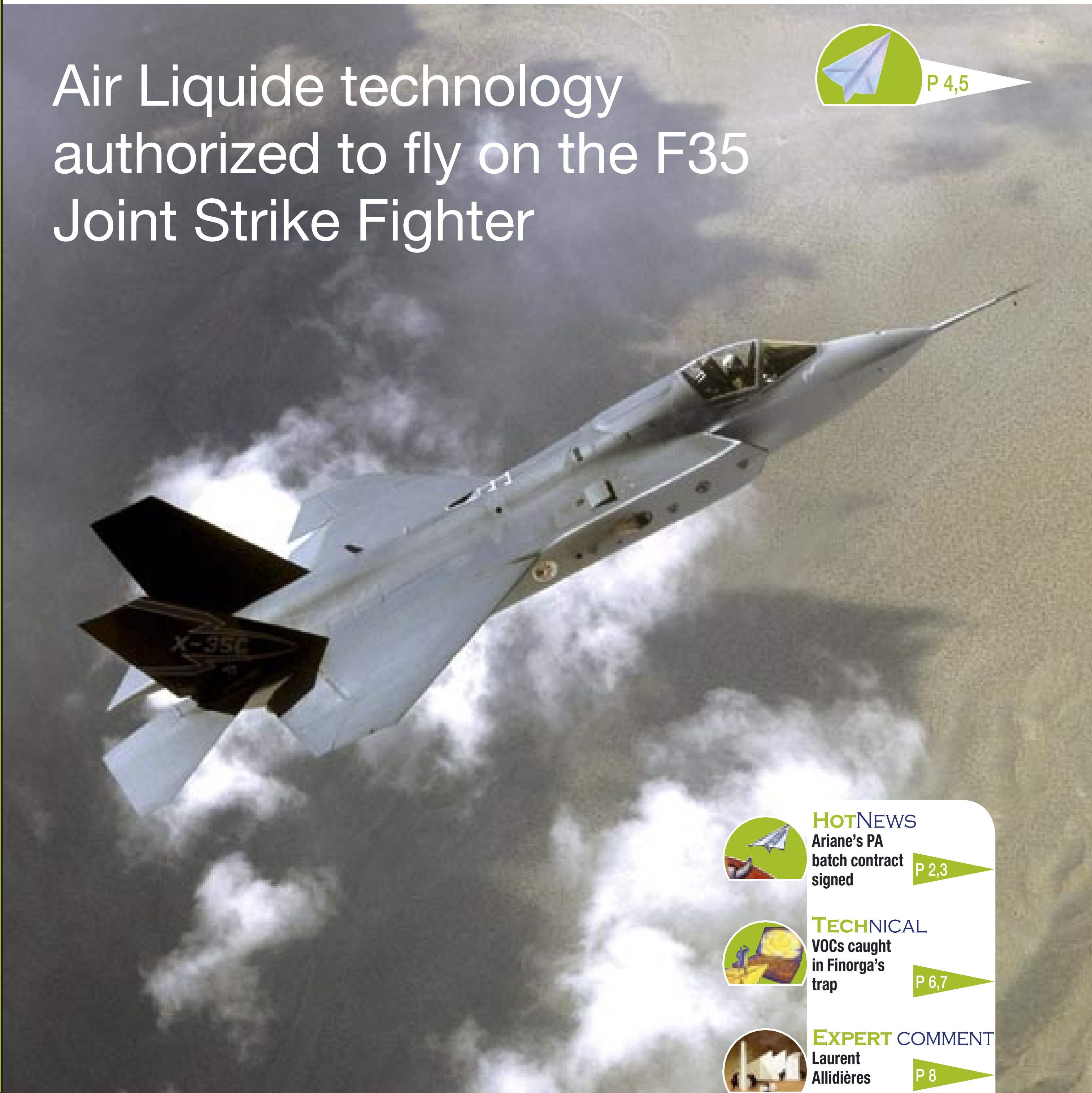
AIR LIQUIDE
TECHNIQUES AVANCEES

Quarterly – 2006 – No. 31

Le Cryoscope



Air Liquide technology authorized to fly on the F35 Joint Strike Fighter

P 4,5

Ariane's PA batch? It's signed!

