2nd quarter 2007 – No. 35

AIR LIQUIDE
ADVANCED TECHNOLOGIES

# THE CRYOSCOPE

## The Test Center as much in demand as ever!

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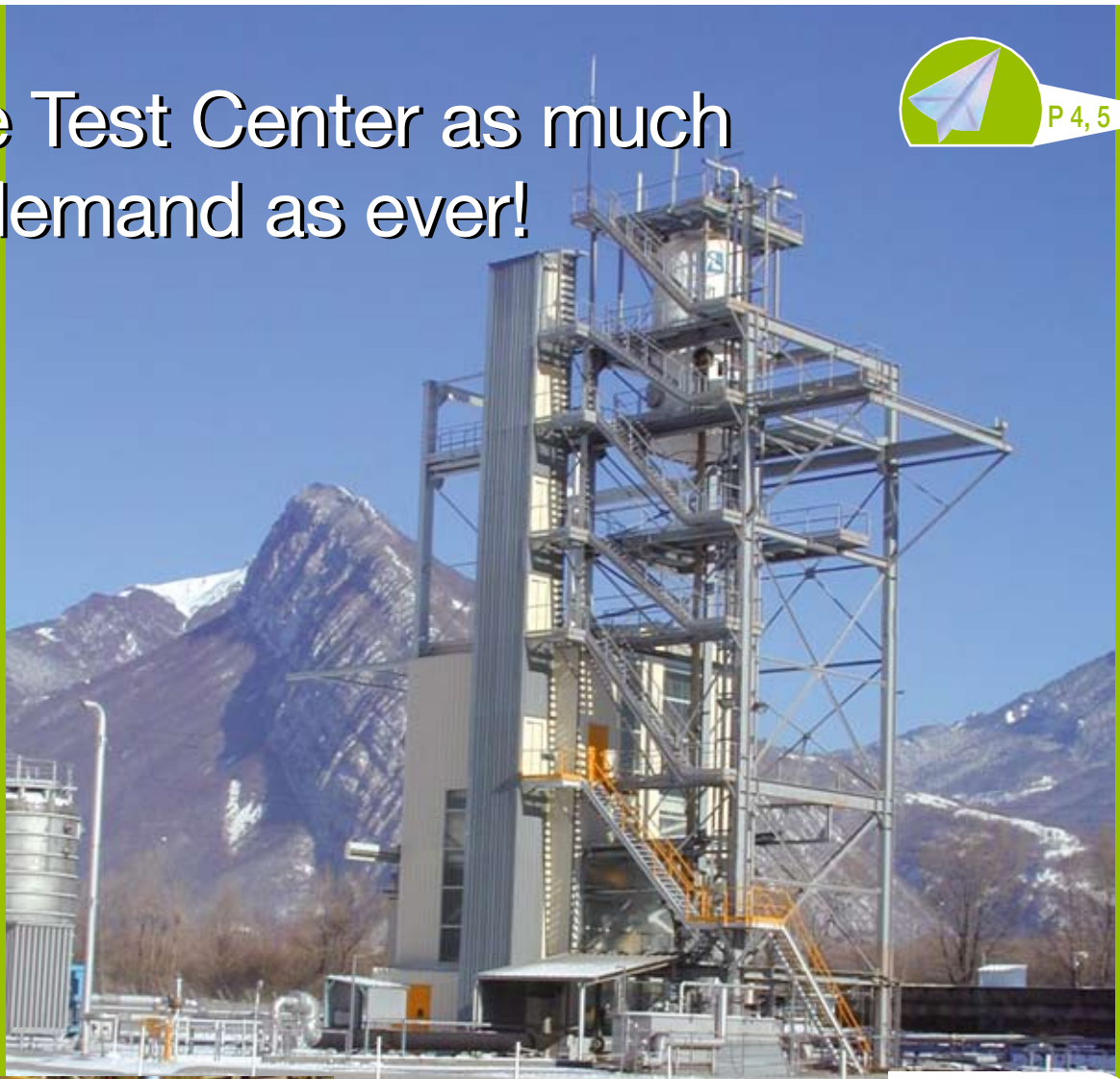

The control room at the DTA test center.



