Cleaning and consolidation of the CMS helium refrigerator after hydrocarbon contamination

Caroline Fabre on behalf of the TE-CMS cryo task force

With contributions from: Johan Bremer, Udo Wagner

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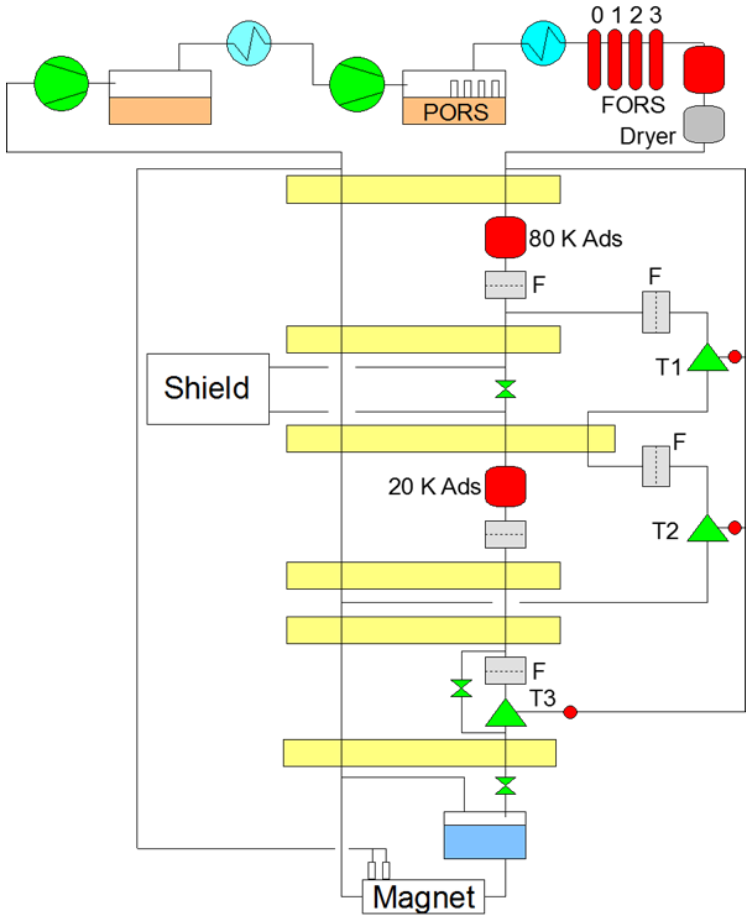
- Refrigerator characteristics
- 2015 symptoms
- Causes of contamination
- Consolidation of oil removal system
- Cleaning of cold box
  - agent selection and qualification,
  - methods and procedures,
  - control points
- Results
- Lessons learnt

The CMS refrigerator

- 1.5 kW @ 4.5 K equivalent
  - 800 W @ 4.5 K
  - 4500 W @ 60 / 80 K
  - 4 g/s current lead supply

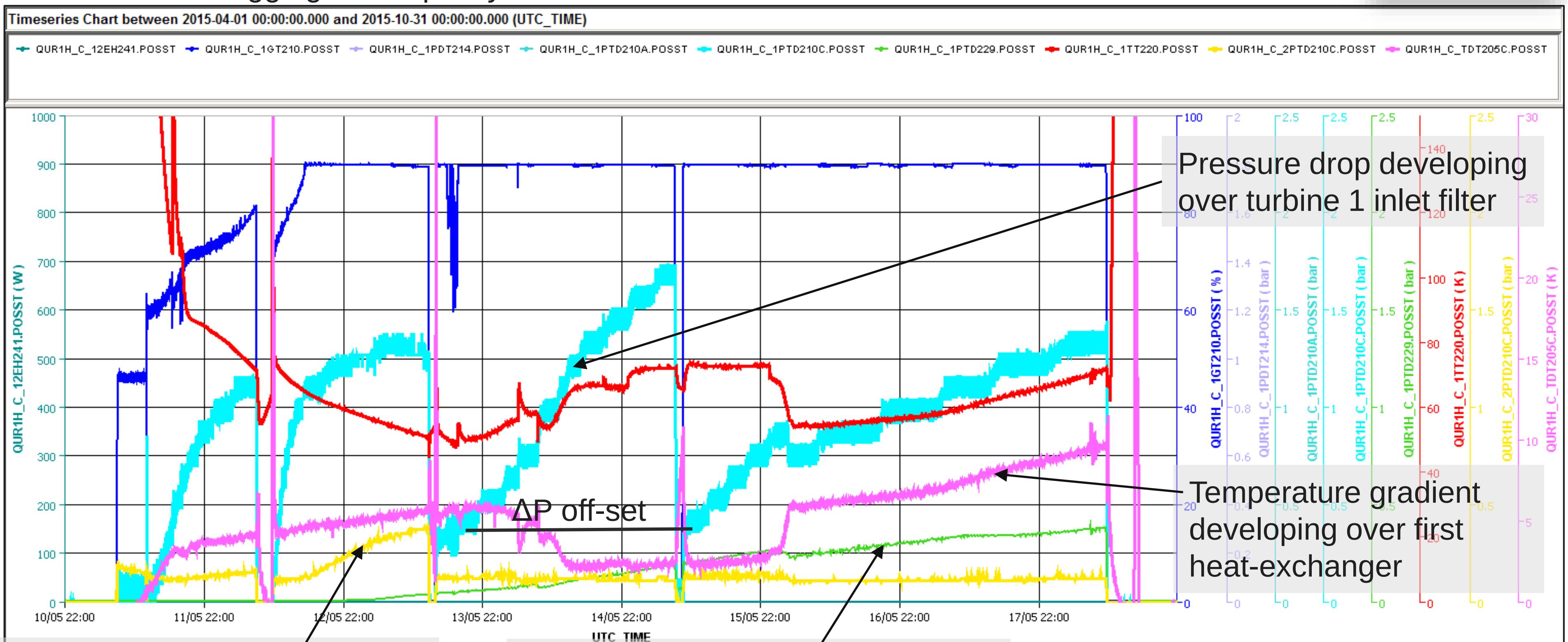
- Compressor station:
  0-18 bara; 220 g/s

- Operated for ~10 years with only minor impurity problems



2015 observed symptoms

2015 cold box clogging and capacity evolution



Pressure drop developing over turbine 1 inlet filter

Temperature gradient developing over first heat-exchanger

Pressure drop developing over turbine 2 inlet filter

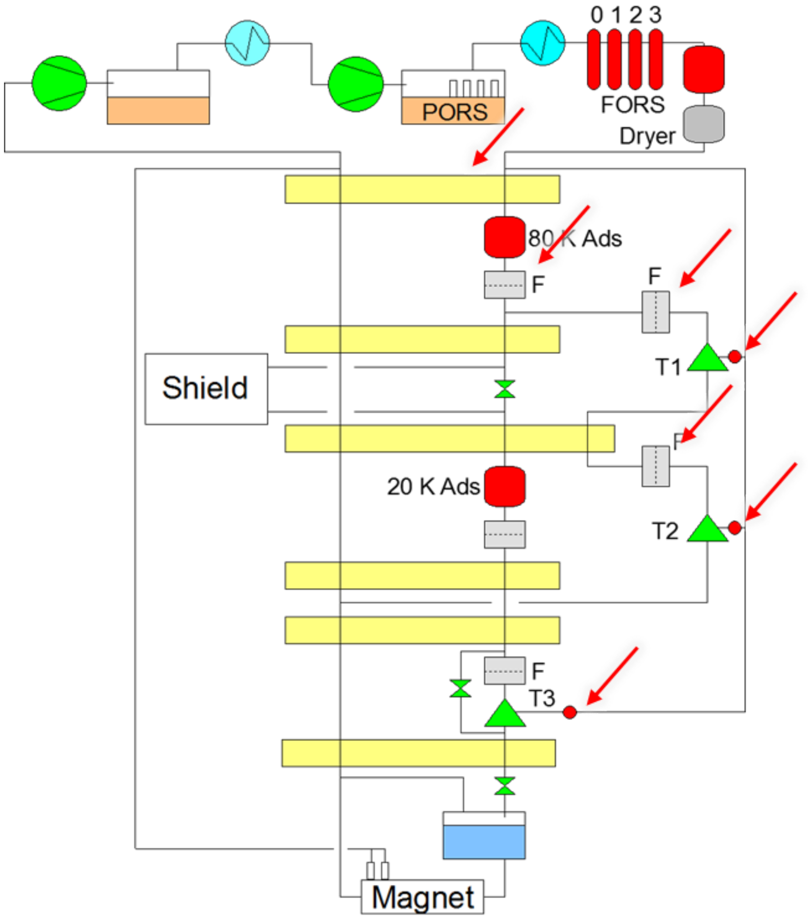
Pressure drop developing over 80K adsorber outlet filter

Oil contamination

- A considerable amount of Breox® B35 traces was found on
    1. Outlet filter 80K and 20 K adsorbers
    2. Inlet filter T1
    3. Inlet filter T2
    4. Turbine gas bearing inlet filters
- Breox® is thought to diminish the heat exchange surface of the first heat-exchanger.

At the same time:

- No visible oil traces on piping surfaces
- Contamination level measured downstream of the oil adsorber:
    - ➢ Order of magnitude (few ppb(w)) compatible with the acceptance limit of a new installation
    - ➢ No significant signal would be expected at all …



Identified causes

The peak of Breox® contamination results from the cumulated effect of:

1. Under-sizing of the oil separator

    Known from 2004 commissioning:

    - Oil carry over from oil separator ~ 750 ppm(w)
    - CERN specification: < 100 ppm(w)

    Compensatory measure = add a 4^{th} coalescer

2. Change of coalescers cartridges brand during 2014 maintenance

    Claimed to give equivalent remaining oil aerosol contents

    However, a factor 4 increase in the separated amount at the last coalescer is measured

3. Change of oil adsorbent material from activated charcoal (coconut shell) to activated coal (anthracite) during 2014 maintenance

    Intent: reduce dust, change shape from broken to pellets

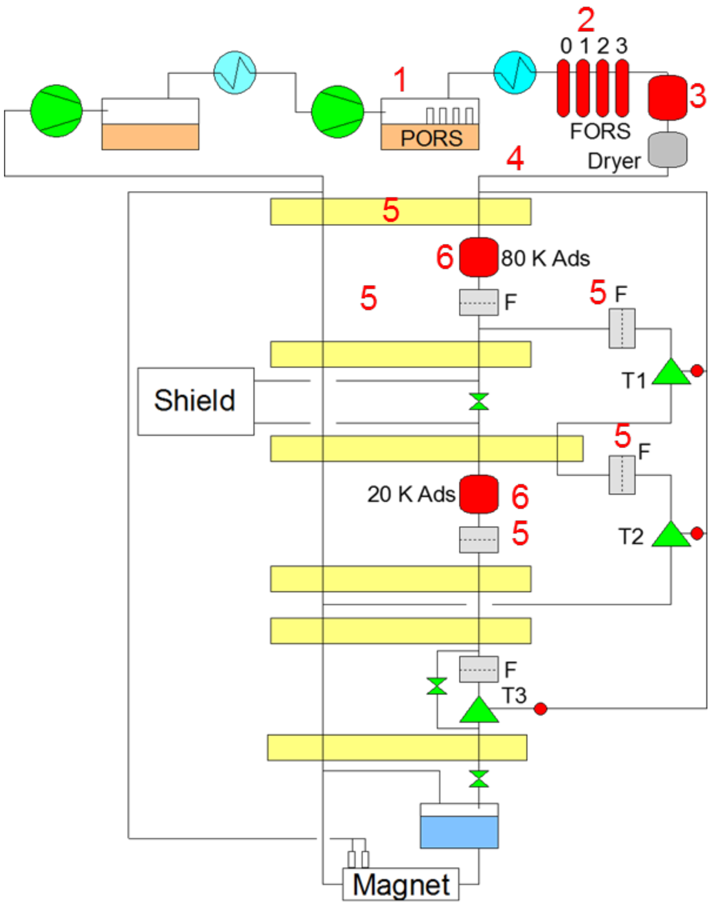
    Cost driven selection

2015 degraded operation

- 2015 average availability: 52%
  - The installation was kept alive with regular 80K adsorber and turbines inlet filter regeneration and replacement and partial warm-up sequences of the 1st heat exchanger.
  - Normally a cold-box having suffered such a Breox pollution is stopped to be cleaned. This was however impossible in the CMS case, so we have worked on a continuous operation.
- Consequences for the CMS experiment of the cold-box problems:
  - Of the integrated (p-p) luminosity delivered to CMS, about 73% of the data is taken under nominal field conditions;
  - The interventions on and regenerations of the CMS cryogenic system have lead to 12 magnet ramps