Following successful flights in February and November 2005, the qualification phase for the ECA Ariane 5 is complete, and commercial launches will now increase. At the end of December, the PA batch contract (30 launchers), announced by EADS at the 2003 Le Bourget Show, was turned into a firm order for the Advanced Technology Division (DTA), and this will mean a large volume of work over the next five years. DTA will deliver the liquid oxygen tanks for the upper stages of twenty-five Ariane 5 launchers. Of thirty launchers, twenty-five are of the ECA type (with an upper stage ESC-A) and five of the GSTAR type (with an upper stage using storable propellants). Additionally, for the thirty launchers, DTA will deliver 30 SSHel (Liquid Helium SubSystems) to pressurize the upper stage oxygen tank. The delivery rate agreed between DTA and EADS is about four oxygen tanks and five SSHel every year until 2010.

The Liquid Helium SubSystem which will pressurize the main stage oxygen tank in the next thirty Ariane launchers.

Nitrogen for helicopters

In 2005, five Mobile Nitrogen Gas Generators (GMAG) were ordered by the French Defense Ministry's integrated aeronautical support structure (SIMMAD), responsible for keeping National Defense aerial equipment in a state of operational readiness. This brings to fifty the number of units delivered since 1997, the date the generator developed by DTA qualified to satisfy the operational constraints of the armed forces. This air-portable generator produces (from the air and at a rate of 12 m^3/h) pure nitrogen (99.5%) for aeronautical application at 385 bars of pressure. It replaces a liquid nitrogen production and storage line which required significant logistical resources in order to ensure the availability of aircraft and on-board weapons systems. The experience acquired by the Air Force technical team for aeronautical gases meant that the GMAG has been developed so as to increase its availability. The French Army has noted the reliability of the equipment and is now interested in order to meet the needs of its Light Aviation Unit (ALAT), responsible for helicopter inter-operability. About fifteen units should be delivered by the end of 2008.

A Mobile Nitrogen Gas Generator for aircraft and on-board weapons systems.

Analysis under control at Texas Instruments

At the end of December, the most impressive Continuous Quality Control (CQC) system to be designed by DTA was delivered to Texas Instruments for a microprocessor production unit in Dallas (USA).

Shawn Bratt, Project Manager (INGA department) and Ken Grosser, Quality Manager at Air Liquide Electronics US (in the background).

This set of gas analyzers is intended to guarantee the purity of the fluids which Air Liquide US will ultimately deliver to the Texan site. The process control system is adapted to our customer's specifications and takes up six cabinets, i.e. twice as much as the systems usually put together. Another feature is centralized computer control of all the gas analyzers. At the request of the customer, the batches have been put together to optimize the hunt for impurities in the process gases. Detection thresholds to be observed for nitrogen, oxygen, hydrogen, argon, helium and air are 0.2 ppb.

A trouble-free journey for Herschel and Planck

It's been official since December: in 2008 Ariane 5 will launch into space both the Herschel space telescope, which aims to understand the mechanics of the formation of the galaxies and the stars, and the Planck Science Observatory, which is tasked with analyzing fossil radiation and determining the age of the universe. Meanwhile, and for Herschel, at the ESA's test Centre at the ESTEC, teams from EADS Astrium have completed thermal tests on the superfluid helium cryostat, which brings together equipment supplied by the Air Liquide DTA.

It includes tanks and helium lines, thermal links for the instruments, internal thermal screens of the cryostat. Vibration tests are scheduled for early in 2006.

As regards Planck, the flight model of the dilution refrigerator designed by the DTA was qualified in October 2005, then delivered to the Space Astrophysics Institute. This is the structure in which the HFI (High-Frequency Instrument) is to be integrated. The cooler, which has to cool the infrared detectors to 0.1 K (one tenth of a degree from absolute zero), with a helium leakage rate 1,000 times less than in a conventional system, has reached the required level of performance. Once the HFI is integrated by the IAS, the unit will return, in June 2006, to the premises in Cannes of Alcatel Alena Space, the project manager for Herschel and Planck.

