# Advanced In-Lab Helium Recovery System for NMRs

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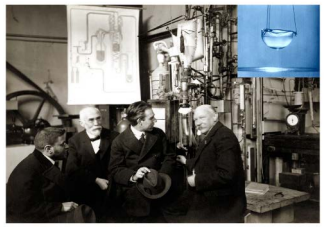
## Introduction

In 1908, on July 10th, Dutch physicist Heike Kamerlingh Onnes finally succeeded in liquefying helium (the last gas ever to be turned into liquid) in his cryogenic lab at the University of Leiden. After a long day's work, he produced just 60 milliliters, essentially a small tea cup of this precious liquid. That special day proved to be monumental for the development of a brand new research field known now as low temperature physics. Superconductivity was soon discovered, which in turn led to the development of the high field superconducting magnets that are currently used in all NMR systems.



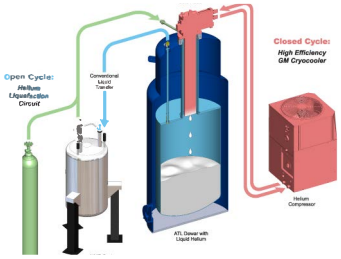
A constant supply of liquid helium needed periodically to top off the cryostat is crucial for the on-going operation of NMR magnets. Today many labs still rely on external deliveries of helium storage tanks. However the increase in demand for liquid helium worldwide in recent years has caused ever more frequent price jumps, supply shortages, and delivery cancellations.

Some labs have an on-site liquefaction facility. However the traditional, industrial scale liquefier which is designed to produce a minimum of 70 liters per hour is not an efficient solution for NMR labs. These traditional liquefiers are typically only operated part time, and the all-too-often cooling and warming cycles are prone to malfunctions.

Quantum Design's Advanced Technology Liquefier (ATL) offers a new method of helium liquefaction and recycling that can be located within the laboratory. The available ATL liquefaction rates of 12, 22 or 30 liters per day are perfect for labs with one or multiple NMR cryostats. In addition, different recovery systems can be configured according to recovery goals.

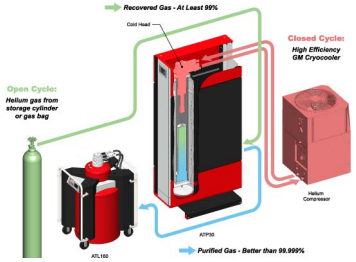
The recent installation of an ATL160 liquefier and ATP30 purifier in the Kamerlingh Onnes Lab at the University of Leiden (left) is noteworthy in that it marks a return to the origin of in-lab helium liquefaction, only now this process can be tailored to the needs of individual labs, with the most advanced cryogenic technology and system automation available.

## Advanced Liquefier Technology




The working principle of the ATL is characterized by two cycles: the Closed Cycle, formed by the cold head (at ~ 4K) and the compressor removes heat from the helium gas space and continues to cool the gas until precipitation and liquefaction take place; and the Open Cycle, where a pure helium gas source (either user instruments or UHP gas cylinders) flows to the ATL dewar is liquefied, and then is transferred back to user cryostats.

## Advanced Purifier Technology




When the input gas to the ATL is not of the highest purity, contaminates form an ice layer that coats the cold head which then reduces liquefaction rates. Similar to the ATL, the Closed Cycle of the ATP also consists of a cold head and compressor. The Open Cycle starts with recovered impure helium (green) which passes through the ATP to freeze out the contaminates, and ends with purified gas (blue) entering the ATL for liquefaction.

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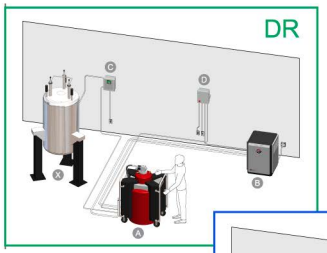
## Helium Recovery Systems for NMR Labs

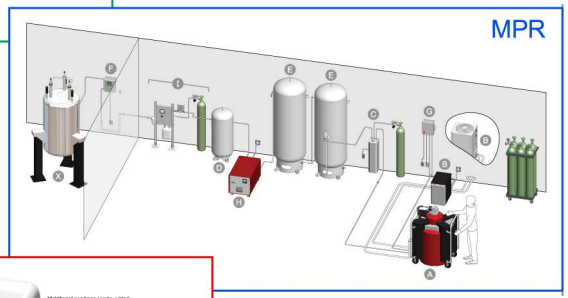


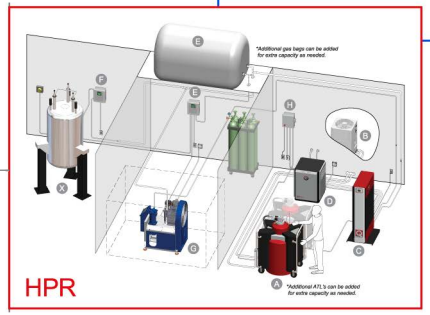

**Direct Recovery (DR)**
A – ATL
B – ATL Compressor
C – Back Pressure Controller
D – ATL Power Distributor
X – NMR cryostat

Lab with single or multiple NMRs.
Simple set up; Recover 100% normal boil off; Transfer boil off not collected.

**Medium Pressure Recovery (MPR)**
A – ATL160 (or ATL80) Liquefier
B – Compressor for ATL160
C – Helium Purification Unit
D – Low Pressure Buffer Tank
E – Medium Pressure Storage Tank (1000 liters)
F – Back Pressure Controller
G – ATL160 Power Distribution Unit
H – AT Recovery Hub – MP
I – Low Pressure Stand
X – NMR cryostat

Lab with single or multiple NMRs.
Recover 100% normal boil off;
Recover transfer boil off up to 10 liter/hr




**High Pressure Recovery (HPR)**
A – ATL160 (or ATL80) Liquefier
B – Compressor for ATL160
C – ATP30 Purifier
D – Compressor for ATP30
E – Helium Gas Bag and Controller
F – Back Pressure Controller
G – HP Recovery Compressor
H – ATL160 Power Distribution Unit
X – NMR cryostat

Labs with multiple systems, high normal boil off;
Recover 100% normal boil off;
Recover large transfer boil off; high pressure storage.