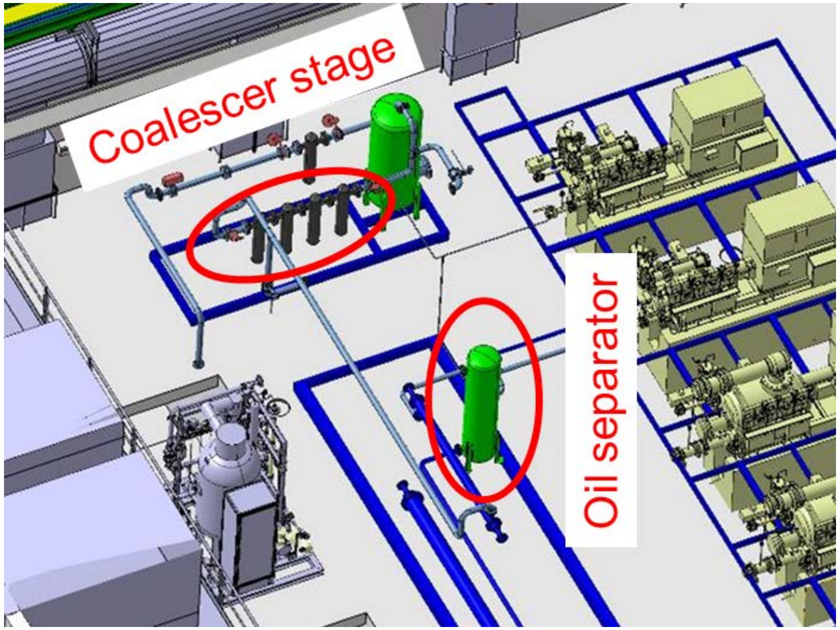
Consolidation YETS* 2015-16

1. Exchange the final oil separator
2. Exchange the coalescer stages
3. Exchange the oil adsorbent material
4. Exchange the high pressure piping between compressor station and cold-box
5. Clean the cold box circuits
6. Exchange the 20 K and 80 K adsorbers

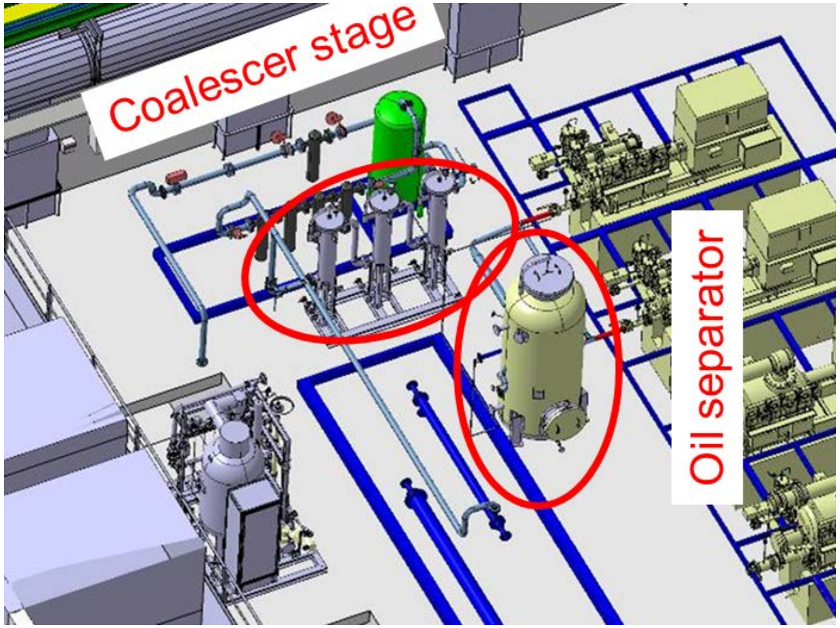


*YETS: year end technical stop ~3 months

ORS consolidation



Original ORS



New ORS

On purpose generous design to ensure low oil carry over from the separator

Result (estimate from measurement after 1000h) : ~ 20 ppm(w)

Cold-box cleaning

- History recall:
  - ➢ One major accident of oil contamination in 2004:
    ~ 2L accumulated in the cold box
  - ➢ System installed at the surface for commissioning
  - ➢ Cleaning agent: isopropyl alcohol
  - ➢ Very difficult drying afterwards: ~1 month

- 2016 cleaning:
  - ➢ <u>In underground environment</u>
  - ➢ Cleaning agent: DuPont™ Vertrel® XF ($C_5H_2F_{10}$)

    Density: 1.6 kg/L @ 25°C

    Boiling point: 55°C @ 1013 mbar

    Vapor pressure: 301 mbar @ 25°C

Solvent selection and qualification

- Criteria for the selection of Vertrel® XF
  - No flammability range (personal safety in the case of underground operation)
  - Low toxicity, environmental compatibility
  - Miscible with Breox® B35 (cleaning efficiency)
  - Easy to dry out of the system after cleaning (~rate of evaporation):
    - high vapour pressure (301 mbar @ 25°C),
    - low latent heat (136.5 kJ/kg @ 25°C)
  - Compatible with cold-box materials (metals, plastics, elastomers)

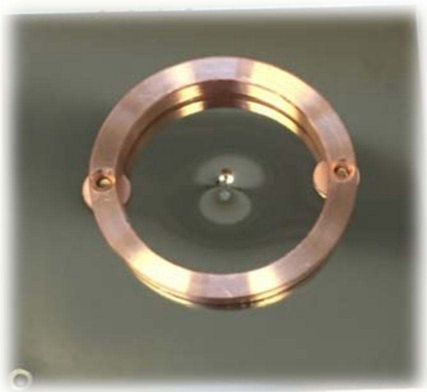
→ Safety measures: trained personnel, PPE, air extraction, spill containment tray, ODH detection

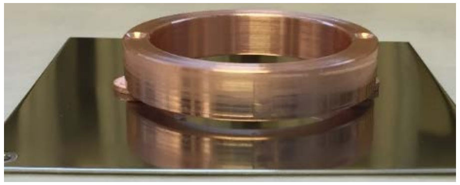
- Qualification tests with chemistry lab
  - Cleaning efficiency tests
  - Exhaustive inventory of plastics & elastomers present in the cold-box circuits
  - Immersion tests carried out on samples of each material to assess mass increase and degradation

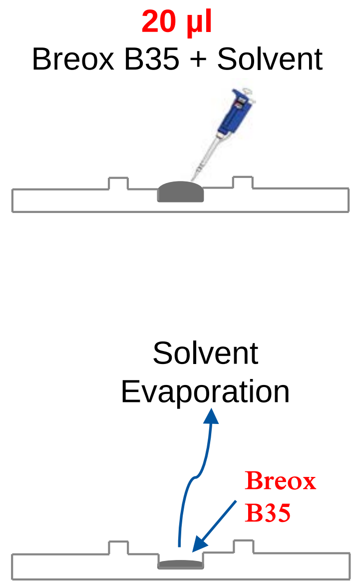
→ Only Viton turned out to be sensible to Vertrel® XF: mass increase (absorption) in presence of either liquid or vapor phase of Vertrel® XF. Exchanged at the end of cleaning.

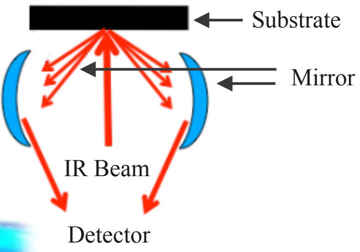
Cleaning quality control

- FT-IR In-Situ measurement of Breox® B35 in Vertrel® XF
  - Detection limit: 10 mg/L
  - Analysis time: 5 min.





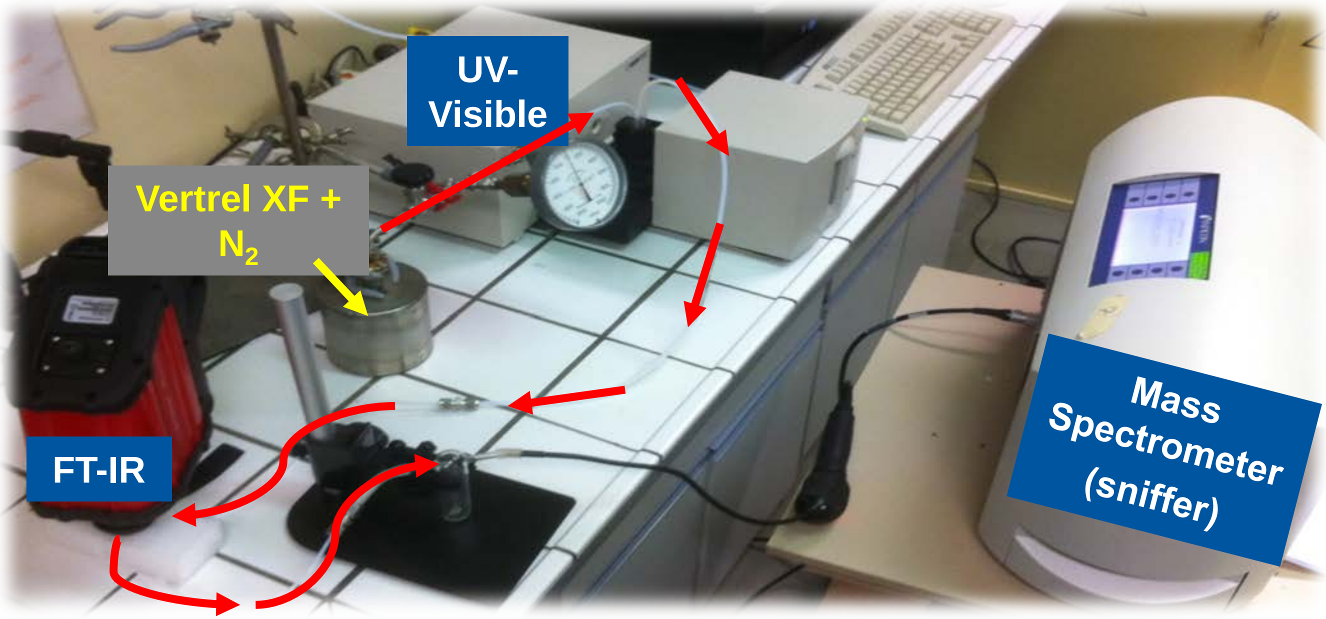




Courtesy: Benoit Teissandier,
CERN Vacuum, surfaces & Coatings group

Drying quality control

- 3 methods for measurement of Vertrel® XF in GN2
  - Detection limit: 2 ppm(v)
  - Analysis time: few sec.