At the CRETA (CNRS Grenoble), Jean Delmas, test engineer for the Planck program, standing in front of the test cryostat. The dilution cooler is in the lower vacuum chamber.

A sudden rush of orders for Rafale

The history of the on-board oxygen generation system (OBOGS) is bound up with that of the Rafale fighter plane, started in the early 90s by Dassault, at the request of the DGA's Aeronautical Programs Department (SPAe). Fifteen years later, in the second half of 2005, a contract was signed for the delivery of 59 systems, which will bring to 120 the total number of units delivered by the DTA by 2010. This huge order confirms that the Rafale program has now reached a stage of maturity.

With the development of this first on-board aircraft equipment, the DTA has earned its stripes as a system builder. Since then, the OBOGS has been expanding in other markets such as civil aviation and military transport and new prototypes were presented at the 2005 Paris Air Show. OBOGS is a piece of equipment for which the DTA has had very positive feedback, which no doubt explains why it is proving attractive to new players in the Defense sector (see the report on the American F35 on pages 4 to 5).

ITER underway

The European Fusion Development Agreement (EFDA) responsible for managing the scientific parts of the ITER project, has approached the CEA and the Air Liquide DTA with a view to a qualification to conduct future tests on materials which will be used when the experimental thermonuclear fusion reactor is built at Cadarache.

For a few years now, the CEA and Air Liquide have been building up joint expertise in the characterization of ultra low temperature materials (4K). The former assumes responsibility for physical measurements and the DTA for mechanical characterization (traction, fraction mechanics).

This qualification phase is supported by the Karlsruhe Research Centre, FZK, and its aim is to validate laboratories capable of conducting these characterizations at ultra low temperatures, according to procedures set by ITER. There are two issues for the DTA – tracking changes in standards and maintaining a very high level of materials characterization.

An operator sitting at the Air Liquide DTA test bench for the mechanical characterization of materials.

Air Liquide recently hit its last major milestone in the development phase of the US government's Joint Strike Fighter aircraft programme. The company's Service Connection Package, part

Military aviation

Service Connection Package

Air Liquide has successfully completed its role in the development of the Joint Strike Fighter (JSF), the huge US-led project to produce a flagship next-generation military aircraft for America and its close allies. The company's Service Connection Package (SCP), part of the aircraft pilot's Life Support System (LSS), has come through a gruelling test programme on schedule, and been cleared for safety of flight, as has the LSS itself. Certification of the SCP and LSS is now in progress to support the first flight of the JSF in August 2006.
Air Liquide's Advanced Technologies (DTA) was chosen in 2001 to develop the SCP by Honeywell Aeronautic Yeovi, an England-based subsidiary of US avionics group Honeywell and the LSS prime contractor.[1] The decisive factor in the deal? The world-beating performance of Air Liquide's design, thanks to an unchallenged step-change technology (see box). The appeal of Air Liquide's SCP is highlighted by the fact that very few French companies are taking part in the JSF project.
The JSF project consists of three mainphases: prototype development (2001-04); equipment development, safety-of-flight testing, and in-flight testing (2005-07); and lastly, the start of series manufacture in 2007.
The SCP test programme that Air Liquide successfully completed in

The SCP is fitted to the side of the ejection seat, and continues to provide oxygen to the pilot if he ejects.

A Superbly Competitive Product

Air Liquide is one of three companies that develop and manufacture SCPs for use in military aircraft. The device, combined with an OnBoard Oxygen Generation System (OBOGS) and a Backup Oxygen Supply to form the LSS, is fixed to the pilot's seat. It regulates the flow and pressure of the gases the pilot needs, e.g. oxygen-enriched air to breathe, and pressurised air for the G-force-fighting bladders in his/her jacket and pant.

Honeywell selected Air Liquide's SCP for the unmatched performance of its exclusive electro-mechanical design. In mechanical-only designs, the valves are operated by pneumatic pressure: this varies greatly according to altitude and in some flying conditions can be insufficient, especially for the pressurised-air functions. With Air Liquide's SCP, incoming air pressure is not a factor. The electronic controls manage distribution with greater speed and precision, and can be preprogrammed for variable – rather than just linear – in-flight operation.