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## Setting up in China and the United States: an exercise in getting close to other cultures, being able to react to them and to understand them

DTA is gradually achieving penetration in the huge markets represented by China and the United States. Evidence of this can be found in two recent projects, one to deliver cryostats to the Long Island Power Authority in the United States in 2006 and the other to supply a HELIAL 2000 to the Shanghai Synchroton Research Facility scheduled for the summer of 2007 – but distance is still acting as a curb on our burgeoning conquest.

Our customers certainly want us at their side, particularly when it comes to providing them with technical support.

To this end a DTA representative will be settling in at an Air Liquide site in Delaware from next June and by August 2007 a senior engineer will have left to join our Chinese marketing man operating in Shanghai.

In the United States, we will be committing our skills and our technologies to strengthening the Air Liquide Group offer, particularly in the electronics field and we will also be stepping up our presence alongside our partners operating in superconductivity and cryogenics. Lastly we will be developing the "hydrogen energy" option, in support of its roll-out in North America. We will be more effective as a result.

In China our aims are similar. Our first target is the electronics market, to help to expand the Group's business activities in this area. We also intend to further expand our business activities in the cryogenics sector, mainly in the direction of research laboratories.

But our effort to "go global" will not stop there. In the short term, we are seeking to beef up our presence in Japan which already has one entity – Advanced Gas Division – in close liaison with DTA.

Setting up in these countries will help us to grow. We are in no doubt about this. It means we can get closer to our customers, present and future, react more appropriately to their needs and gain a better understanding of their culture.

**Didier Magnet**
Director of DTA

## CCS refrigerators land a bonus from CERN

LHC particles (see insert p.3) will be accelerated using superconducting magnets, operating at 1.8K (-271°C). *"This degree of cooling is achieved in a number of stages,"* explains Jean-Christophe Courty. *"A first system cools the helium to its boiling point of -4.5K and the liquid helium is then sent to other refrigerators, or CCS (Cold Compression Systems) which lower its temperature still further."*

CERN has ordered four CCS from DTA, housed in subterranean cavities, all around the accelerator. Each CCS includes, inter alia, three compressors on magnetic bearings with rotation speeds of 40,000 revs/min! When the pressure is reduced the temperature of the liquid helium is brought down from 4.5 to 1.8K.

*"It took only 9 months to start up and deliver the four CCS. Air Liquide know-how and capacity for industrialization meant that we were able to meet CERN specifications in full,"* says a very pleased Jean-Christophe Courty. *"In reaching this level of performance we felt we had achieved the impossible, considering that we were working 100 m underground and that we were reliant on other facilities. And in the end, Air Liquide even secured a bonus for reducing the consumption of electricity in the system."*

**The CCS refrigerators are installed in cavities around the LHC.**

## A record order for gas purifiers

**A DTA hydrogen purifier**

Seven gas purifiers have just been ordered from DTA by one of the largest European microelectronics sites, AMD (Advanced Micro Devices) at Dresden in Germany, namely three Argon purifiers, two for Helium and two for Hydrogen. These machines will be installed in the extension to the Fab38 production unit at the German site, which manufactures "wafers" – the silicon discs used as the medium for electronic chips. *"The ultra-pure gases emerging from the purifiers are used as carrier gases in all processes in the manufacture of electronic chips – deposition, etching, doping, etc"* explains France Hamber.

This is not the DTA's first involvement with AMD, but it is the first time the German customer has asked for so many purifiers at the same time. This contract on its own represents no less than 50% of DTA's annual business in "purifiers"!

Only the cryogenic hydrogen purifiers will be manufactured at Sassenage, since only DTA has the technology. *"The others will be made by our partner Japan Pionics,"* France Hamber goes on, *"but we have helped them to adapt their products to the European market. It will mean that deliveries will be staggered from April to August 2007."*

This order confirms DTA's leading position in the purifier market in Europe.

## Hydrogen station: multiple projects on the American continent

North America is playing a major role in rolling out the hydrogen option as the energy vector of the future, as can be seen quite clearly from the projects being conducted by DTA on the new continent.