## HPR for NMR Lab at the Complex Carbohydrate Research Center, University of Georgia, Athens, USA (also see UGA Poster #442*)

The world's first high pressure helium recovery system utilizing a compact, cryocooler-based helium liquefier and purifier has been fully operational since November 2013, at the NMR lab in the Complex Carbohydrate Research Center at the University of Georgia, Athens. The recovery system consists of one ATL160 liquefier one ATP30 helium gas purifier, and integrated helium recovery components including electronic back pressure controllers, gas bags, and a high pressure compressor complete with on-board controller to allow automated operation. The current recovery rate is close to 90% as all boil-off is recovered, including daily normal boil-off from the NMR cryostats, and the boil-offs incurred during liquid helium transfers from the ATL160 to the NMR systems.


The extensive helium recovery lines for 6 NMR systems were designed and installed by the UGA NMR lab staff members. The ATL160 and ATP30 (both with very small footprints) are installed inside the lab (Figure, right). The gas bags are hung from the wall to conserve space (Figure, left). The ATL160 produces an average of 25 liters of liquid helium per day using high purity (99.999%) gas supplied from the ATP30. The ATP30 purifies the recovered gas that is stored at 2500 psi in a cylinder bank.

Clearly a traditional large scale helium liquefaction plant capable of liquefying 30-50 liters per hour is a poor match for NMR systems, which have very low daily boil off. The ATL160 and ATP30 represent a new generation of compact helium recovery technology that proves an ideal solution for NMR laboratories. Different configurations of ATL, ATP, and recovery components, can produce recovery systems that are suitable for NMR labs of various sizes and needs.

*UGA Poster #442
Implementation of a Dedicated NMR Lab Helium Recovery System
David Live[4], John Glushka[1], Greg Wylie[2,3], and James Prestegard[1]
[1]Complex Carbohydrate Research Center [2]Department of Chemistry University of Georgia, [3]Present address Department of Chemistry Texas A&M University

## ATL160


The ATL160 is designed for ease-of use, with a friendly, touch-screen user interface and remote access available over the internet. Unlike other "experimental" or industrial liquefiers, the ATL is meant for general users, requiring minimum experience or knowledge in low temperature instrumentation.

The physical size of the ATL160 (with a capacity of 160 liters) is comparable to a 100 liter helium storage tank, and the dewar cart is extremely portable and can be pushed around effortlessly on its four wheels. In addition, the ATL dewar is a low loss cryostat. In the case of a power outage, the ATL160 will maintain the liquid helium inside with only a small loss of 1-2 liters of helium per day.

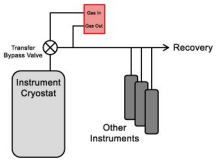
| | |
|---|---|
| **Average Liquefaction Rate:** | 22 liters/day (0.42 liters/hr) * |
| **Dewar Capacity:** | 160 liters |
| **System Dimensions (L x W x H):** | 70 x 31 x 61 in (liquefier without compressor) |
| **Compressor Package Model:** | Split Air Cooled or Water Cooled |
| **Typical Power Consumption and Voltage Range:** | 6.5/7.5 KW; High & Low Voltage Available |
| **Gaseous Helium Requirement:** | Flow Rate: 0 - 20 SLPM (0 - 0.72 SCFM); Industrial Quality He (99.99%); 0-5 psi |

*Liquefaction rates vary based on input helium quality and pressure

## Back Pressure Controller (BPC)

Pressure changes in the NMR cryostat indirectly impact the noise spectrum of measured data. When an NMR system is connected to a helium recovery network, a Back Pressure Controller (BPC) is necessary to maintain pressure stability

A BPC works to control the cryostat pressure by opening and closing a proportional valve to raise or lower the pressure. Changes in boil off rate are automatically adjusted for by allowing more or less gas through the BPC. The BPC also isolates the cryostat pressure from pressure changes in the recovery network.




## ATP30


The ATP30 is the world's first cryo-cooled low pressure helium purifier. It provides a helium gas stream with 5 ppm purity to the ATL160 for improved liquefaction performance and long term operation without fouling.

Some drawbacks of traditional Liquid Nitrogen cooled purifiers where a molecular sieve is the main purification agent include:

- Amount of impurities that can be stored is limited
- When cartridge is saturated, impure helium gas will exit purifier and enter recovery stream
- If molecular sieve is contaminated by water, only high temperature (>100 C) can regenerate cartridge
- Needs frequent nitrogen refill
- Pulling the cartridge is a laborious effort; regeneration process is complicated
- Often needs a second cartridge

For the ATP30, weekly regeneration can be done easily through software by warming the dewar to above 100 K - 120 K. The whole process takes less than 3 hours.

| | |
|---|---|
| **Performance:** | • Purifies 30 liters of helium gas per minute<br>• Purifies helium gas to 99.9995% (better than UHP)<br>• Fail Safe Operation – Stops operation before "dirty" gas passes through system<br>• Full regeneration of system in 5 hours |
| **Dimensions (L x W x H):** | • 24 x 24 inch base, 9 x 26 x 48 inch case<br>• 18 x 18 x 22 inch air or water cooled compressor (not shown) |
| **Electrical:** | • 2.2 KW<br>• 3 phase, low or high voltage |

*Liquefaction rates vary based on input helium quality and pressure.

RECYCLING HELIUM