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SFR: Coolant quality control & Purification

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WHY IS IT NECESSARY TO CONTROL QUALITY AND TO PURIFY THE COOLANT IN LIQUID METALS

- Primary coolant of XFR:
 - [O] is a key parameter of corrosion → contamination→dosimetry → necessity to decontaminate (handling, repair, ISI,..): necessity to master [O]
 - [O] well mastered can help to maintain oxide layer stable (protection against hard corrosion in heavy liquid metals HLM, enhancement of tribology)
 - [O] can induce precipitation of coolant oxide (issue for HLM: PbO due to very low dissolution rate, no problem for Na), and modify composition of binary alloys, Pb-Bi...
- For Intermediate circuits of SFR (Na) :
 - [H] has to be maintained as low as achievable in order to detect as soon as possible a water ingresss in Na (Na-H₂O reaction generates H₂).
 - Moreover, Na purification allows to minimize tritium release. (Nota: Tritium release is a common issue for all nuclear systems, including FRs
- For all the circuits :
 - Control risks of plugging in narrow gaps, tubing, openings, seizing of the rotating parts, reduction of thermal transfer coefficient in IHX (Intermediate Heat Exchanger)...

→ to limit the plugging risk, necessity to maintain [O] < [O]* and [H] < [H]* at the coldest point of the circuits, for all operating conditions ; value for SFRs: Tsat < Tcp - $30^{\circ}C_{\perp PAGE 2}$

Content



- Why is it necessary to purify Na?
- Basic crystallization phenomena involved in sodium
- Design of Generation 1 cold traps
- Purification campaigns
- Design of Generation 2 cold traps for SPX and EFR
- Mass balance of cold traps

Why is it necessary to purify Na?

CADARACHE

Two main impurities : O and H, even if other impurities (radionuclides) can be considered with other specific purification systems.

Primary Na :

•

- [O] is a key parameter of corrosion → contamination
 - Consequences on dosimetry → necessity to decontaminate (handling, repair, ISI,..)
- Intermediate Na :
 - [H] has to be maintained as low as achievable in order to detect as soon as possible a water ingresss in Na.
 - Moreover, Na purification allows to minimize tritium release.
- For all the circuits :
 - Control the risks of plugging, seizing of the rotating parts, reduction of thermal transfer coefficient...



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O & H solubilities in liquid Na



Continuous or discontinuous ingress



Continuous

- Diffusion of hydrogen through SGU walls
- Impurities in cover gas (Ar)
- Discontinuous
 - Oxide dissolution in Na
 - Air ingress after repair (air + moisture)
 - Water ingress in SGU
 - Oil ingress (pumps)
 - Metallic filling due to maintenance, etc...

Main processes for Na purification



- Specific methods to obtain nuclear grade sodium
- Initial cleaning of loop, components and vessels
- Filtering
- Cold trapping
- Hot trapping
- And of course limiting ingress of pollution by appropriate operating rules

Crystallization methods



Liquid evaporation (like salt)

 Reduction of the solubility by adding some additive impurity

Cooling the liquid below saturation temperature

- → This last option is the most attractive :
 - Convenient solubility laws for O and H (reach almost 0 near the Na melting point)
 - Easy implementation : cooling and solid retention























Free enthalpy of nucleation



Nucleation phenomena



- If sodium is supersaturated, presence of agglomerates
- These agglomerates can turn into nuclei if free enthalpy of the system reaches a limit value (primary nucleation)
- Nevertheless some existing crystals can produce some small particles by attrition (secondary nucleation)
- The primary nucleation can be homogeneous (if it occurs in the sodium bulk) or heterogeneous if it occurs on metallic surfaces. Wetting characteristics have a large influence on heterogeneous nucleation



Growth phenomena



- Theory of diffusion through a boundary layer
 - Growth can be considered as two consecutive phenomena :
 - A diffusion step through a boundary layer
 - Dm/dt = kD.A.(C-Ci)
 - An integration step at the crystal solution interface
 - $Dm/dt = kR.A.(Ci-C^*)^{nR}$
 - Due to the fact that it is difficult to know Ci, we use an overall equation :
 - Dm/dt = kG.A.(C-C*)^{nG}
 - Two situations:
 - If growth is limited by diffusion step, then kG = kD and nG = 1
 - If growth is limited by integration step, then kG = kR and nG = 2
 - These values of nG have been established by Burton Cabrera and Franck