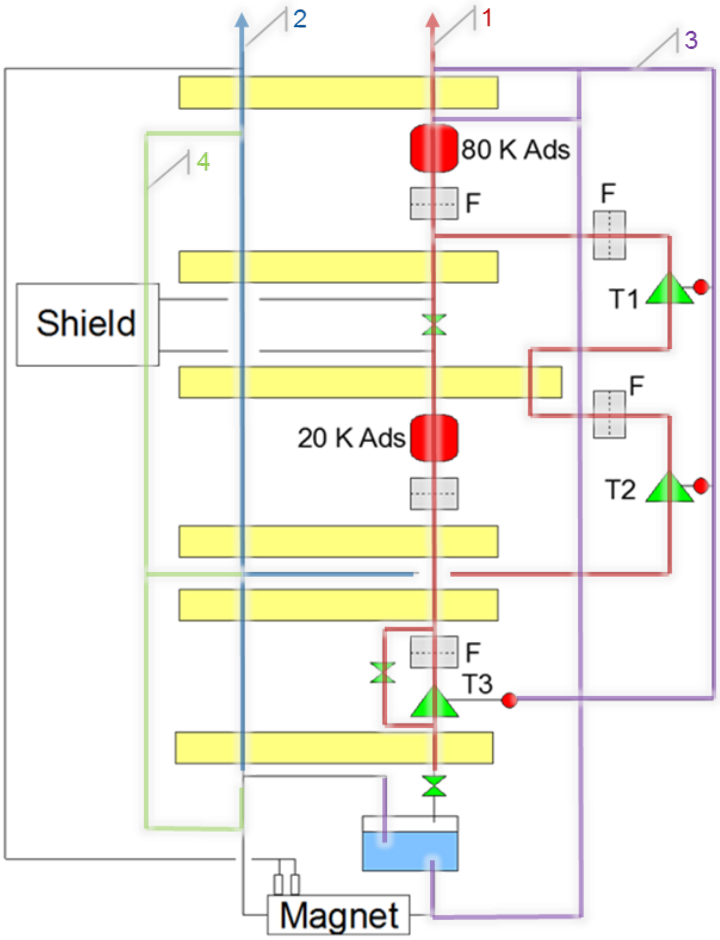


Courtesy: Benoit Teissandier,
CERN Vacuum, surfaces & Coatings group

Circuits volumes ≤ 950 L

A comprehensive cleaning

N°	Circuit	Justification
0	Purge collector	useful to cleaning process
1	HP	evidence of oil
	Turbines inlet-outlet piping	evidence of oil
	Precooler he circuit	likely presence of oil
2	LP	likely presence of oil. Possible contamination via cold-box warm-up procedure
3	HP to turbines' bearings	evidence of oil
	HP bypass (including ADS pressurization)	likely presence of oil
	Phase separator	Useful to cleaning process
4	Adsorbers regeneration circuit + LP cold return	likely presence of oil. Possible contamination via adsorbers regeneration

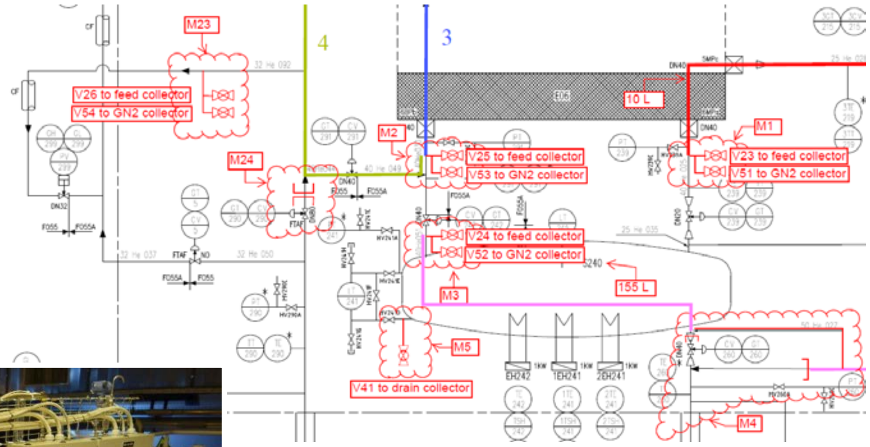


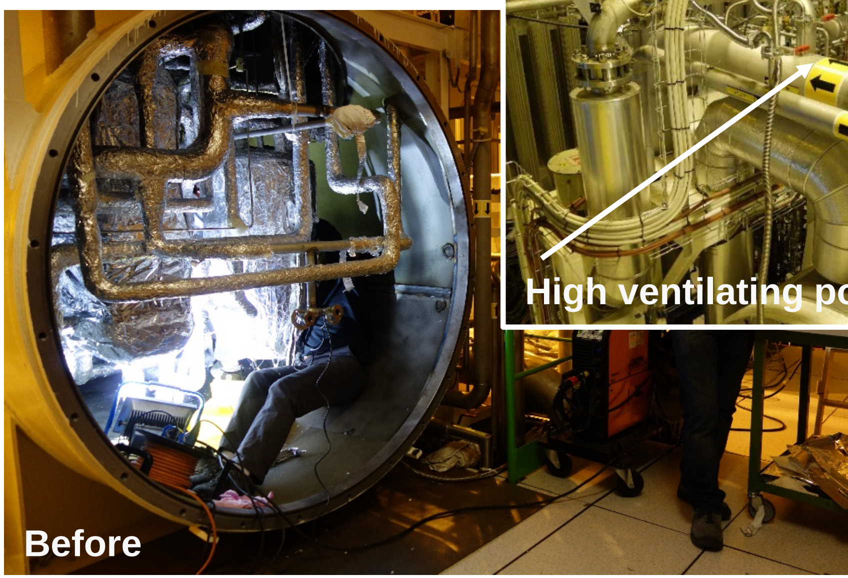
- Turbines' circuitry excluded
  (not a source of oil + small sections + risk of damage)
- Turbines returned & cleaned at supplier's premises

Cold box preparation (1/2)

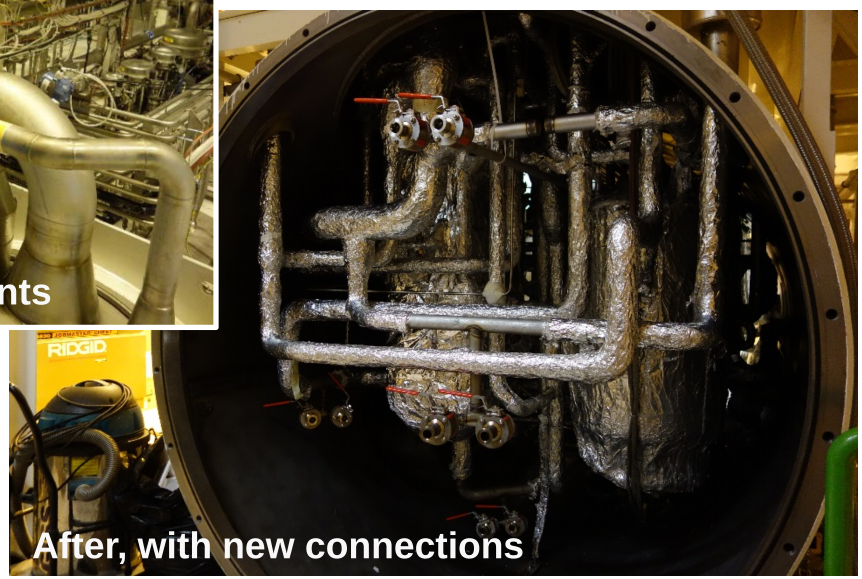
Heavy pipework modifications:

- New connections for solvent in/out
- New high ventilating points
- New gravity drain points
- **Lines towards client cut unless isolated by two leak-tight valves**




Before


High ventilating points



After, with new connections

Cold box preparation (2/2)

<u>Removed:</u>

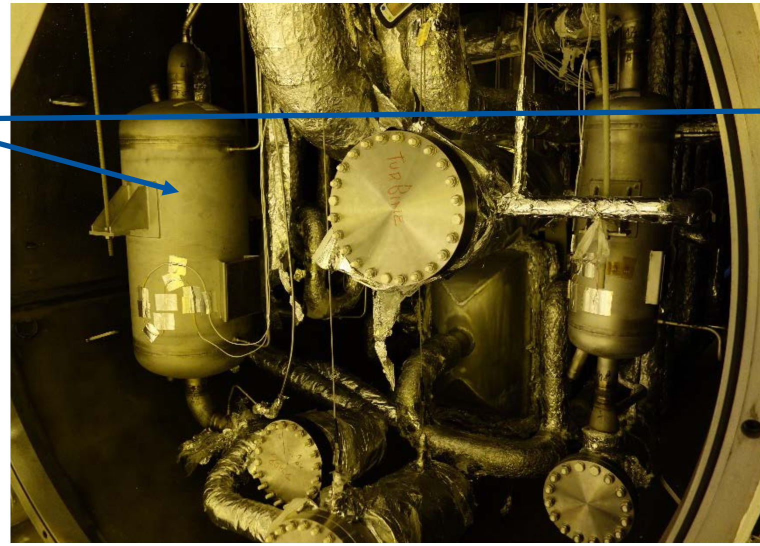
- Adsorbers (x2)
- Turbines (x3)
- Turbines instrum.
- Filter cartriges (x6)
- Lakeshore TE (x22)

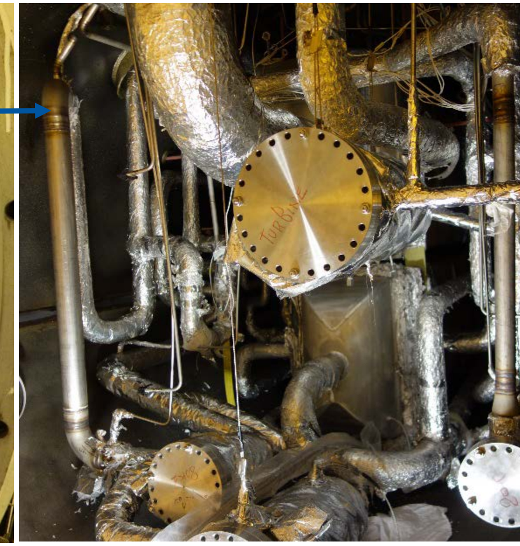
<u>Kept:</u>

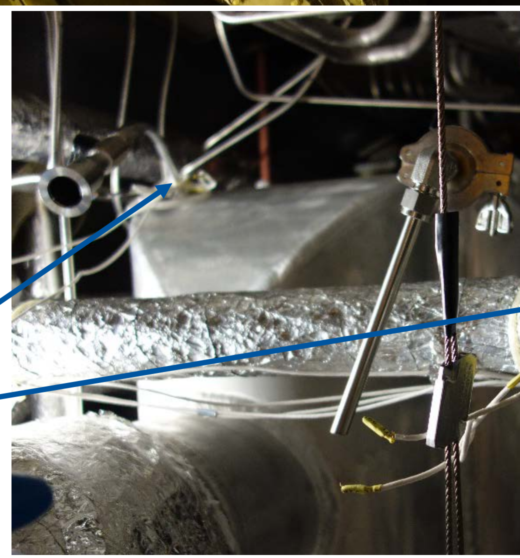
- Cryo valves (x20)
- Purge valves (x53)
- PT/DPT valve blocks (sensor isolated) (x15)
- Safety valves (x18)

