Mixed refrigerant (MR) processes are used in the majority of baseload natural gas liquefaction facilities. Their high efficiency, attractive capital cost, operational flexibility and simple controllability have made these processes the workhorse in the industry. Dual MR processes are often chosen for larger capacity floating LNG (FLNG) applications due to their smaller footprint and low refrigerant inventory. These processes benefit from having very low losses of refrigerant during normal operation and the ability to retain refrigerants within the system during short outages. However, environmental considerations and the potential need to import refrigerants to remote locations may make it desirable to recover and store refrigerants during extended outages or extended turndown operation.

Nitrogen recycle processes have also gained prominence in the industry, particularly for FLNG applications in the 1 - 2 million tpy capacity range. These processes can be equipped to liquefy nitrogen for storage and eventual use when changing rates or after outages or turndown periods. This article discusses the benefits of and options for conserving and recovering refrigerants for these processes.
Various MR and nitrogen liquefaction cycles have been employed for the liquefaction of natural gas. These include the propane-precooled MR cycle (AP-C3MR™), dual MR cycle (AP-DMR™), single MR cycle (AP-SMR™), C3MR-Nitrogen hybrid cycle (AP-X™) and N2 recycle process (AP-N™). A majority of these cycles utilise a MR stream, which is typically a mixture of nitrogen, methane, ethane/ethylene and propane, and in some cases includes butanes and isopentane. The composition of the MR stream is optimised based on the cycle configuration and operating conditions.

A simplified flow diagram for a natural gas liquefaction facility is shown in Figure 1. The figure shows an AP-C3MR™ or AP-DMR™ cycle and a coil wound heat exchanger (CWHE) as the main cryogenic heat exchanger (MCHE). CWHEs are state-of-the-art exchangers for natural gas liquefaction and contain helically wound tube bundles, housed within an aluminium or stainless steel pressurised shell. The MCHE in Figure 1 contains three bundles: warm, middle and cold. Pre-cooled natural gas feed enters at the bottom end of the MCHE, where it is cooled and liquefied as it flows through tubes and exits as LNG. The LNG is let down in pressure and sent to storage. Flash and boil-off gas (BOG) generated during pressure letdown can be compressed and recycled to be mixed with the feed natural gas or used as fuel. The MR is passed through a phase separator to produce a lighter mixed refrigerant vapour (MRV) stream and a heavier mixed refrigerant liquid (MRL) stream. Both streams are cooled in the MCHE tubes. At the top of the middle bundle, the MRL is flashed across a Joule-Thomson (J-T) valve and sent to the shell-side. At the top of the cold bundle, the now liquefied MRV is flashed across a J-T valve and sent to the shell-side. The MRL and MRV refrigerate the fluids in the tubes as they vapourise in the shell-side. The warm low pressure MR stream from the MCHE is compressed in a series of compressors, cooled, and sent to the phase separator.

**Benefits of refrigerant recovery**

Often, C2 - C5 refrigerant components can be extracted from the feed natural gas using an NGL recovery system, either integrated with, or prior to, natural gas liquefaction. However, in some cases, refrigerants are imported into the liquefaction facility. This includes scenarios where the feed is lean in NGL components or when plot space is at a premium, such as in FLNG facilities. In such cases, refrigerant recovery becomes a critical aspect to consider.

<table>
<thead>
<tr>
<th>Table 1. Case study results</th>
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<tr>
<td>Shell-side refrigerant from MCHE</td>
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<tr>
<td>Pressure (bar)</td>
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<tr>
<td>Temperature (°C)</td>
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<tr>
<td>Vapour fraction</td>
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<tr>
<td>Flow (kgmole/hr)</td>
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<tr>
<td>Composition (%)</td>
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<tr>
<td>N2</td>
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<tr>
<td>C1</td>
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<td>C2</td>
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**Figure 1.** General schematic of natural gas liquefaction facility.

**Figure 2.** Reclamation column for mixed refrigerant recovery.
scenarios, it is advantageous and economical to recover key refrigerant components to minimise the import requirements. Recovering refrigerants is also desirable from an environmental standpoint, since it eliminates or significantly reduces the need for flaring refrigerants.

Ethane is typically a major component in MR systems. If it is not present in a high enough concentration, allowing easy recovery from the feed, ethylene may be imported for use in the MR system, since ethane may not be conveniently procured externally. Propane and heavier hydrocarbons are less volatile, less likely to be lost and are usually easily procured. However, it is still advantageous to avoid losses. Methane can be easily recovered from the feed natural gas. Make-up nitrogen may be easily obtained, since a nitrogen generator will typically be present in the facility for purging and inerting purposes. Thus, recovering ethane and heavier hydrocarbons from MR may be advantageous from economic and environmental standpoints.