Following DTA's demonstration of the feasibility of a 700 bar hydrogen station at the last Challenger Bibendum, one order has followed the other for this type of equipment, particularly in North America. One car maker has ordered a station in order to test it against the extremes of climate, both very cold weather (-40°C), and intense heat (+ 85°C). A prototype was sent out in January 2007 prepared for testing in winter conditions, to a climate test center in Canada. The automated version, with the capacity to supply 40 kg of hydrogen per day, has just joined the prototype. It will be used to supply the fuel cell vehicle fleet, which was rolled out earlier this year by the manufacturer in North America. Another project is for a hydrogen station which fuels the buses for the Ottawa Senate. This project is being conducted in collaboration with Air Liquide Canada, and Ford. The station will be operational from the end of March 2007. Finally, Air Liquide's brand-new research center in Delaware has also been fitted with a station in collaboration with the local university.

**A hydrogen bus for the Ottawa Senate.**

**The station being cold-weather tested in Canada.**

## Cold-trapped tritium

We've done it! The treatment system for the "heavy water" produced by the nuclear power station at Wolsong in South Korea is ready to roll. As part of this project, DTA has delivered a 20K helium refrigerator to the South Korean electricity provider KHNP (Korean Hydraulic and Nuclear Power).

*"But our work did not stop with this delivery,"* stresses Eric Fauve. *"We were asked to connect our cold box to the customer's tritium/deuterium distillation column, and to manage the interfaces between the two systems. This is the first time we have taken part in a project of this kind. The distillation column will not only allow the tritium to be isolated from the deuterium but also means it can be recovered for another use, i.e. nuclear fusion."*

Although nuclear fusion reactors, like KSTAR (Korean Superconducting Tokamak Advanced Research)*, only operate today on an experimental basis, they are intended to produce the electricity of tomorrow, energy which will be "clean" and plentiful.

** N.B.: DTA is also participating in the KSTAR Korean project, providing it with a 4.5K refrigerator and a valve box.*

**The team dedicated to the KHNP project.**

## LHC cryogenics inaugurated

The cryogenics for the LHC (Large Hadron Collider), the particle accelerator eagerly awaited by physicists, will be inaugurated on May 31st and June 1st 2007 at CERN.

The LHC is the largest cryogenic complex in the world. It also represents 15 years of human investment, research, technological record-breaking etc, for DTA.

*"This inauguration is probably a unique opportunity to visit the LHC Tunnel, the related physics experiments and our installations,"* enthuses Hadi Moussavi, of DTA.

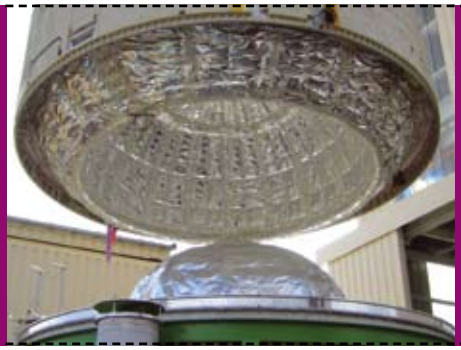
## After the HELIALs comes the HYLIAL

In the 1980s, DTA marketed the HELIAL range, standard helium liquefiers. Today, DTA has decided to launch standard hydrogen liquefiers or HYLIALs, to meet growing needs for hydrogen worldwide.

*"The principle is similar since a helium cycle is used to cool the hydrogen to be liquefied,"* explains Christophe Mantileri. *But hydrogen has a special feature in that it exists in two forms – ortho and para. In the liquid state, para-hydrogen is the stable form. If it is liquefied too quickly, the hydrogen does not have time to pass into the para state and the ortho liquid obtained reverts to gas! We found the answer in a catalyst that accelerates conversion from ortho to para during liquefaction. The para-hydrogen collected remains in liquid form."*

The first HYLIAL has been ordered from DTA by a Chinese laboratory.