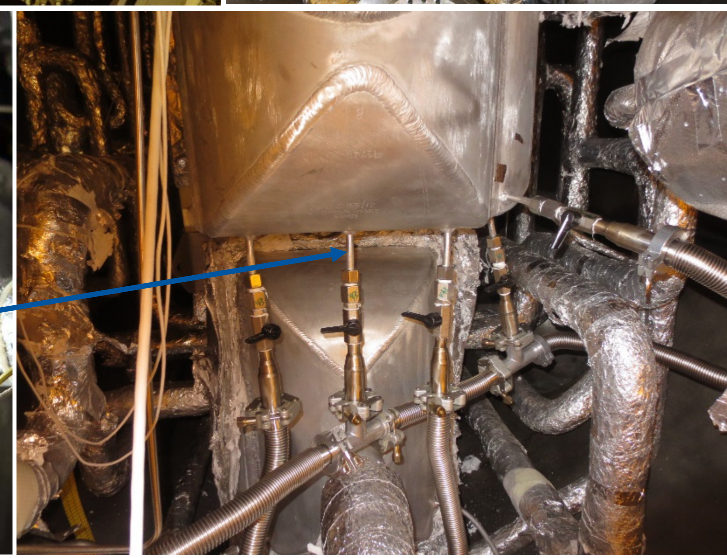
<u>Heat exchangers :</u>

- Add purge tapping
- Add drain tapping
- Shim to secure supporting



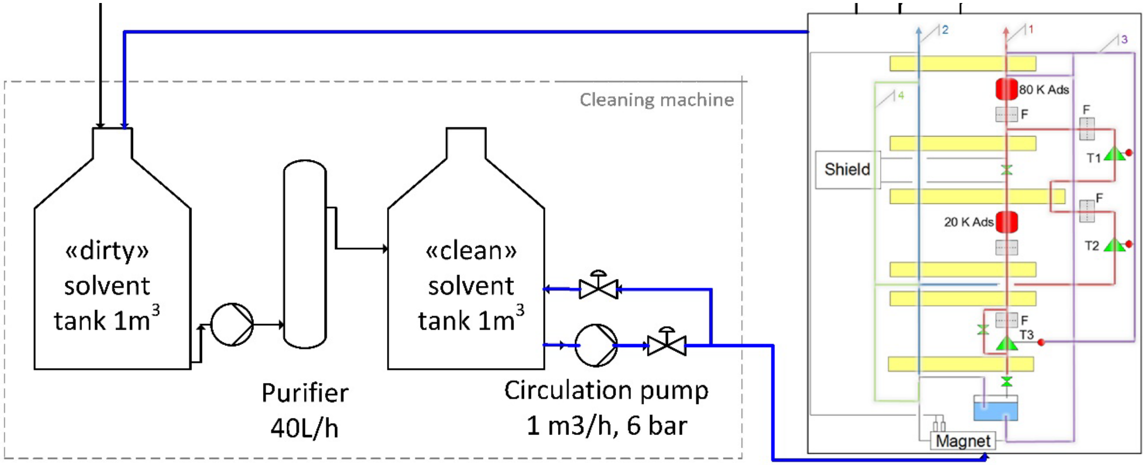






Cleaning method

- **Forced circulation of pressurized (2-4 bara) cleaning agent in the contaminated circuits**
- Flow direction from clean towards contaminated parts
- Circulate in parts in contact with process flow, NOT in dead ends (pressure sensors piping, cryo valve bodies)
- Flush circuit derivations / branches in sequence

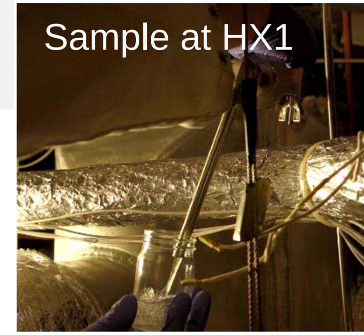


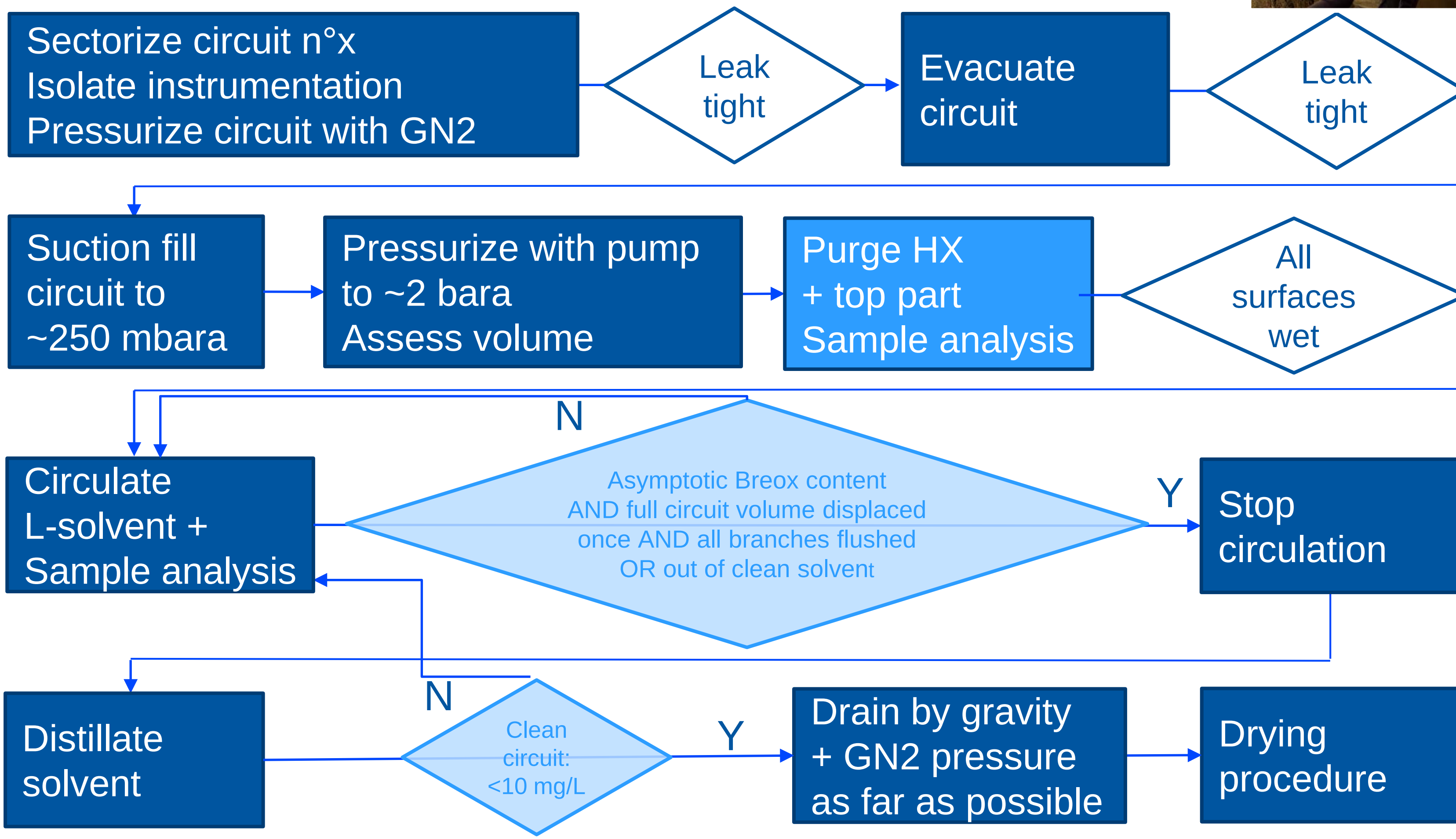
- "Cleaning" machine bought on purpose

→ Method & equipment validated beforehand on a test stand

Cleaning procedure
& control points

Duration per circuit:
2–3 working days

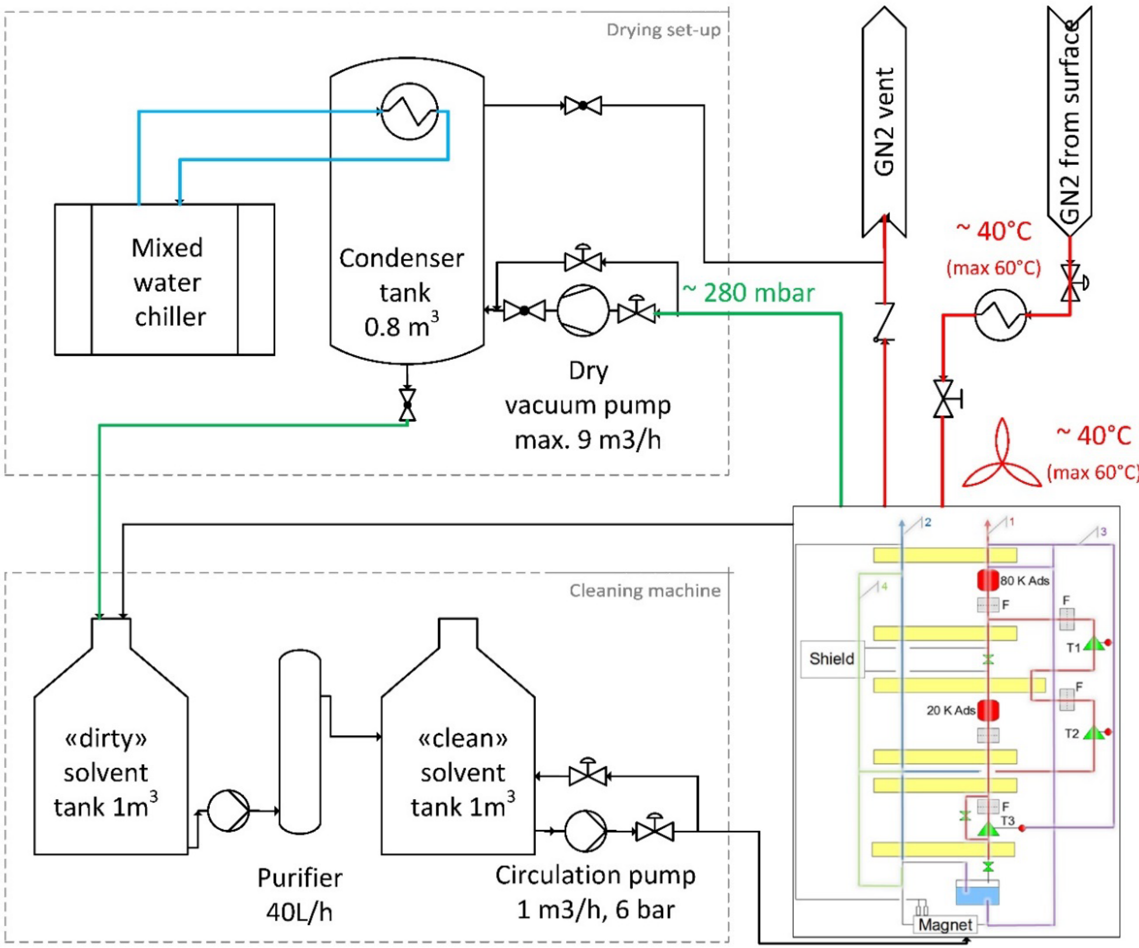




Drying method

1. **Vaporize the residual liquid-phase solvent after drain:**
   - Maintain temperature ~40°C:
     - ➢ Circulate warm gaseous nitrogen in counter-flow circuit (not the cleaned circuit)
     - ➢ Blow warm air through cold-box vacuum tank
   - Pump cleaned circuit down to P_{vapor}~280 mbar < P_{sat}
   - Re-condense vapors