Growth phenomena



Model of the kinematic wave (Franck-Vermilyea) : explain the formation of steps





Na₂O in liquid Na





Two growth phenomena for Na₂O :

- low supersaturation : regular growth
- high supersaturation : dendritic growth



REGULAR GROWTH (X500)



DENDRITIC GROWTH (X20)

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Growth phenomena



→ Kinetics of the overall phenomena = kinetics of the slowest step



Kinetics of NaH and Na₂O

- Nucleation
 - $Na_2O : E_N = -60 \text{ kJ/mol}$ $n_N = 5$
 - NaH : $E_N = -450 \text{ kJ/mol} n_N = 10$
- Growth
 - $Na_2O : E_G = -45 \text{ kJ/mol} \quad nG = 1$
 - NaH : $E_{G} = -43.6 \text{ kJ/mol} \text{ nG} = 2$
- Thus, different cristallization mechanism
 - Possibility to obtain crystallization of NaH on cold walls (large influence of supersaturation and nucleation)
 - Necessity to provide steel packing for Na₂O

C. Latgé, "A study of Sodium oxide crystallization mechanisms and kinetics in cold traps", 3rd LIMET Conference 1984 April 9th 13th Oxford (UK)

C Saint-Martin, C. Latgé, C. Laguérie, P. Michaille, « Etude expérimentale de la cristallisation de l'hydrure de sodium dans un piège froid garni » Journal of Nuclear materials 151 (1988) 112-119 North Holland Editor

C. Latgé, « Sodium quality control; French developments from Rapsodie to EFR". Conférence FR09 Kyoto Décembre 2009

Cold trap: kinetics

Crystallization kinetics, given for one impurity O or H: in [kg Na_2O/s] or [kg NaH/s]

$$r_{jX}(T,t) = k_{oX} \exp(-\frac{E_X}{RT}) A_{jX}(t) \left[\frac{(C-C^*)}{1.10^{-6} \rho_{Na}}\right]^{n_X} = Ko_X A_{jX}(t) [\Delta C]^{n_X}$$

In this equation:

Index X refers to Nucleation (N) or growth (G)

Index j refers to the location on wire mesh packing (p) or cold walls (w).

k0 is the rate constant (kg/(s.ppmnx.m²)),

E is the activation energy (J/mol),

R is the Boltzmann constant (J/(mol.K)).

A is the crystallization surface of reference (m²)

(wire or walls for nucleation, nuclei and crystals for growth).

nX is the order of the crystallization process.

C*(kg/m³) is the saturation concentration (from solubility law.)

ρNa is the sodium density in (kg/m³)

 $(C-C^*)$ is the supersaturation at temperature T(K).

Phenomena	Nucleation (N)		Growth (G)			
Impurity	Na ₂ O	NaH	Na ₂ O	NaH		
E (kg/mol)	-60	-450	-45	-43.6		
n	5	10	1	2		

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Nucleation kinetics:

 $R_N (NaH) >> R_N (Na_2O)$

Large impact of T for NaH nucleation

Phenomena	Nuclea	Nucleation (N)			Growth (G)			
Impurity	Na ₂ O	NaH		Na ₂ O	NaH			
E (kg/mol)	-60	-450		-45	-43.6			
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 $(C-C^*)$ is the supersaturation at temperature T(K).

Growth kinetics:

Limiting step for NaH: 2 Limiting step for Na₂O: 1

Phenomena	Nucleation (N)			Growth (G)				
Impurity	Na₂O	NaH		Na₂O	NaH			
E (kg/mol)	-60	-450		-45	-43.6			
n	5	10		1		2		

Cold Trap: principle of operation

•Na is first cooled thanks to an Integrated Heat Exchanger (« Exchanger Economizer »): T reaches a value close to Tsat

•Then Na goes through a cooler: temperature T reaches a value Tcp lower than Tsat.

•Crystals are produced (nucleation + growth) on cold walls or meshed packing. Nucleation occurs when T< Tpt.

• The Na flow is then heated, thanks to Integrated Heat Exchanger.