*"The customer is well acquainted with our know-how in relation to HELIALs and with our hydrogen expertise,"* specifies Christopher Mantileri. *"In addition to this HYLIAL, which produces 600 liters of liquid hydrogen per hour, DTA is also providing the analysis system and can therefore monitor gas purity at every stage in the liquefaction cycle."*



The test center, spread over nearly 6 hectares, or half the surface area of the Sassenage High-Tech Center, was designed to test space equipment for the Ariane launcher. It is used today by a great many

**Integrating the tank during assembly of ESCA's liquid hydrogen drain rig, the cryogenic upper stage for Ariane 5.**

Innovation

# The Test Center is as much in demand

In 1962, the space industry market led Air Liquide to set up a secure controlled-access area at Sassenage for testing the components (materials, insulating agents) and the equipment (cryogenic fluid tanks) that DTA delivers to its customers. Infrastructure (testing stands, vacuum chambers, confinement enclosures etc) were set up and a high level of expertise was developed which is now of increasing value to research centers or industrial interests concerned about the quality and reliability of their own technological developments.
In the area of materials characterization at very low temperature, for example, there is a team that operates to meet internal needs or on behalf of companies such as CNES, Alcatel and Sagem. Thermal, mechanical and dynamic tests are carried out on metal or composite materials, or parts that may be soldered or bonded together for example. In 2006, the DTA site was approved under the EFDA – European Fusion Development Agreement – to conduct mechanical tests on materials to be used in the ITER project, for the experimental thermonuclear fusion reactor which will be set up at Cadarache.

### In the service of innovation

The hydrogen energy option is another example of the usefulness of the Sassenage test center and the contribution it makes to innovation. Indeed the first 350 bar gas hydrogen filling station was built here in 2003 and in 2006 the 700 bar mobile unit already used at events such as Challenge Bibendum or the World Hydrogen Energy Conference was developed. Furthermore, the test center approved a number of constituent components of the stations, such as filling hoses, anti-tear components and connections. High-pressure permeation tests were also carried out on the composite tanks of the manufacturers.
Good use will be also made of the test center under the Hychain-Mintrans project jointly funded with the European Commission's General Directorate on Energy and Transport. As well as fuel

industrial and scientific customers who come, in complete safety, to check the quality and reliability of their equipment, facilities and materials.

**Close to the 700 bar hydrogen station, carrying out a filling test on a high-pressure composite tank.**

# as ever!

cells, components of the hydrogen gas distribution system on board vehicles will be tested at Sassenage. This will mean that testing stands have to be designed and performance tests carried out on the throughput, pressure regulation and vibration strength of gaseous hydrogen at 350 bars and then 700 bars.

**A dedicated team**

The expertise of the Sassenage test center is also called upon to appraise other facilities for both internal and external customers. This applies to the thrust turbines marketed by DTA for helium or hydrogen liquefiers where the characteristics are tested prior to delivery, and also to components for liquid oxygen tanks that are required to resist significant pressure and for which vibration resistance and impact resistance tests are currently in progress. The permanent availability of cryogenic fluids like hydrogen, nitrogen or helium is an undeniable advantage. It means for example that regular thermal cycle testing, between 20 and 300 K can be carried out on components like mirrors or composite panels.

Given the use of cryogenic fluids and high-pressure testing, a strong culture of safety is central to the test center's permanent team, currently 12 in number. Their skills stretch well beyond that however, since when any test is carried out, they have to assemble testing stands, take measurements, acquire data in the field of mechanics, electricity, automated control systems, cryogenics etc., and a good knowledge of physical phenomena is therefore indispensable.

They benefit from the collaboration of other DTA entities in carrying out their assignments, particularly research departments and experts in the field of calculation or on legal requirements. The know-how and resources the test center has at its disposal means they can respond quickly to any requests that may be made while new projects are being developed or rolled out.