   → Drying speed depends on free surface area
2. **Flush the residual vapor-phase solvent:**
   - Circulate warm gaseous nitrogen in the cleaned circuit
3. **Outgas under vacuum**



Drying procedure
& control points

Duration per circuit:
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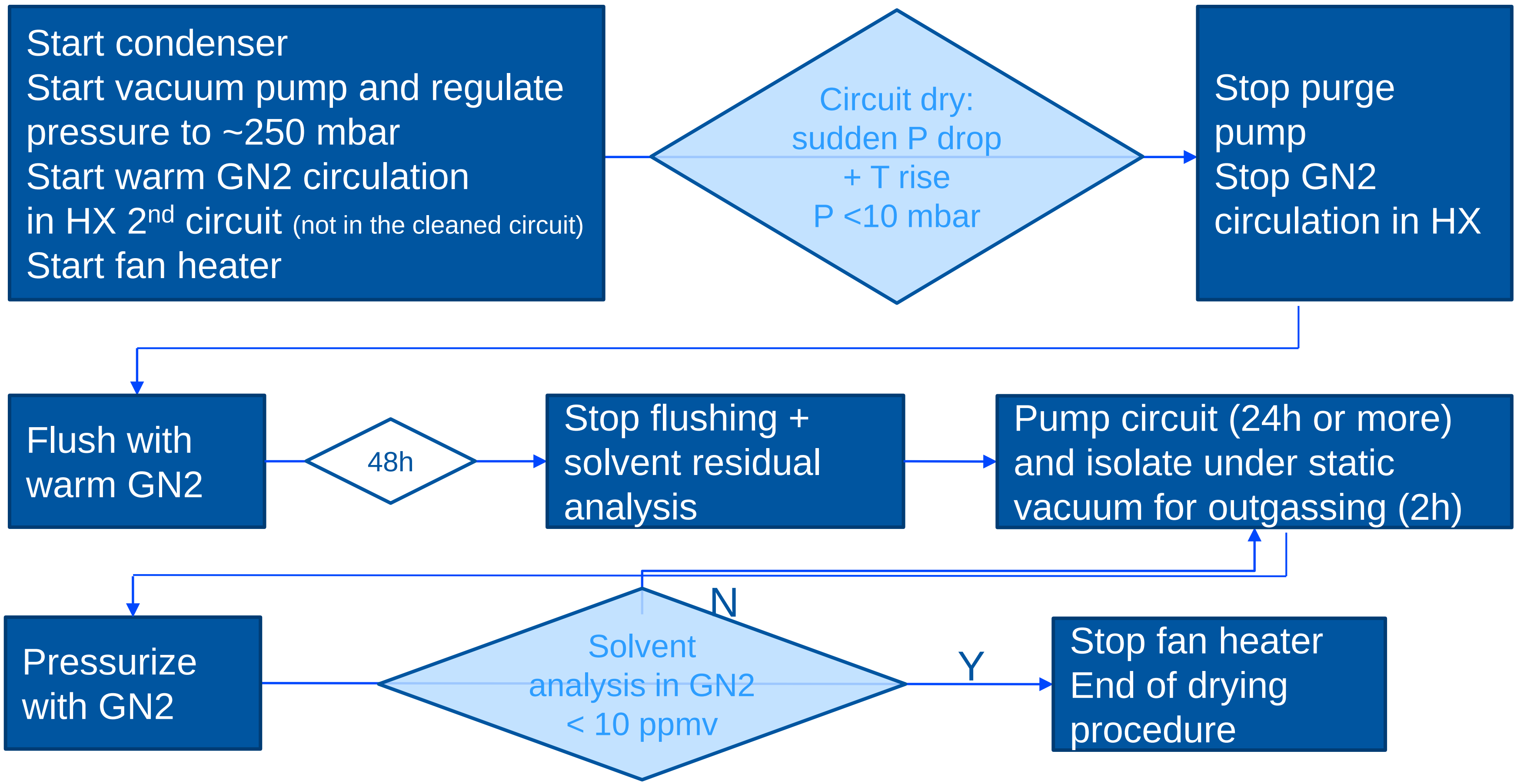
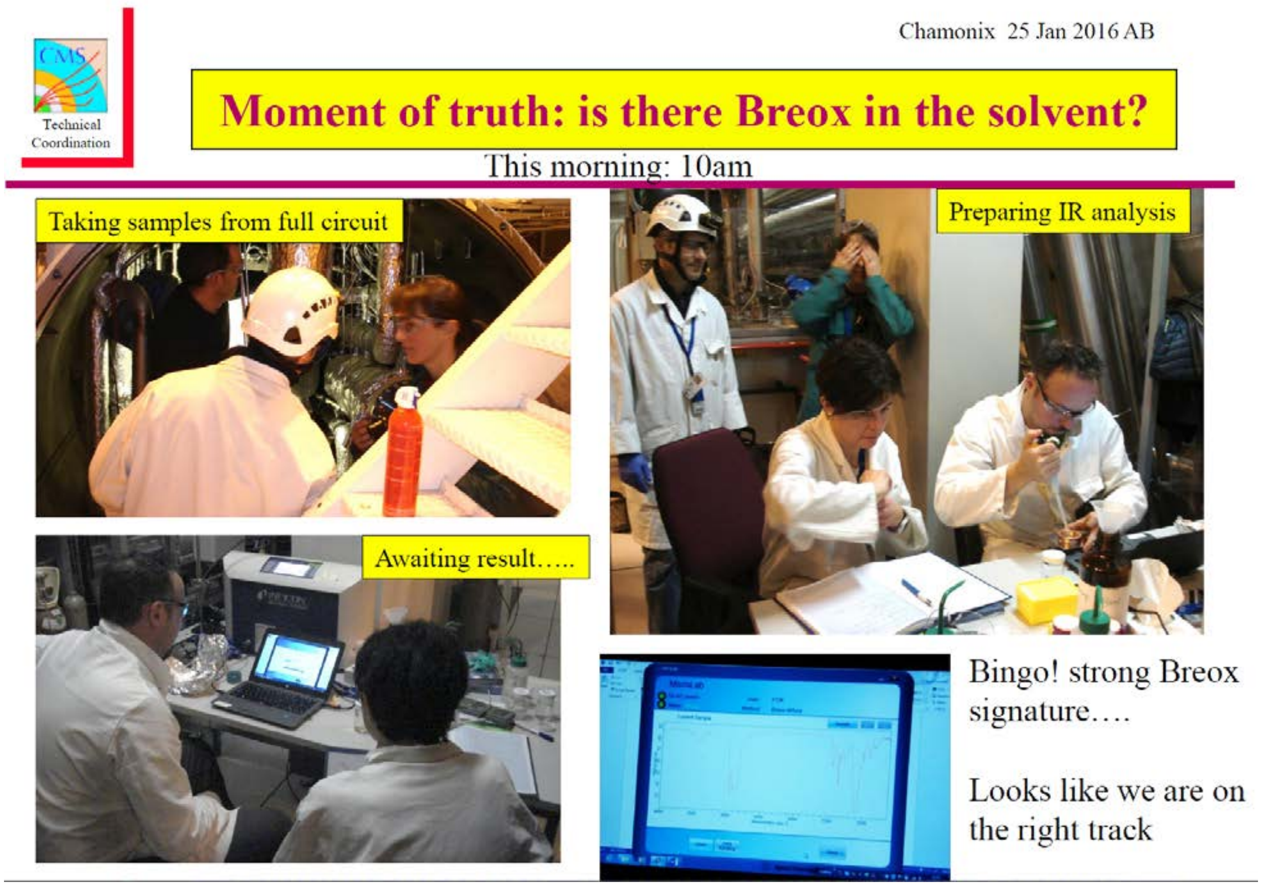


Nota: most outgassing items (Viton O-rings) exchanged during cold-box reconstruction

Results (1/3)

1st sample at HX1 > 1g (Breox® B35) / L (Vertrel® XF) !



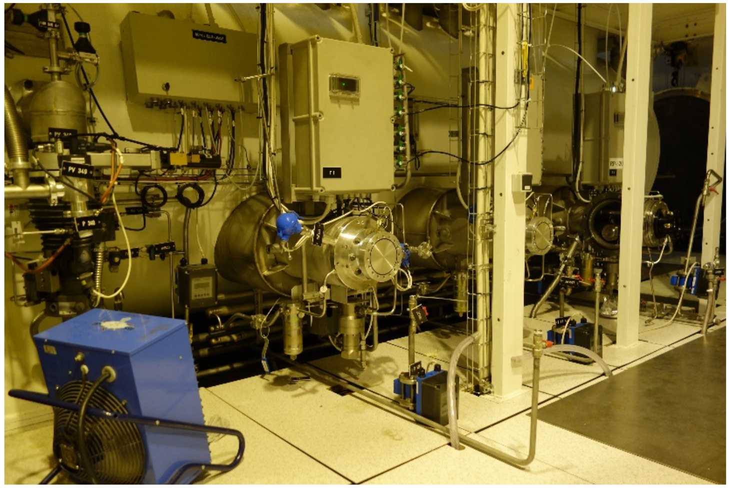
Courtesy: Austin Ball,
CMS Technical Coordinator

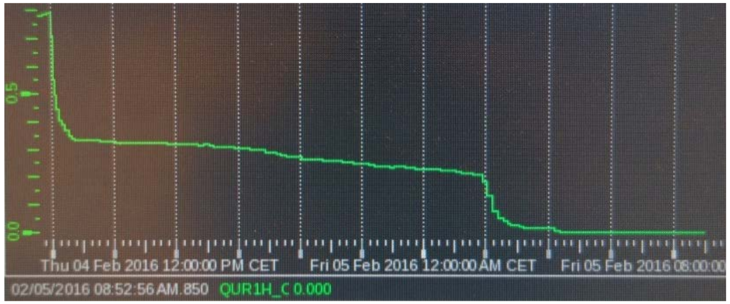
Results (2/3)

Circuit 1-HP: ~260 L of Vertrel® XF drained and ~300 L dried in < 1 day !

Cleaning machine & condenser

Cold box ambient conditions: 40°C !





End of drying signature

Results (3/3)

- In total 375 g of Breox® B35 extracted from the cold-box
- Low outgassing rate of Vertrel® XF (< 100 ppm(v) over few days)

Circuit	Duration	Circuit volume (L)	Effective cleaning time (h)	Breox amount collected	Drained volume (L)	Drying time (h)	Residual Vertrel® XF (ppm(v))*
Circuit 1: clean & dry	1 week	550 (HX1: 115)	3	353	260 (~50%)	< 24	< 10
Circuit 2: clean & dry	1 week	950 (HX1: 350)	2	10	760 (~80%)	< 24	< 10
Circuit 3&4: clean & dry	1 week	250 (PSD: 150)	1.5	13	190 (~75%)	< 24	~20

* after exchange of most outgassing items (Viton O-rings)

- A 2.5 month intervention

Preparation of cold-box and ancillary equipment	circuit 1: clean & dry	circuit 2: clean & dry	circuit 3&4: clean & dry	Overall outgasing and purge	Re-construction of cold-box
3 weeks	1 week	1 week	1 week	1 week	3 weeks

Lessons learnt

- Cleaning a cold-box is a heavy intervention.