During turndown, it is advantageous to remove and store refrigerants for future use. During an extended shutdown, heat leaking into the MCHE will eventually cause an increase in pressure, and it is beneficial to remove methane and nitrogen, recover ethane and heavier components, as well as provide cooling to the MCHE in order to control the pressure.

For nitrogen recycle processes, liquefying a portion of the nitrogen refrigerant for storage and eventual reuse can reduce the size of the nitrogen generator. This can be particularly advantageous for FLNG, where plot space is at a premium.

**Methods to recover and conserve refrigerants**

**Reclamation column**

Key MR components can be recovered and conserved using a reclamation column, as shown in Figure 2. During an extended shutdown or turndown, some refrigerant can be removed from the shell-side of the MCHE and sent to a multi-stage reclamation column that separates the light and heavy refrigerant components to produce an overhead vapour stream and a bottoms liquid stream. The overhead stream consists of light components, such as methane and nitrogen, but is largely free of the valuable ethane and heavier refrigerant components. It typically contains less than approximately 1% ethane and higher hydrocarbons and can be sent to the fuel gas header. The bottoms liquid stream mainly contains the recovered ethane, heavier components and typically less than 10% methane and nitrogen.

The bottoms stream is then either returned to the shell-side of the MCHE, sent to a recovery drum, or stored temporarily at the bottom of the reclamation column.

The overhead stream from the column is partially condensed against a cold LNG stream and is then separated to produce an ethane-free vapour stream and liquid reflux for the reclamation column. The cold LNG stream is vapourised in the condenser and can be used as fuel, or compressed and recycled to the feed or returned to the LNG tank.

**Vapour-phase refrigerants in recycle cycles**

Expander cycles employing vapour phase nitrogen refrigerant are commonly used to provide the cooling required to liquefy natural gas. The N₂ recycle process shown in Figure 3 includes the capability of liquefying a portion of the nitrogen. Nitrogen is compressed, cooled, expanded and used to provide refrigeration to cool and liquefy natural gas. The refrigeration produced is matched to the refrigeration needed for liquefaction by the following two methods:

- For small changes, the N₂ flowrate can be changed.
- For large changes, the N₂ compressor suction pressure is changed; lowered for less refrigeration, increased for...
more refrigeration. The pressure is changed by adding or removing N₂ from circulation.

To remove N₂ during turndown operation, a portion of high pressure nitrogen is sent through to the MCHE and liquefied to make liquefied nitrogen (LIN), which is sent to storage for future use. To reintroduce N₂ and increase the N₂ compressor suction pressure, LIN from storage is vaporised and returned to the suction of the nitrogen compressor. A similar arrangement may be employed for a methane expander cycle for natural gas liquefaction. This method eliminates or greatly reduces refrigerant make-up needs.

External shutoff valve
Another method for storing refrigerant in-situ during short outages is to maintain the refrigerants at an optimal location in the MCHE during the shutdown. This can be done using external shutoff valves to prevent cold end refrigerants from flowing to the warm end of the MCHE. An example is shown in Figure 4. If desired, the shell-side refrigerants may be drained from the MCHE and sent to a refrigerant recovery drum for temporary storage.

Case study
The basis of this case study is a 5 million tpy LNG facility that produces approximately 600 tph of LNG and has been shut down for an extended period of time. When the pressure on the shell-side of the MCHE has built up to 6.9 barg due to heat leak into the system, refrigerant is removed from the shell-side of the MCHE and sent to the reclamation column. This is a small column, approximately 200 mm dia. x 4.5 m long and contains 25 mm Pall rings. The column separates the refrigerant into two streams: overhead vapour and bottoms liquid.

The overhead vapour from the condenser primarily contains nitrogen and methane and the bottom liquid product contains ethane and heavier components. The column’s condenser is cooled by partially vaporising LNG from storage and the two-phase stream is sent back to the LNG tank. The conditions of the streams during the initial operation of the column are listed in Table 1. A majority of the light components are removed in the overhead stream from the column and can be used as fuel. The bottoms product constitutes valuable heavier refrigerants and can be sent to the shell-side of the MCHE.

The reclamation column efficiently separates the light and heavy refrigerant components, thereby recovering valuable refrigerants during an extended shutdown.

Conclusion
Refrigerant recovery in a natural gas liquefaction facility can provide environmental and economic advantages. It is often particularly beneficial to recover ethane since it is not always available in sufficient quantities in the feed gas to satisfy the refrigeration demand, and it is not always easily procured externally. The reclamation column efficiently separates the MR into two streams: ethane and heavier hydrocarbons; and lighter components such as methane and nitrogen. The lighter components can be used as fuel, while the valuable heavy components are returned to the MCHE or temporarily stored. Refrigerant recovery during turndown operation may be achieved in a nitrogen recycle process by liquefying the excess nitrogen in the MCHE and storing it in a recovery drum. Overall, proper refrigerant handling and inventory management minimises environmental impacts and improves project economics.

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