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**Thermal, mechanical and dynamic tests are carried out on metal or composite materials, or on parts that are soldered or welded together.**

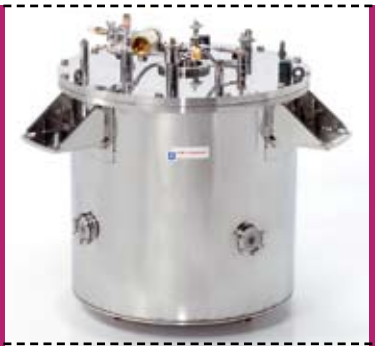
## A guarantee for ISIS

**A second neutron source is being set up, not far from Oxford, by the CCLRC(1). ISIS TS2 is aimed at research applications in the area of physics, chemistry, biology, materials etc. To scan the material, the neutrons have to be slowed down by two moderators, which are themselves cooled using an equipment set designed by DTA.**

**In the first of these, gaseous helium at 16K causes the methane to solidify straight away. This load then puts a curb on the neutrons. In the second moderator, the liquid hydrogen pre-cooled with gaseous helium to 14K solidifies the methane load. In both cases, the cold helium comes from Brayton cycle refrigerators, a dedicated "loop" allowing the hydrogen to cool and circulate.**

**The CCLRC already availed itself of the services of the Sassenage test center two years ago, in order to validate the operation of the $CH_4$-$H_2$ moderator, by supplying it directly with liquid hydrogen. Satisfied with the results obtained at that time, and encouraged by the expertise and resources available at Sassenage, the British laboratory ordered the refrigerators, hydrogen loop and cryogenic lines it needed from DTA. In February and March 2007, both systems formed by the moderators and the DTA equipment were tested at the test center before being delivered to the Rutherford Appleton laboratory.**

*(1) Council for the Central Laboratory of the Research Councils.*



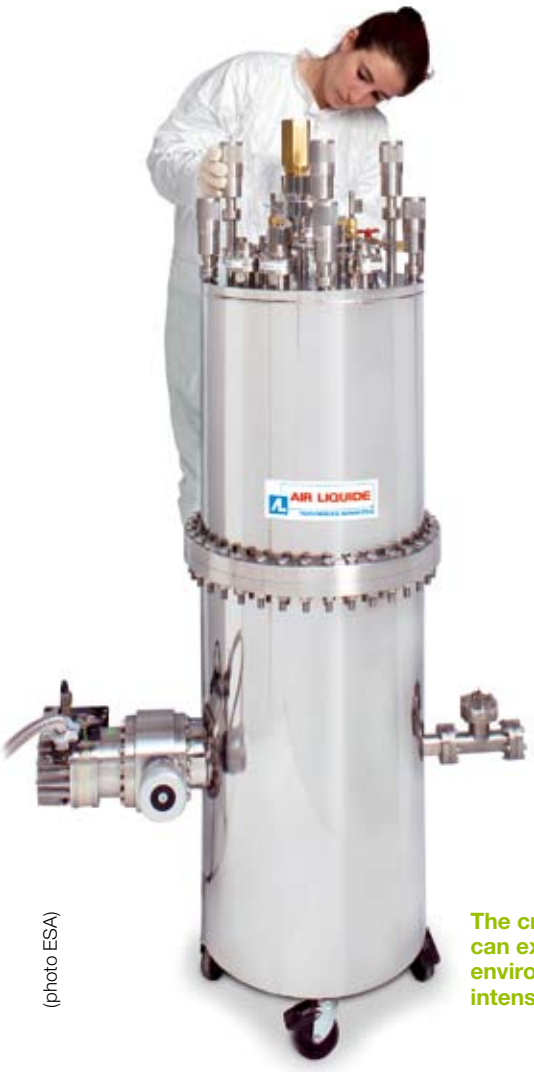
Superconductivity, optics, electronics, materials etc., – an increasing number of fields where research programs require samples to be cooled so that their characteristics can be defined. For all these applications, and under

**"Dual" coil cryostats.**

## Laboratory cryostats

# Know-how at the service of research

Before developing a cryostat, the first criterion to be analyzed is the temperature range that needs to be covered to meet the conditions of the experiment, and the degree of accuracy to be attained. Since it is known that nitrogen is liquid at 77K and helium at 4K, this approach therefore allows the cold source to be determined straightaway. The second criterion is the space available around the sample. If there is no constraint, it is essential to use a bath cryostat since, being equipped with its own tank, it is stable and more cost-effective in terms of consumption. Conversely, if environmental constraints require it, a circulating cryostat is preferred. This choice is not without effect on the operational cost, since it is heavier on consumption than a bath cryostat – but it proves to be cheaper when short-term operations are involved since it gets cold very quickly.