- ➔ Tcp: Tcold point
- ➔ Tsat: Tsaturation
- → Tpt: Tplugging temperature
 → Ci: Cinlet Co: Coutlet



REGULAR GROWTH (X500)



DENDRITIC GROWTH (X20)



- Instantaneous efficiency: E = (Ci-Co)/(Ci-C*) 0<E<1
- Ci: concentration at the cold trap inlet
- Co: concentration at the cold trap outlet
- C*: concentration at equilibrium (solubility)
- Purification rate : Vp = E.(Ci-C*).DNa
- **Capacity : Cap =** $\Sigma \tau r$ **.Ed (in unit of volume)**
- where $\tau r = filling rate$
- and Ed = deposited element concerned (area, volume)
- Compactness: Comp = V1/V2
- V1 = Maximum volume of the impurities retained in the trap
- V2 = Internal volume of the trap where the deposit zones are located



- Vp : highest value, in case of large pollutions. There is generally no specifications on efficiency, nevertheless a high value of efficiency allows generally to obtain a high purification rate
- Comp : the highest value is the best
- Cap : depends on the policy chosen for the cold trap
 - If the source of impurities is low, possibility to size the trap for the whole service life duration
 - If the cource of impurities is high, possibility to size a large trap for a long service life or a smaller cold trap which can be removed or regenerated and hence reused

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SOLUBILITIES OF O AND H IN SODIUM



Noden solubility law $\log_{10}[O(ppm)] = 6.250 - \frac{2444.5}{T(V)}$

T(K)

O and H solubilities are negligible close to 97.8° C

Consequences: Na can be purified by Na cooling, leading to crystallization of O and H as Na₂O and NaH in a "cold trap"

Temperature, °C

Quality of Na has been always well mastered with cold traps, in normal or transient situations (start-up purification, large air pollution in SPX) C. LATGE, *"Sodium quality control, In International Conference on Fast reactors",* Kyoto, Japan, (December 2009).



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PLUGGING-METER (STANDARD 180L/H)



PLUGGING-METER CURVE 2/3



= minimum Flow-rate value which initiates temperature increase

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PLUGGING-METER CURVE 3/3





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Purification procedure



Some design options



- Sodium distribution in the cold trap : ring type header
- Cooling fluid : air, oil, NaK oil in static NaK, ...
- Internal exchanger-economizer : to avoid plugging of the inlet pipe
- Cooler : one cooler or modular coolers (to adapt thermal gradient with source of impurities (i.e. : hydrogen)
- Support for impurities : knitted mesh, pall rings, cooled wall,..
- Location of support : location and surface per volume depends of the design requirements.

C. Latgé"Sodium purification: a key process to operate safely sodium systems". 10th PAMIR International Conference - Fundamental & Applied MHD, 2016, June 20th 24th, Cagliari (Italy)



Primary Integrated Purification system (2 cold traps)

Nominal Flow-rate: 10 m³.h⁻¹




Intermediate purification system of SPX

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Efficiency of a « one-zone » cold trap





For a cold trap designed with an upper packless cold zone:

- → Na₂O crystallization only on wire mesh packing
- ➔ NaH crystallization essentially on cold walls of the system, (if thermal flux is enough)

However, in case of a non-optimized packless cold zone, hydride might as well crystallize on wire mesh packing. Thus, co-crystallization occurs between sodium hydride and sodium oxide.

→ Hypothesis: kinetics parameters for NaH crystallization on cold walls identical to those on wire mesh packing.



PSICHOS CONCEPT



- Designed for SuperPhenix
- Main advantages
 - Efficiency = 1/ NaH and 1/ Na_2O
 - Flowrate minimized to limit heat loss in the circuit
 - Large capacity : for NaH and Na_2O : more than 0,420 m³
- History
 - Patented by CEA in 1987
 - Sized by STEIN-Industrie (GEC-Alsthom Company)
 - Sized for SPX, tested in experimental loop (mock-up)
 - Sized for CHEOPS project

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PSICHOS CONCEPT



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PIRAMIDE CONCEPT



- Designed for European Fast reactor Project (UK, GERMANY and France)
- Main advantages
 - Efficiency = 1/NaH and 1/Na₂O
 - Flowrate minimized to limit heat loss in the circuit (about 17 m³/h)
 - Large capacity : for NaH and Na_2O : more than 0.520 m³
- History
 - Patented by CEA in 1987
 - Sized by STEIN-Industrie (GEC-Alsthom Company)
 - Sized for EFR, SPX, tested in experimental loop (mock-up)
 - Sized for ASTRID



PIRAMIDE COLD TRAP



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Cold Traps (Russia)

F.A. Kozlov et al./Nuclear Energy and Technology 2 (2016) 5-13



Cold traps of BR-5 reactor - (a); cold trap cooled with Na-K eutectic - (b); cold trap of BN-350 reactor - (c); cold trap of BN-600 reactor - (d).





Prototype of a 180 liter gas-cooled cold trap: 1) settler; 2) final-cooling zone; 3) filter.