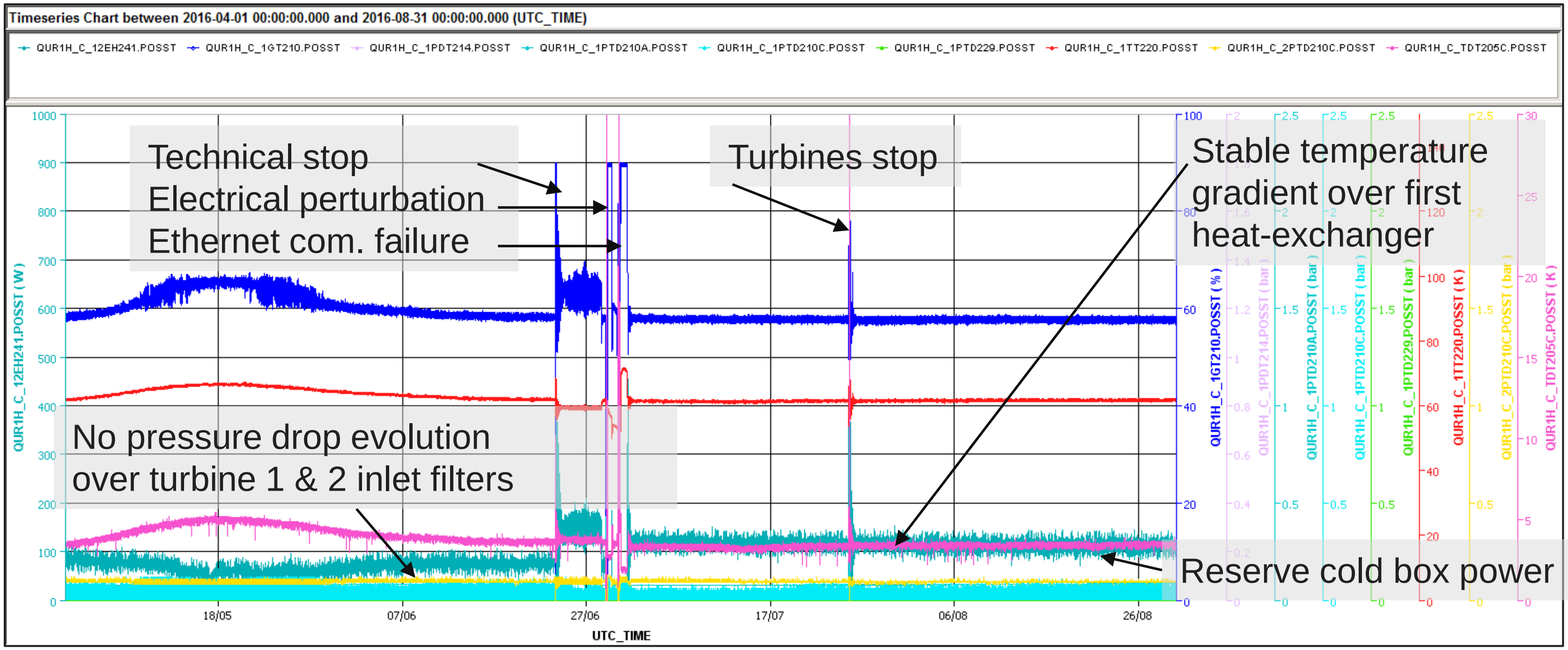
  … which nevertheless spared another 4000 clogged turbine filter exchanges …
  - Cleaning the HP branches would probably do it
  - Purge and drain tappings in biggest HX is worth it
  - Prevention is better than cure!
- Even very small amounts of oil in process flow will accumulate over years. (Present specification < 10 ppb(w))
  - We would like NO significant oil signal at the outlet of the oil adsorber
- The primary oil separator is an important element in the oil removal system.
  - The bad performance of a separator cannot be (fully) recovered by adding coalescers
  - We seriously consider to tighten the specified limit of 100 ppm(w)
- Be weary in case different / cheaper alternatives for spares or replacement material is proposed.

Nominal functioning through 2016

- The smoothest behaviour ever observed with this cold-box.

… more oil removed than accumulated during and after LS1*.

2016 cold box clogging and capacity evolution



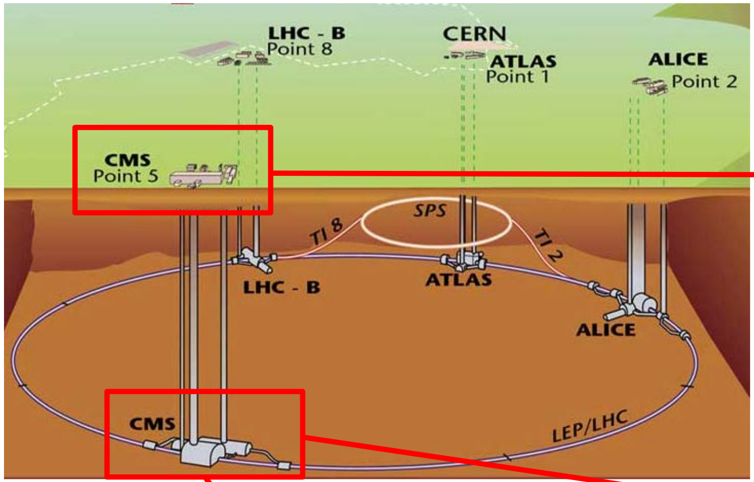
- The result of a team effort

(cryogenics – vacuum, surfaces & chemistry – safety – CMS – mechanics – cooling – transport …)

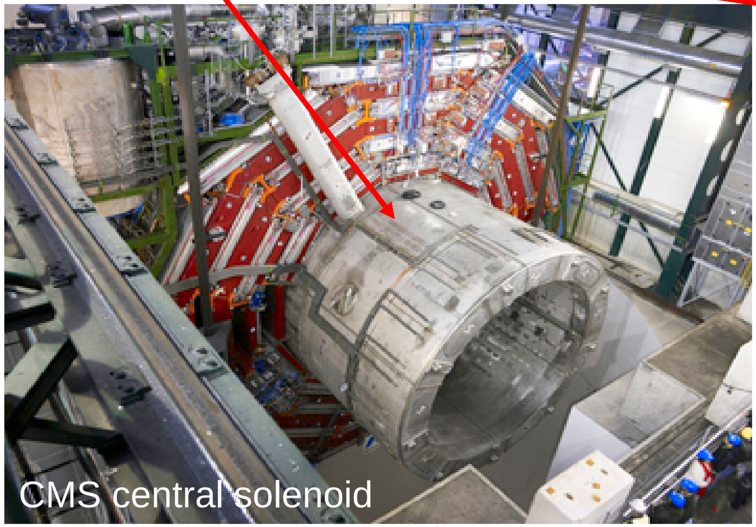
*LS1: Long shut-down 2013-14

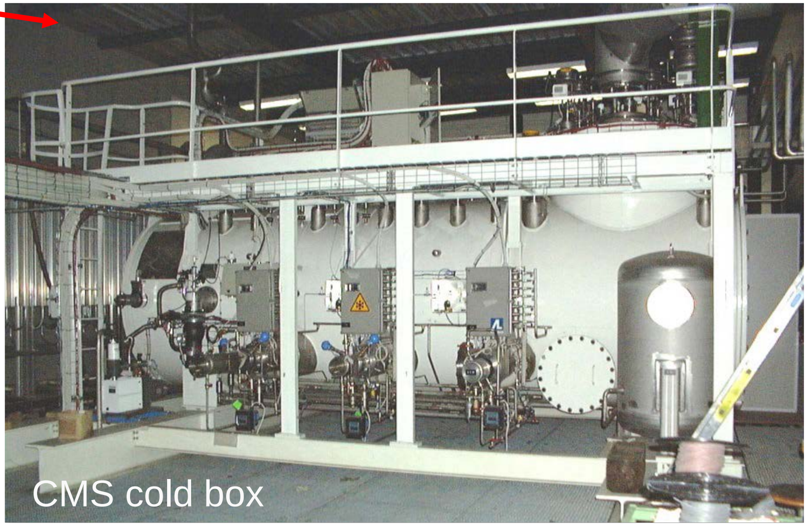
Back-up slides

CMS cryogenics

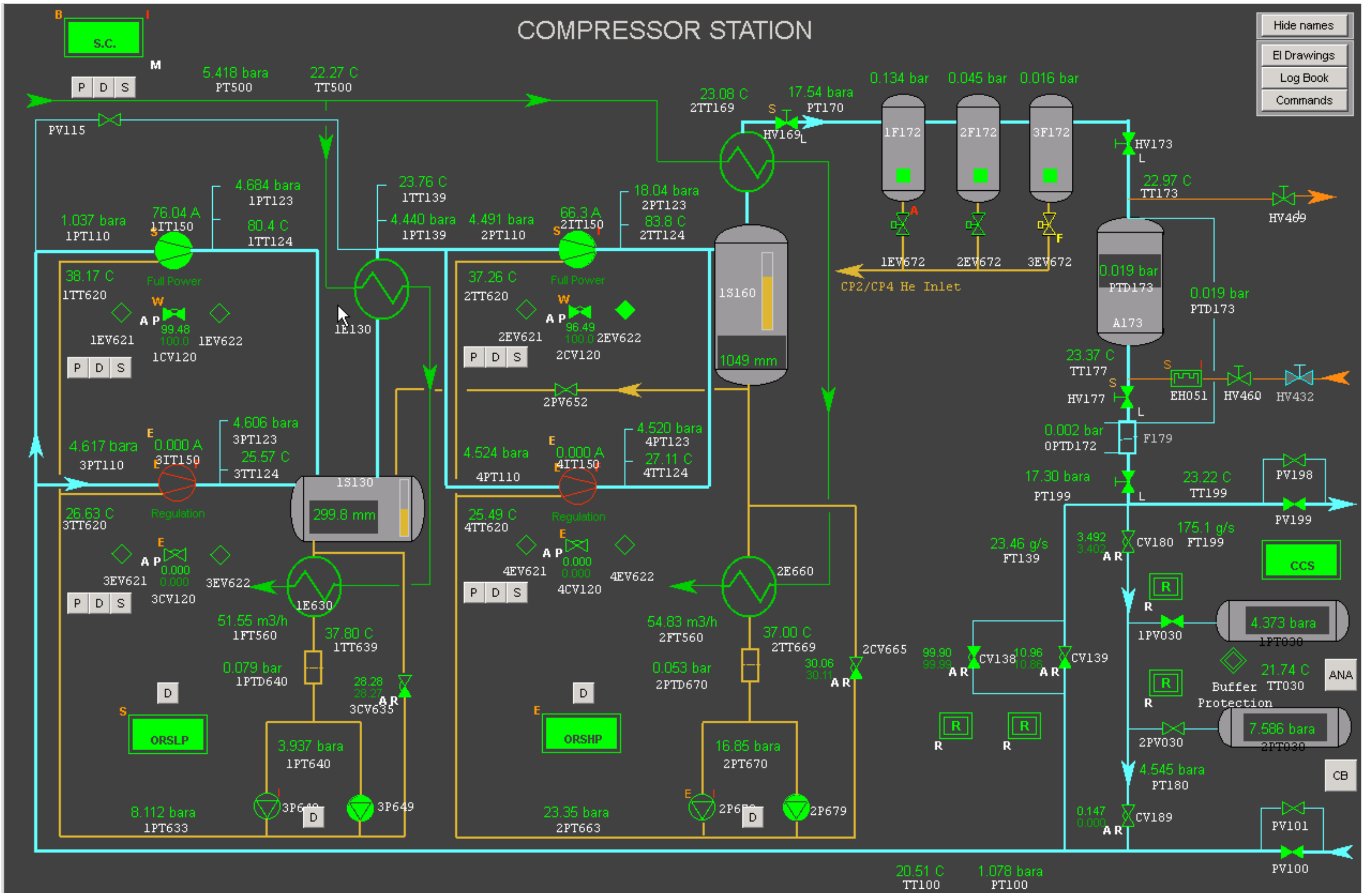



CMS compressor station (2012)