In addition, Air Liquide's SCP facilitates prognosis and health management. All parameters are measured electronically and thus easier to monitor constantly. To monitor such parameters, a mechanical-only device would require a set of sensors separate from the SCP – making the Life Support System more complicated and costly.

of the aircraft's Life Support System, has achieved safety-of-flight clearance after passing an extremely demanding test programme – on schedule.

Air Liquide's world-beating Service Connection Pack, the only electro-mechanical design on the market.

cleared to fly on the F35

the fourth quarter of 2005 marks the end of its contribution to the JSF's development. *"These tests were very important because they green-lit our Package for in-flight testing – the final stage in the validation of systems performance,"* explains Richard Zapata, Head of the Aeronautics Department. *"By meeting all our deadlines and performance targets, we've repaid Honeywell's faith in us."*

The SCP test programme was shared by Air Liquide and its client: the DTA site in Sassenage, France, checked the Package's performance in the rated temperature range; and Honeywell Normalair Garrett conducted other environmental tests at its Yeovil plant in south-west England. (See box). *"This is the biggest, most stringent set of tests our SCP has been subjected to,"* says Project Manager Didier Gaget. *"Staying in-spec was a tremendous challenge."*

In some respects, Air Liquide proposed features that actually exceeded the client's initial requirements. For example, if the pilot's pant or jacket fails and depressurises, the SCP automatically equalises the pressure in the other garment: this enhances safety, as any gap can affect the pilot's blood circulation – thus impairing reactions and field of view.

Phase two of the project continues. The LSS, including Air Liquide's SCP, is now undergoing man-rated tests in Texas in a centrifuge and altitude chamber, prior to in-flight testing in August. For Didier Gaget and his team, the four-year development push is over; now the low-rate initiation production phase is under negotiation.

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Pushed to the limit

During the fourth quarter of 2005, Air Liquide's Service Connection Package underwent an exhaustive series of tests to achieve safety-of-flight clearance. These were non-man-rated tests – i.e. using breathing simulators, not humans – and the accelerations and altitudes were simulated electrically. The specifications were all the more demanding because the component had to meet the specifications for three JSF variants (conventional, short take-off and landing, vertical take-off and landing).

At Sassenage, all performance parameters were tested in the rated temperature range (-55°C to +71°C). Would the Package deliver the pressures and flows as programmed? Would its integrity be unimpaired by storage?
Honeywell's team in Yeovil verified – by testing or analysis – that the Package would continue to operate correctly in a wide range of adverse environments. One of the most challenging tests was the explosive decompression test.
If a bullet strikes the cockpit, cabin pressure drops suddenly: the gap between cabin pressure and pilot-mask pressure will widen, and the mask pressure will in effect increase. The SCP must react within 1/20th of a second – bleeding the system before the pressure rises too far – or the pilot's lungs will explode. Air Liquide's Package saved the "pilot"… and passed the test!

In situations with low throughputs and high concentrations in solvent, cryogenic condensation is ideal for trapping the Volatile Organic Compounds arising out

The gaseous effluent treatment plant at the site at Mourenx (Pyrenees Atlantiques) is nine meters high

Cryogenics

VOCs trapped by Finorga

At Mourenx in the Pyrenees Atlantiques, Finorga(1) is manufacturing active ingredients for the pharmaceutical industry belonging to the classes of neuroleptic drugs, antidepressants, calcium inhibitors and beta-blockers. Given the use of solvents in obtaining these and in order to comply with regulations relative to the discharge of gaseous effluents, Finorga was keen to equip itself with a plant that would allow it to recover the Volatile Organic Compounds produced by its production lines. *"The solution sought had to enable the continuous (24/7) treatment of discharges that vary in nature and throughput (between 600 Nm^3/h and 1000 Nm7 pts/h)"* explains Jerome Beauvisage, project leader at the Air Liquide DTA. *"Since Finorga wanted to optimize investment and operating expenses at the plant, its choice has fallen on our proposal, namely cryogenic trapping using VoxalTM, a high-performance cryo-condensation solution."*

Required agility

The plant has been in operation since April 2005 and arises out of the analysis of a number of criteria. First of all, the nature of the discharges from the hundred odd reactors where the active ingredients are developed – a mix consisting mainly of alcohols, aromatics, ketones, in variable concentrations of a few tens of g/Nm^3, and water.