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Cold Traps (India) (a mix of PIRAMIDE and PSICHOS concepts)



In the eighties:

- **Definition of the main requirements**: pollution sources, purification rate, capacity, compactness)
- Design of the scale-one cold trap for the reactor, i.e. type of cold trap, cooling fluid, maximum allowable temperature, trapping zones to be implemented, ...
- Identification of similitude rules (heat transfer, residence time in each zone, mesh density, loading capacity...) and down-scaling of the mockup
- Manufacturing of the mockup and implementation of dedicated chemical instrumentation (O-meter, H-meter) on the facility used for qualification tests
- Loading of the ancillary cold trap, used as a polluting device, with Na₂O or NaH or both
- Operating the cold trap in various conditions in order to assess the allocated performances (efficiency, purification rate and capacity) and to confirm the operating rules.

Today:

- Use of the new code (ANAÏS) described here, validated using the previous experimental results, allows for a quite efficient design procedure, based on sequential calculations, avoiding long and very expensive experimental tests.
- C. Latge et al., « Sodium Purification Systems: requirements, tools and gualification strategies. », ICAPP, Charlotte, USA, 2014, vol. Paper 14069, p. 8.
- N. Khatcheressian, C. Latgé, X. Joulia, T. Gilardi, et X. Meyer, « Development of a transfer model for the design and the operation of sodium purification systems for fast breeder reactors », The Canadian Journal of Chemical Engineering, vol. 93, nº 2, p. 213-224, 2015. C. Latge, N. Khatcheressian, Th. Gilardi, X.Joulia, X.Meyer, H.Paillère Design and Modelling of Cold Traps for Sodium Purification systems in Fast ReactorICAPP '12 Chicago, USA, June 24-28, 2012

ANAÏS: Advanced model for NA Integrated purification Systems

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Transport Phenomena Coupling



Two types of crystallization mechanisms:

- → two different modeling approaches (on cold walls or on mesh).
- → For both approaches:

System : a porous media with a porosity φ which refers to the void fraction of the liquid phase. **Porosity is initially:**

- equal to 1 with deposit on walls (NaH),
- equal to the void fraction of the mesh packing if the deposit occurs on wire mesh (Na₂O).

Mixture of crystal and liquid sodium then treated as a diphasic media in which void fraction changes with time and space, as well as its physical parameters such as permeability, diffusion or thermal conductivity.

Fluid assumed to be incompressible and each infinitesimal volume assumed isothermal.

→The model takes into account hydrodynamics in porous media, heat transfer, mass transfer, and nucleation and growth kinetics.

- ➔ Both approaches are dealing with void fraction evolution on wire mesh packing for oxide and on cold walls for hydride deposit.
- → The numerical model is solved by finite element method in Comsol Multiphysics® software.

Two types of crystallization behavior:

→ Two different modeling approaches (on cold walls or on mesh).

Crystallization	Wire mesh packing	Cold walls (packless zone)
Impurities	- Na ₂ O - NaH	- NaH
Models	- Porous medium densification	 Porous medium densification Interface tracking
Surface to crystallize	- Wire mesh - Oxide and/or hydride crystals	- Hydride crystals

→ For both approaches:

System : a porous media with a porosity ϕ referring to the void fraction of the liquid phase.

Porosity is initially:

- equal to 1 with deposit on walls (NaH),
- equal to the void fraction of the mesh packing if the deposit occurs on wire mesh (Na₂O).

Mixture of crystal and liquid sodium in which void fraction changes with time and space, as well as its physical parameters such as permeability, diffusion or thermal conductivity...

Fluid assumed to be incompressible and each infinitesimal volume assumed isothermal.

→ The model takes into account hydrodynamics in porous media, heat transfer, mass transfer, and nucleation and growth kinetics

→ Numerical model solved by finite element method in Comsol Multiphysics® software.