CMS central solenoid

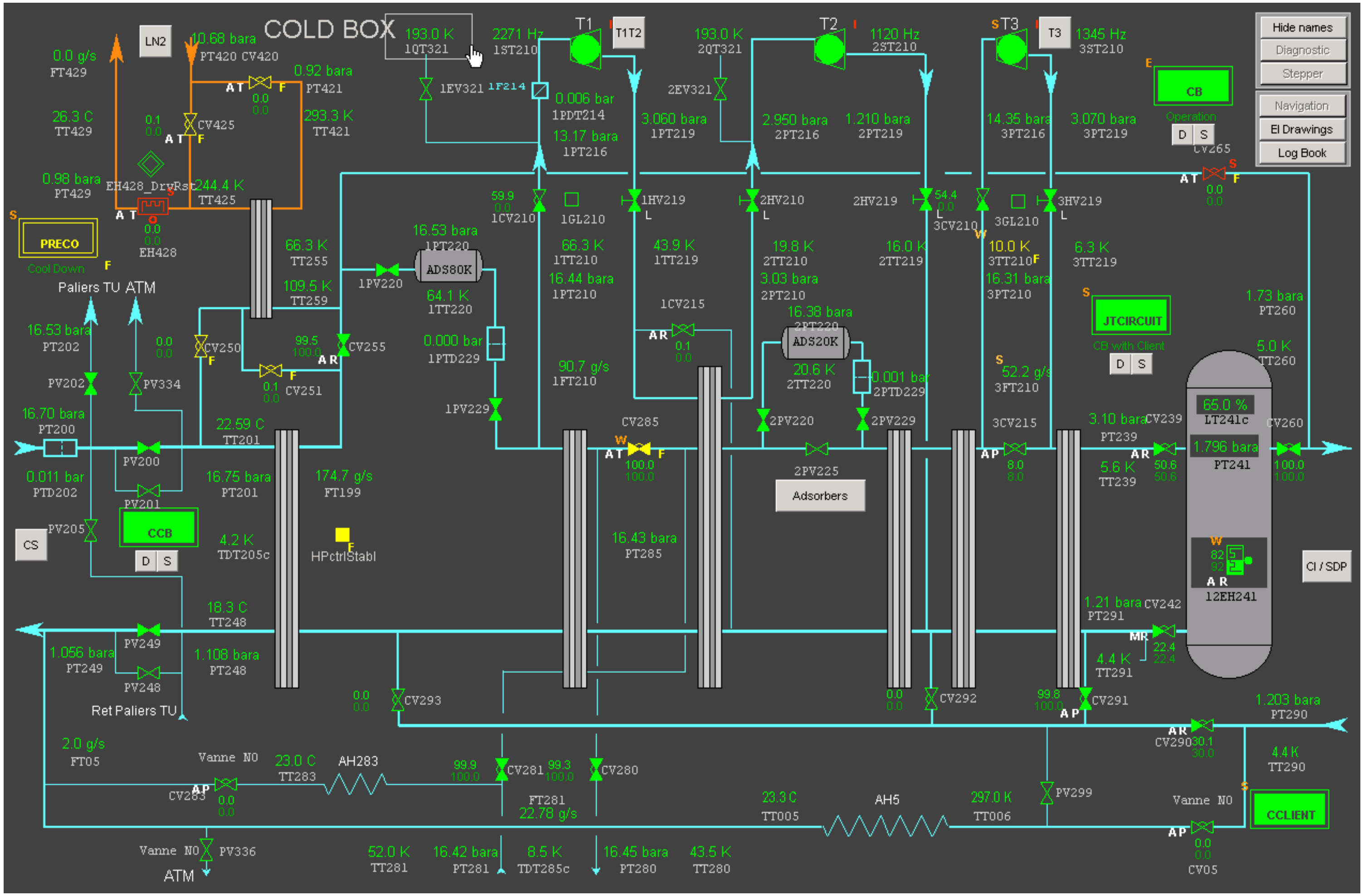

CMS cold box

Process parameters (1/2)



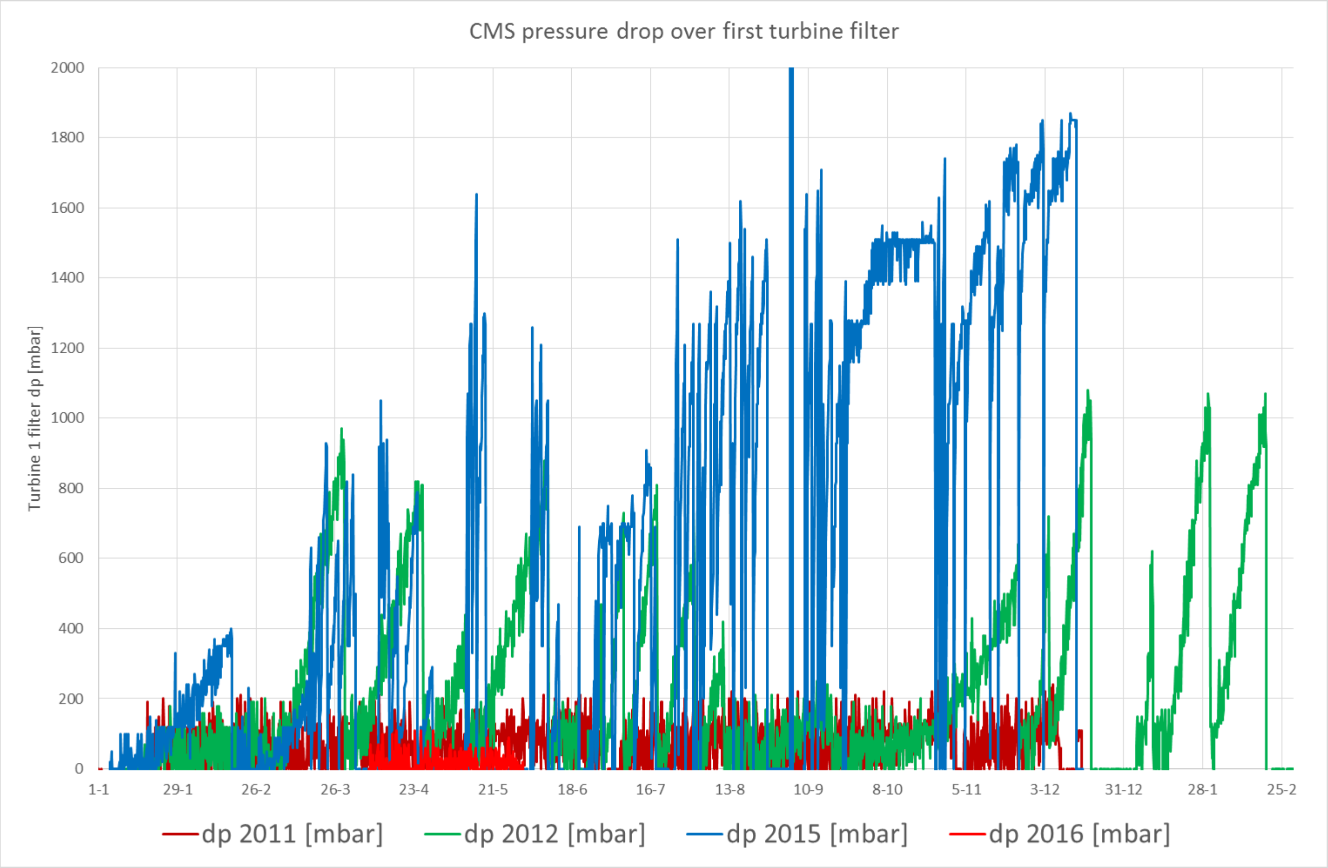
May 2016

Process parameters (2/2)



May 2016

CMS filter clogging 2011 to 2015



Courtesy: Udo Wagner, CERN Cryogenics group

DuPont™ Vertrel® XF

Safety Data Sheet

DUPONT

DuPont™ Vertrel® XF specialty fluid

Version 2.1

Revision Date 01/27/2015   Ref. 130000000559

This SDS adheres to the standards and regulatory requirements of the United States and may not meet the regulatory requirements in other countries.

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	DuPont™ Vertrel® XF specialty fluid
Tradename/Synonym	:	Vertrel® 4310 HFC-43-10mee HFC-4310
Product Use	:	Cleaning agent
Restrictions on use	:	For professional users only.
Manufacturer/Supplier	:	DuPont 1007 Market Street Wilmington, DE 19898 United States of America
Product Information	:	1-800-441-7515 (outside the U.S. 1-302-774-1000)
Medical Emergency	:	1-800-441-3637 (outside the U.S. 1-302-774-1139)
Transport Emergency	:	CHEMTREC: +1-800-424-9300 (outside the U.S. +1-703-527-3887)

SECTION 2. HAZARDS IDENTIFICATION

Not classified as a hazardous substance or mixture according to the Occupational Safety and Health Administration (OSHA) Hazard Communication Standard 2012.

Other hazards
Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing., Prolonged skin contact may defat the skin and produce dermatitis., Effects of breathing high concentrations of vapour may include:, May cause cardiac arrhythmia.
Misuse or intentional inhalation abuse may lead to death without warning.

1 / 10

Technical Information

Introduction

DuPont™ Vertrel® XF is a proprietary hydrofluorocarbon fluid with zero ozone-depletion and a low global warming potential ideally suited for use in vapor degreasing equipment for cleaning, rinsing, and drying. It can replace current hydrochlorofluorocarbon (HCFC) and perfluorocarbon (PFC) fluids in most applications.

DuPont™ Vertrel® XF is HFC 43-10mee or 2,3-dihydrodecafluoropentane; empirical formula $C_5H_2F_{10}$. It is a clear, colorless liquid with the properties shown in **Tables 1–2**.

Unique physical properties include a high density, low viscosity, and low surface tension. This combined with nonflammability, chemical and thermal stability, low toxicity, and ease of recovery by distillation make DuPont™ Vertrel® XF ideal for a broad range of applications. Solvency is selective, but can be enhanced by use of appropriate azeotropes and blends with alcohols, hydrocarbons, esters, etc. (see **Table 3**).

Typical Applications

- Cleaning and rinsing agent
- Drying fluid
- Particulate remover
- Fluorocarbon lubricant carrier
- Solvent and dispersion media
- Heat transfer media
- Dielectric fluid
- Replacement for many HCFC, PFC, and CFC-113 applications

DuPont™ Vertrel® XF is ideally suited for cleaning fine particulate matter (submicron range) from metal and nonmetal parts. Removal of particle contamination requires a solvent that can minimize the thickness of the laminar boundary layer where particles are bonded to the substrate. If the boundary layer thickness is less than the particle diameter, momentum from the flowing solvent can efficiently dislodge the particles and carry them away. DuPont™ Vertrel® XF, with its lower viscosity and higher density, results in a thinner boundary layer, which enhances cleaning. Common aqueous cleaning fluids, mixtures of water and detergent, have higher viscosities and lower densities compared to DuPont™ Vertrel® XF, making these fluids less efficient.

The electronic attraction between particle and surface can be overcome further by increasing the polarity of the fluid through the addition of small amounts of alcohols. DuPont offers a series of proprietary azeotrope and blend compositions which exploit this property (see **Table 3**).