These solvents are easy to trap and their total final throughput after treatment must be below 5 kg/h. Dichloromethane in fact posed more problems since it is a VOC targeted in appendix III (and therefore subject to more restrictive regulations). The throughput treated must be below 100 g/h. In fact, to attain this rate, it is necessary to operate at -120°C and therefore to use liquid nitrogen (-196°C), although it is not used continuously since dichloromethane is only present in the effluent at certain times.

Four cascaded traps

"To optimize the costs of treating VOCs, the plant recommended in the end by the Air Liquide DTA was one with four cooling levels" explains Arnaud Fossen (see diagram below)

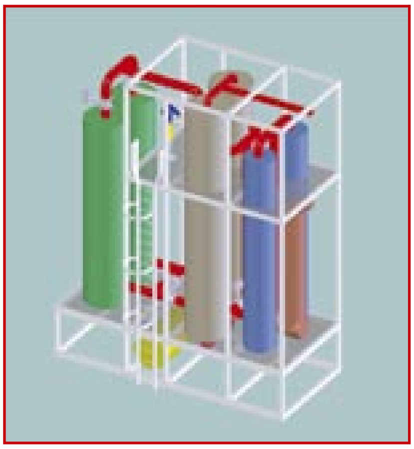

Diagram of the DTA installation for Finorga. The Finorga installation breaks down into four stages – in pink, the first stage; in blue, the second stage; in grey, the third stage; in green, the fourth stage; and in yellow, the recovery tank.

The first stage consists of a heat exchanger in which glycol water allows the temperature of the effluent to be brought down to 2°C. The VOCs and water liquefy on the exchange surfaces and are then recovered in a tank. In stage 2, the glycol water is there again as a coolant but in order to reach the temperature of -10°C in the effluent. At this temperature the residual trapped humidity is in a solid-state and the exchanger therefore has to be regularly defrosted, which is why, since in this case the air is arriving continuously, two exchangers are installed in parallel – when one is in a trapping phase, the second is regenerating by reheating.

At each successive stage, and for the same reasons, there are two exchangers operating alternately. This time, the temperatures to be reached are -40°C and then, where dichloromethane is present, -120°C. A cost-effective coolant solution has therefore been looked for. Given its temperature level, the last stage can only be cryogenic with the two cryo-condensers then operating alternately with liquid nitrogen. At the output of this stage 4, the effluent is at a temperature of -120°C and it is thus routed towards stage 3 where, when relieved of its VOCs, it acts as a coolant to trap the VOCs in the effluent coming from stage 2. This allows Finorga to reduce its liquid nitrogen consumption, which is why stage 3 is also known as the gas economizer.

of processes in the chemical industry. As shown by the example of Finorga where the type of VOC is variable, this technology is in line with optimized operating costs.

Accumulated solvents and water solidified on the tubes of a cryo-condenser

N2: useful twice over

The condensates trapped then liquefied during the regeneration phases are routed by gravity to a recovery tank in order to be stored prior to incineration.

As far as nitrogen is concerned, Marc Caillet, new operations manager at the Mourenx site, is in no doubt about the importance of the *"recovery of gaseous nitrogen for our production lines. This tailor-made plant exactly meets our needs."* Indeed, after releasing its cold energy at stage 4, the nitrogen passes into an atmospheric heater. At ambient temperature and at 3 bars of pressure, it is then re-used in inertage operations in the plant.

Finally it should be noted that all the components installed at Mourenx comply with the Atex II Gas standard (explosive atmosphere). In addition, Finorga now benefits from an annual on-site preventive maintenance inspection.

In the plant, a technician during the assembly phase of thermal isolation on the integrated ductwork.

(1) Finorga is a subsidiary of the Novasep Group.

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The technologies for treating VOCs

The presence of chlorinated compounds and associated CO2 emissions, the operating cost – just so many criteria to be taken into consideration before choosing a treatment type. To that end, it is important to identify the nature of the VOCs to be trapped, to determine the throughput and concentration of the emissions to be treated, and their variability over time. And of course, to know the forward trend for gaseous effluent generation.