(photo ESA)

**The cryostat at the University of Taiwan can examine samples at 300 mK, in a UHV environment ($10^{-10}$ millibars) and a very intense magnetic field (7 tesla).**

### To each his own cold

Nitrogen or helium, bath or circulating? Design work has only just started since, as Bruno Doddi, the Sassenage High-Tech Center's expert in the field, makes plain: *"A cryostat is a tool for controlling temperature, but all experiments are conducted in a particular environment where constraints increase as a function of other equipment. It is often therefore the case that only one objective needs to be defined when developing an engineering design with the customer."* Let's consider one example – for an industrial group with research centers in Switzerland, the Netherlands and in the United States, simple nitrogen cryostats were sufficient. But to trap the gases with which it is concerned, DTA delivered equipment that is much more complex than standard products.

To obtain extreme levels of cold – and in harsh conditions – the team at Sassenage has also developed some actual cryogenic machines. From the helium 3 adsorption cryostats known as Socool (Sorption Coolers), using technology developed by the CENG(1), DTA is making specialist equipment such as PT Socool which include Pulse Tube technology (minimum temperature 4K). When the refrigerator is integrated, the temperature can be brought down to 300 mK, and this can be done without any manipulation of the cryogenic liquid.

### Specific cryostats

To characterize materials in certain environments, DTA designs bath cryostats that satisfy criteria such as the magnetic field or the ultra-high vacuum. This happened with the University of Taiwan which was looking for a device which would enable it to put samples at 300 mK into a UHV (Ultra High Vacuum, i.e. $10^{-10}$ millibars) environment and a magnetic field of 7 tesla. The cryostat made for the LURE – *Laboratoire pour l'Utilisation du Rayonnement Électromagnétique* or, in English, Laboratory for the Use of Electromagnetic Radiation – at the University of Orsay is being used today by other European centers. Here too, in an ultra-high vacuum and in a magnetic field of 7 tesla, the sample is displaced vertically and in

whatever conditions the experiments are conducted, the Sassenage High-Tech Center designs and produces cryostats.

**This PT Socool cryostat, made for the ESA, is fitted with shock absorbers to prevent vibrations.**

# centers

rotation upon itself, at a temperature varying between 1.5 and 350K.
This laboratory is associated with the Soleil project, and it was therefore quite natural for DTA to be consulted about making a similar cryostat, to be fitted to the synchrotron's beamlines. The second device already delivered to this customer is "dual", since the outer cryostat, which allows a magnetic field to be maintained, itself contains a cryostat operating at 2K (see photo of UHV cryostat). It is fitted with a press so that mechanical tests can be conducted on the samples. Finally, and still at Soleil, a new bath cryostat will soon be delivered so that experiments can be conducted on radioactive samples (see photo with Bruno Doddi).
Standard circulating cryostats can also be adapted to multiple applications. The LADIR – *Laboratoire de Dynamique, Interactions et Réactivité* or, in English, Laboratory for Dynamics, Interactions and Reaction Capability – set up at the Institute for Chemical Sciences at Vitry-Thiais, analyses and models the atomic and molecular interactions and chemical connections making up the material, using optical and vibration or spectrometries, and mass spectrometry. To help it with its experimentation needs, the LADIR asked DTA to design and implement an OM cryostat, in other words a cryostat dedicated to optical microscopy and operating between 4.2K and 300K. Since each sample has to be examined in a vacuum, the cryogenic fluid is routed towards an exchanger block which, by conduction, cools the copper support on which the sample is placed. It is top loaded and then placed as close as possible to the lens.
From customer request to testing, all special projects(2) are developed in close collaboration with the end user, so that any cryostat delivered on site is entirely suited to the experiments it is to conduct.

