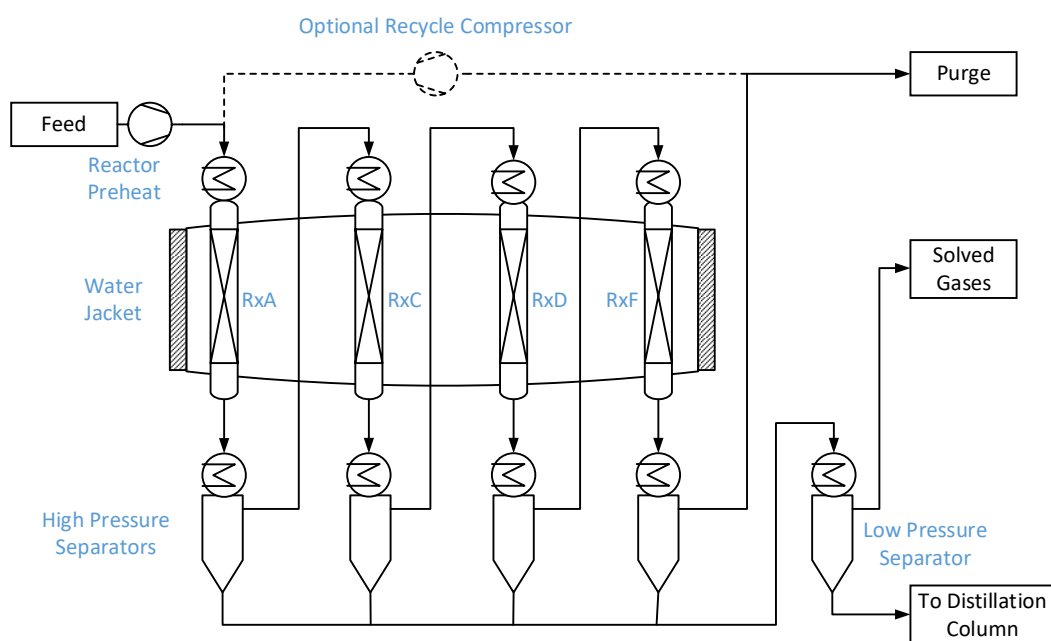


Figure 1.2: Schematic process flow diagram of the methanol pilot plant

2. System Description

Pilot Distillation Unit Overview

The pilot distillation unit, shown in Figure 2.1, is designed for the separation of crude methanol into maritime fuel grade methanol and waste water on a pilot scale to gather process data and validate process designs. The column was ordered as a package unit from a vendor according to specification by ALFE and ALEC. Crude methanol is continually fed from an existing methanol synthesis pilot plant to the distillation unit. The new pilot then separates the crude methanol into a methanol rich distillate and a waste water stream continuously. The distillation column is equipped with a side draw to collect and analyse the distribution of any by-produced formed in the methanol synthesis. The distillation unit uses water as cooling medium and electrical heaters for the heat input. The reboiler is realised as a falling film reboiler. A schematic process flow diagram of the pilot unit is depicted in Figure 2.2.



Figure 2.1: New distillation pilot plant at Air Liquide Innovation Campus Frankfurt

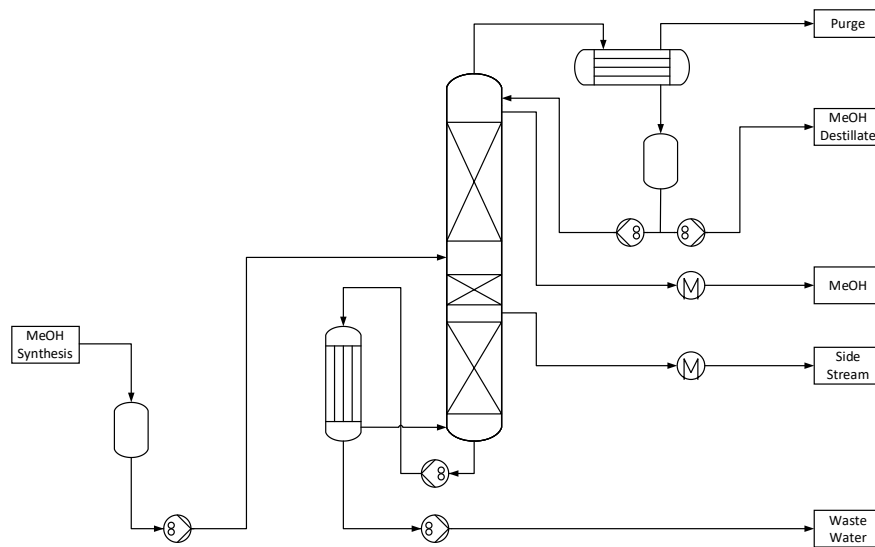


Figure 2.2: Simplified process flow diagram of the new pilot distillation unit

Design Basis

The unit is designed to operate within the following key parameters:

- Nominal Feed Capacity: 20 kg/h of crude methanol provided by the methanol synthesis pilot plant
- Feed composition is dictated by operation of methanol synthesis pilot plant, for CO₂-to-methanol operation, typically 36 wt.% water and < 2000 ppm of other by-products along with methanol are expected
- Operating Pressure Range: 0 to 10 barg
- Operating Temperature Range: up to 200 °C
- Key Separation Target: 0.5 wt.% of water in distillate
- Column Internals: Structured Packings
- Height of Column: 5.3 m

3. Prerequisites and Readiness

Mechanical and Electrical Installation

The distillation unit was delivered to Air Liquide Forschung und Entwicklung in Frankfurt, Germany, on 2 April 2025. Subsequently the column was installed and all mechanical and electrical integration into the existing infrastructure was carried out. Due to challenges in the electrical installation as well as the integration of the new equipment by the vendor the installation of the column and the subsequent distillation were delayed.

Documentation Availability

In accordance with all relevant safety protocols and regulations the following documentation among other is available:

- **Technical Documentation**, including: operating manual from manufacturer, EC declaration, type plates, cause and effect diagram, circuit plan, P&ID as build
- **Permits and Approval by Inspecting Organisation** including for pressure vessels, handling of substances hazardous to water, and explosion-hazardous areas
- **Risk Assessment**, including HazOp and LOPA studies, as well as risk assessments for explosion protection, hazardous materials and overpressure
- **Operating Procedures**, including operating, maintenances, and inertization procedures

4. Inspection and Verification

The Inspecting Organisation DEKRA conducted two separate inspections at the plant, both concluding with "No faults" (German: "Keine Mängel").

The first inspection, concluded on July 5, 2025, checked all pressure vessel equipment of the plant, including heat exchangers, feed vessels, and intermediate storage vessels for products and side streams.

The second inspection, concluded on June 18, 2025, addressed explosion-hazardous areas. This involved an evaluation of technical documentation and a technical inspection, which included visual, close-up, and detailed inspections, as well as an assessment of the condition of safety systems. Table 4.1 provides a summary of all flammable substances potentially present in the pilot plant.

Table 4.1: Parameter of all possible flammable substances in the pilot plant

Flammable Substance	Flash Point, °C	LFL, vol.%	UFL, vo.%	Temperature Classification	Explosion Group
Methanol	11	5.5	36.5	T2	IIB
Hydrogen	-240	4.0	77.0	T1	IIC
Carbon Monoxide	-191	12.5	74.0	T1	IIA
Methane / NG	-188	4.4	17.0	T1	IIA

5. Functional Testing

A thorough Pre-Startup Safety Review (PSSR) was completed before beginning the functional water test. This comprehensive review involved verifying the availability of all necessary technical documentation, finalizing all required risk assessments, completing all essential external tests, and establishing the detailed operating procedures. Some time was required between the inspections and SAT for electrical installation of the equipment and instrumentation as well as integration into the existing control system.

A series of water test runs followed the PSSR. The initial runs were conducted at ambient pressure and limited temperature. Once the equipment's functionality was confirmed and the tightness of all equipment was confirmed, the temperatures and pressures were gradually increased. The final water test run, which occurred on November 17th, 2025, successfully demonstrated safe and reliable operation, achieving operation at pressures up to 10 barg.

Throughout the water test runs various control loops were checked. Critical aspects of the system, such as the successful integration of the new equipment into the control system of the existing methanol synthesis pilot plant, were confirmed. Furthermore, the functionality of key equipment, such as the pumps, were thoroughly tested.

Following the SAT, key areas for process control optimization were identified. The next phase involves starting with the predefined test program for the distillation column by initiating the start-up of the distillation column. This will be done by supplying a methanol-water mixture from the pilot plant as the feed for the column. The objective is to produce various grades of methanol, including the specified distillate with a maximum of 0.5 wt.% water content.

6. Conclusion

The newly installed distillation pilot unit at **ALFE** represents a crucial step in the M²ARE project. Going forward the crude methanol produced in the synthesis pilot plant can be purified to various grades of Maritime Methanol that is then used by **MAN** for engine and engine equipment tests. The successful installation and passage of the Site Acceptance Test protocol conclude the last but crucial step before stating operation.