Destructive techniques

Thermal incineration consists of oxidation by combustion between 600 and 1,000°C. This technique enables large throughputs to be treated (from 750 to 100,000 Nm^3/h). By using precious metals or metal oxides, catalytic incineration is carried out between 300 and 600°C but the throughput range treated is smaller: from 100 to 30,000 Nm^3/h. Because of restrictive operating conditions, biological treatment is not very widespread. In this case, the VOCs are transferred in liquid phase and metabolized by micro-organisms.

Recovery techniques

Adsorption on active charcoal enables large throughputs to be treated (from 100 to 100,000 Nm^3/h) at low VOC concentrations (0.05 to 50 g/Nm^3). Adsorption may also be carried out on zeoliths (silico-aluminates). For a throughput from 10 to 2,000 Nm3/h, with concentrations above 10 g/Nm^3, effluent can also be treated by permeation, through a membrane. Finally, condensation is often used in fine chemistry since it is suitable for low throughputs (from 0 to 2,000 Nm^3/h) and to high solvent concentrations (above 10 g/m^3). Its principle is to lower the temperature so as to condense the solvents until the tolerated concentration is obtained.

Since it can react in a fuel cell or directly in an internal combustion engine, hydrogen can bring some major advantages when applied to the transport field. Interview with Laurent Alldieres, manager of hydrogen energy programs at the DTA.

The hydrogen service station set up in Madrid under the CUTE project. An installation signed Air Liquide and Repsol YPF.

Hydrogen Energy

To be a supplier of global solutions

Is hydrogen a solution which will solve our energy problems?

It's still too early to be certain but the hypothesis cannot be discounted. As we speak, hydrogen is being produced from natural gas or from the electrolysis of water. There are solutions for capturing CO_2 and generating green electricity. During combustion – either catalytic in a fuel cell, or directly in an internal combustion engine – hydrogen only generates water. All of which makes it a totally clean energy carrier. It also has the advantage of being able to be produced at local level, which in the end promotes self-sufficiency in energy. Some countries have understood this well. Iceland, for example, has already started its transition towards a hydrogen economy. As regards the European commission, it has set itself an objective of 5% of vehicles driving on hydrogen by 2020.

What is the position of industrialists?

Industrialists are also taking a close interest in hydrogen, as an energy carrier. Oil companies are financing a number of projects. It is in their interest to anticipate the likely development of their business as energy distributors. Fossile fuels will be replaced tomorrow by clean renewable sources likely to meet all the objectives of self-sufficiency in energy and respect for the environment.

What are the issues for Air Liquide DTA in all this?

Our hydrogen sales, which stand at €650 million in 2005, should double between now and 2008. What is more our group has the largest network in the world at its disposal, with 200 production units and 1,700 km of pipelines. From natural gas [1] to the fuel cell, Air Liquide has developed a great many solutions[2]. We are present at all levels in hydrogen chain. Every day, we have a thousand employees working on hydrogen alone. We are in a forward-looking phase and we need a vision of what will happen over the next few years. The task before us is therefore simple – to play an active part in decision-making networks and to ensure our level of expertise is made to count.

Lauent Allidieres seen demonstrating how to fill up a car fitted with a fuel cell at Lake Jackson (Texas) in December 2005.

How far have the European projects got?

The CUTE project[3] which involved nine towns in the EU as well as Reykjavik and Perth (Australia) ended in October 2005. The DTA helped set up the service stations in Luxembourg, Madrid, and part of the one in London, in 2002.

A new phase known as "Hyfleet Cute" has started, but in this too, we are a partner. It is one of the world beacon programs for hydrogen energy, and a formidable opportunity for us to be one of the major players in industrial area as a supplier of global solutions.

(1) Natural gas enables 80% of the available hydrogen to be generated.

(2) Cryogenic flat tanks, storage solutions at up to 700 bars of pressure, service stations enabling a tank to be filled in three minutes for 400 km of self-sufficiency

(3) CUTE Clean Urban Transport for Europe. See report in the Crysoscope *28.*

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