**At DTA, Bruno Doddi standing in front of a cryostat in the process of assembly and due to be delivered to the Soleil synchrotron.**

**The UHV or Ultra-High Vacuum cryostat.**

*(1) Centre d'Études Nucléaires de Grenoble (French Atomic Energy Commission).*
*(2) Other DTA references: European Space Agency (ESA Netherlands), Istituto Nazionale di Fisica Nucleare (INFN, Italy), EPFL (Ecole Polytechnique de Lausanne), National Tsing Hua University (Taiwan), Department of Atomic Energy (India), International Science Technology Center (Moscow), Institut National des Sciences Appliqués de Lyon etc.*

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In 1980, HELIALs were truly revolutionary, in that they were the first helium liquefiers to be fully automatic.

Nearly 30 years later, their refrigeration capacity has grown enormously, but helium liquefier-refrigerators still operate on the same principle, and in 2007, a new range, christened HELIAL Evolution, is being launched.

Interview with Aurélie Caillaud, HELIAL product manager at DTA.

# Cryogenics

# HELIALs perform even better

**Who needs cryogenics?**

In the industrial and research world, requirements for refrigeration capacity vary. A few examples will suffice to demonstrate this. Cold is essential for testing satellites – indeed space simulation chambers are maintained in a cold atmosphere to reproduce the conditions of the interstellar vacuum. Cold also allows magnetic fields of very high intensity to be obtained (up to 100,000 times more intense than the Earth's magnetic field!) so that particles can be accelerated in synchrotrons, without requiring excessive amounts of electricity. Cold is also used in the nanotechnologies, in biology, quantum physics and also in large-scale electricity transmission etc. For all these applications we have been marketing the HELIAL liquefier-refrigerator, for nearly 30 years throughout the world, in the United States, France, Great Britain, Switzerland, Italy, China, Japan, Thailand, India, Korea etc.

**What is a HELIAL and what are its strong points?**

The HELIAL produces cold at a temperature close to absolute zero (-270°C). To reach this ultra low temperature, it uses helium gas, as is suggested by its name, and this is the coldest gas in a liquid state. The special feature of the HELIAL is that it has been designed with fully automatic operation, so as to prevent the customer having any start-up problems.

Ranges have evolved and refrigeration performance has improved over the years. Today, each HELIAL includes a modem for remote support and, in the interests of simplicity and reliability, there are no components in the cold box, which is the core of the device, that can be disassembled.

But above all, even though HELIALs operate on the same principle – two expansion turbines in series, lubricated screw compressors, heat exchangers etc. – ensuring reproducibility of manufacture, synonymous with cost reduction, DTA adapts each HELIAL to the customer and to the application – a level of adaptability which we intend to retain for our new HELIAL range.

**Why this new range?**

For increased performance! Until now, our HELIALs had not been able to deliver to their full potential – simply because our equipment has two possible functions: refrigeration and helium liquefaction. When we manufacture our equipment, we manage to achieve a clever balance, satisfying both our liquefaction customers, research laboratories above all, and those requiring refrigeration, mainly the space industry and cryogenic synchrotrons.

Improving HELIAL performance means making a choice, and the range will therefore be split in two. With more powerful turbines in the system, we can

therefore increase liquefier performance twofold. In plain language, we can produce twice the amount of liquid helium. If the size of the exchangers is increased, refrigeration performance will be enhanced.

But that's not the only thing. We are also working on the compactness of our equipment, on lead times and on costs etc., while continuing to offer state-of-the art technical know-how. We have also sought to gain complete mastery of the manufacture of turbines, the key components of HELIALs, and of testing them in real conditions, as a guarantee of reliability. Finally we are expanding our maintenance services etc., in the interests of ever-increasing customer satisfaction.

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*The Cryoscope* is published by the Air Liquide Advanced Technologies Division • BP 15 • 38360 Sassenage • Tel. : +33 (0)4 76 43 62 11 • Fax : +33 (0)4 76 43 62 71 • E.mail: gcom.dta@airliquide.com • Director of Publication: Didier Magnet • Coordination: Sébastien Lefèvre • Delegate Publishers: 2ème Communication - Groupe Publicis • 22, rue Seguin • 69286 Lyon Cedex 02 • Tel. +33 (0)4 72 41 64 84 • Photos: Air Liquide, DR • Printing: Imprimerie Lamazière • 165, avenue Franklin-Roosevelt • 69153 Décines • ISSN 1270-4369
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