Table 1
Physical Properties

Property[a]	DuPont™ Vertrel® XF
Molecular Weight	252
Boiling Point, °C (°F)	55 (130)
Surface Tension, N/m (dyn/cm)	0.0141 (14.1)
Liquid Density, kg/liter (lb/gal)	1.58 (13.2)
Freezing Point, °C (°F)	−80 (−112)
Solubility in Water, ppm	140
Solubility of Water, ppm	490
Critical Temperature, °C (°F)	181 (357)
Critical Pressure, kPa (atm) (psia)	2288 (22.6) (331.9)
Critical Volume, liter/mol (cc/mol)	0.433 (433)
Heat of Vaporization (at boiling point), cal/g (kJ/kg)	31.0 (129.7)
Specific Heat at 20°C, kJ/kg·°C	1.13
Vapor Pressure, kPa (atm) (psia)	30.1 (0.297) (4.37)
Viscosity, cPs	0.67

[a] At 25°C (77°F) except where indicated.
[b] Pensky-Martens Closed Cup Tester (ASTM D 93)
[c] Tag Open Cup Tester (ASTM D 1310)

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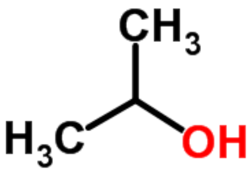
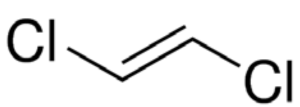
The miracles of science™

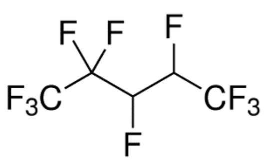
Main properties:



Cleaning agent

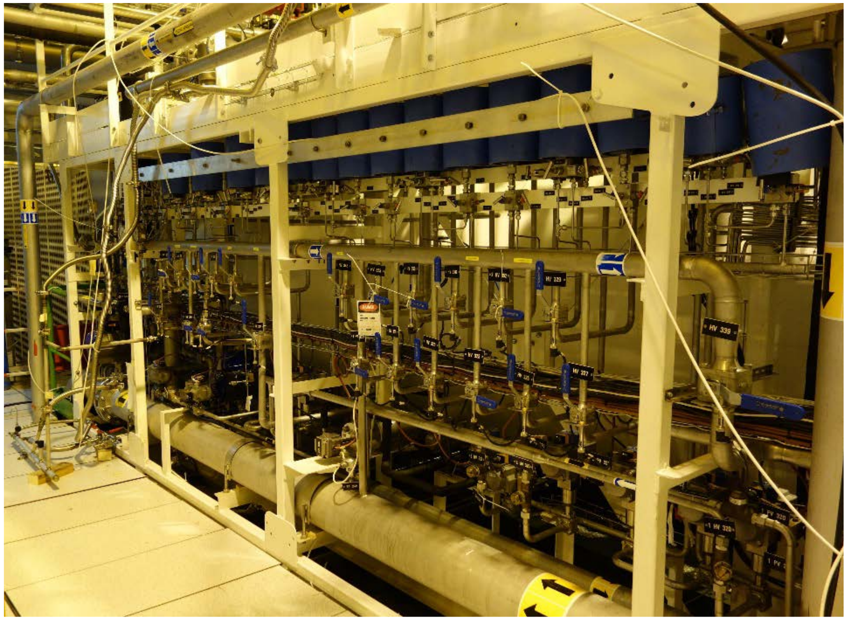
Solvent	Density	Vaporisation heat	Vapour pressure	Boiling point	Freezing point	Flash point	LFL	UFL	Cleaning	cp
	kg/m³	kJ/kg @ 25°C	mmHg @ 25°C	°C	°C	°C	%	%		kJ/kgK
Isopropanol	786	732	40	83	-89	11.7	2	12	Y	2.622
Acetone	791	518	230	56	-95	-20	3	13	Y	2.299
Petroleum ether	650	-	232	52	-73	0	1	6	Y	1.760
Modified alcohol	880	280	0.8	172	-75	63	1	8	?Y	2.000
Perfluorohexane	1669	96	202	56	-90	No	No	No	N	0.250
Methylene chloride	1330	330	430	40	-97	No	13	23	?Y	1.200
Vertrel Sion	1279	226	331	48	-50	No	7	14	Y	1.069
Novec HFE 71 IPA	1420	165	207	55	-135	No	4	17	Y	0.800
VERTREL XP	1530	~165	253	52	-80	No	No	No	*Y*	~0.800
VERTREL XF	1580	129.7	226	55	-84	No	No	No	*Y*	1.130
SOLREM 43i	1430	-	-	50	-30	No	-	-	*Y*	-
Water	1000	2441	24	100	0	No	No	No	?Y	4.186

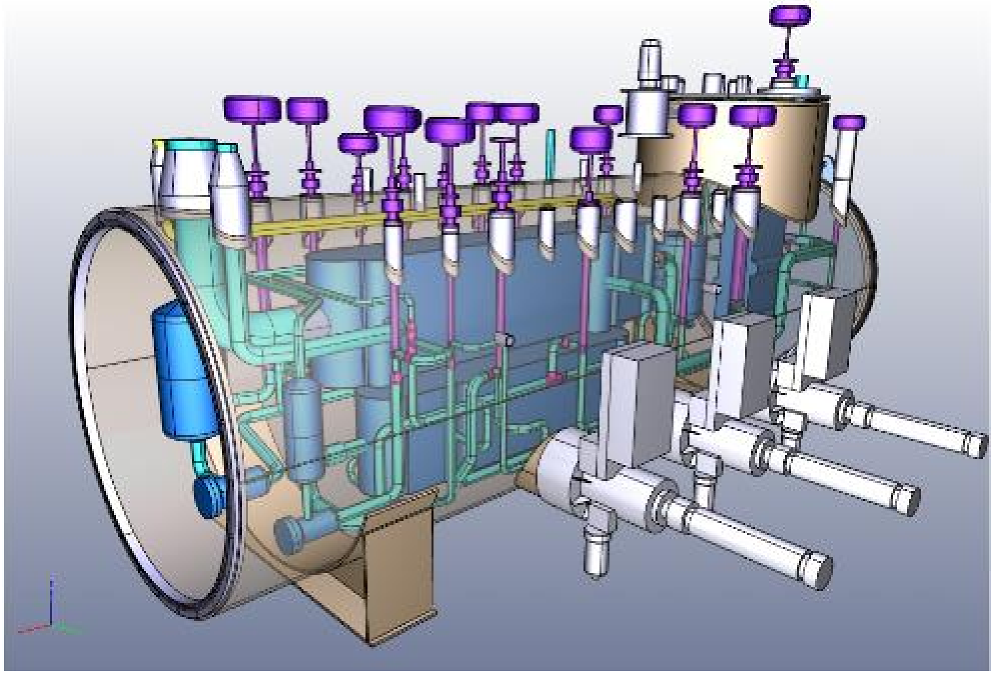




Courtesy: Leonel Ferreira,
CERN Vacuum, surfaces & Coatings group

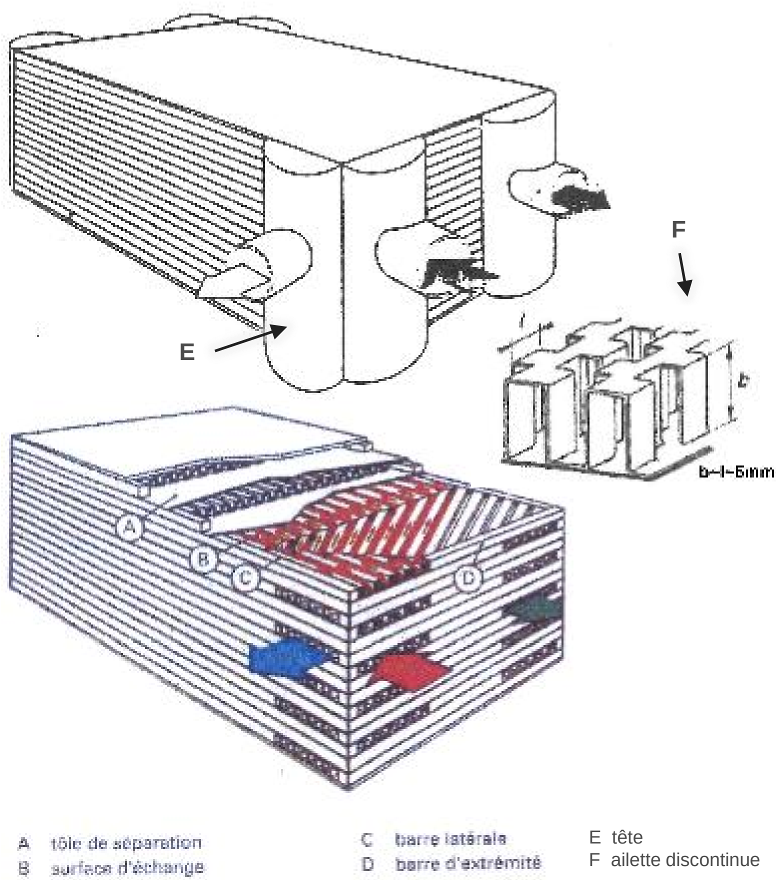
Cold box layout





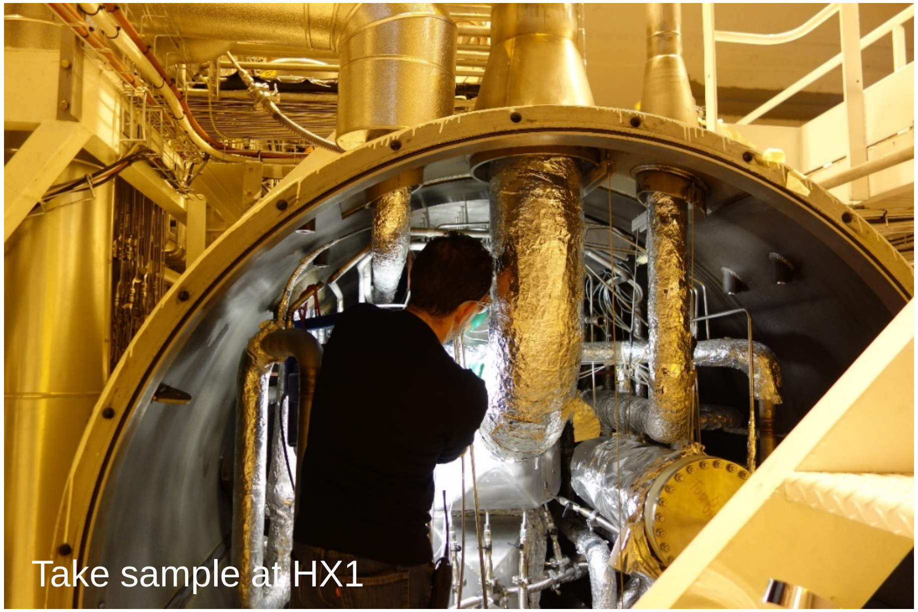
Courtesy: Nebojsa Smiljkovic PH/UMC

Heat exchangers (HX)



Echangeur à plaques brasées (source: doc. Nordon)

- Restriction = candidate oil trap
- Horizontal aluminum brazed plates
- 6 HX - 12 circuits
- HX circuits volume ranges from 10L to 350L
- Ensure HX filling with solvent
  - Filling of HX validated on test stand
  - Purge tappings made in HX1&2 as control points


Take sample at HX1


HX1 full


Take sample on return line


Cleaning quality «on-line» analysis

CERN