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Na₂O crystallization modeling



Front propagation tracking for NaH crystallization

→ Density of NaH deposit experimentally estimated to be around 0.5
 → Crystal growth on cold walls can be seen as a phenomenon involving a moving interface, with propagation of a deposit front.
 → Tracking Moving Interface is a key element of the model



NaH deposits on cold wall (EPINAR experiment)

C. Latgé, G. Hulme, DG Jones, F. Perret «Experimental studies of packless cold traps for validation of the VICSEN code for prediction of cold trap behaviour»

4th LIMET Conference 1988 October 17th – 21th Avignon (France)

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Interface tracking model for NaH crystallization on cold walls



Velocity front propagation

Before any start of deposit progress, the first crystals layers formed on cold walls must get denser. It is assumed that it corresponds to the diffuse interface length since nucleation is initiating the motion of the interface. Thus, hydride deposit will not progress until a first critical porosity profile $(\partial \phi / \partial r)_{crit}$ has not been reached. The front velocity is then defined such that porosity profile over the diffuse interface remains the same. It comes:

$$\forall (r,z) / 0 < \psi(r,z) < 1: \frac{D\phi}{Dt} = \frac{\partial\phi}{\partial t} + \vec{v}_{FS} \left(\frac{\partial\phi}{\partial r}\right)_{crit} = 0$$
(8)

For a given z position in cold trap, radial growth crystallization is assumed. Front velocity field is thus expressed as:



(9)

The equation (9) can be simplified with the approximation of a linear porosity profile over the diffuse interface. $(\partial \phi / \partial r)_{crit}$ is then assigned as a constant.

NaH crystallization modeling



Purification system design methodology

- Identification of the impurities to be removed
 - Assessment of their sources and their production rate (continuous & discontinuous)
 - Assessment of their potential sinks i.e. corrosion of structural material, deposition in the coldest points of the circuits....
 - → induces specifications on the removal rate to be obtained in order to prevent any deleterious effects,
 - Evaluation of the amount of impurities to be removed from the circuits, vessels, during the cold trap(s) operation over a given period depending on the strategy selected for the purification systems
 - → induces specifications on the loading capacity
- Selection of several trapping zones
- Strategy similar to any liquid metal coolant
- Simulation with ANAÏS* code for Na: filling rate, distribution of impurities, detection of plugging hazards

ANAÏS: Advanced model for NA Integrated purification Systems



Four main criteria generally considered for cold trap design:

Efficiency: $\mathcal{E} = \frac{C_i - C_o}{C_i - C_{\mathcal{T}_{cp}} \quad sat}$

→ recommended to get an efficiency of 1 for each trapping zone. (efficiency depends on the crystallization) kinetics, residence time, local conditions....)

Purification rate: $Rp = \varepsilon \cdot D_{na} \cdot (Ci - C_{Tcp-sat})$

→ duration of a purification campaign determined by the initial impurities content in Na and Rp

Capacity: total amount of impurities (mass or volume) accumulated in the cold trap over the entire time of operation, defined by the designer.

Compactness: ratio between the volume of impurities trapped in the cold trap and the overall internal volume of the cold trap.

How to control the filling rate of a cold trap :



- Mass balance
- Monitoring of the pressure drop of the pump
- Neutron transmission measurement,
- Visual control by endoscopy,

C. Latgé, F. Perret, J. Guidez, Y. Cadoret « Monitoring the filling of secondary cold traps in Phenix and Superphenix" International Conference on FBR systems experiences gained and pathe to economical power generation. 1987 September 13th to 17th Richland (USA)



Neutron transmission measurement

Basic principle :

Slowing down of neutrons emitted by a source ,by elastic diffusion with light nuclei.
 Counting of the thermalized neutrons.
 Comparison of the measure with a baseline.
 reference value (trap first placed into operation 4- Calculation of the attenuation ratio.

Implementation :

Source : Californium 252 Counter : ³He ionization chamber – The neutron counter is mounted on the outer

wall ,on the same radius as the measurement point.

 The source is extended from a storage cask to the measurement point.

 Determination of the attenuation ratio profile along several generatrix.

NOTA : This method can be used ,without shutting down the cold trap.

Neutron transmission measurement











Storage cask

③ Neutron counter (³He ionization chamber)



SPX1 : Secondary cold trap





HOT TRAPPING

- Several hot traps were developed in the past
 - ZR_{0.87}-Ti_{0.13} alloy for O : allows to reach very low O concentration (below solubility), but low capacity and necessity to implement a filter downstream
 - Yttrium foils for hydrogen
 - Same comments as for Zr-Ti
 - Hot traps present interest in two cases :
 - Stable trapping in case of temperature rise up (avoid released pollution)
 - Easy to implement in small experimental circuits or vessels or irradiation loops (can be static
 - $R = 41.26 \ 10^{-3} \cdot exp(-40.3x10^3/RT) \cdot C \ kg \ O/(h.m^2)$
 - Kinetics are established in the range of 1 200 ppm
 - No significant effect of the flowrate on the oxidation rate
 - Thus the limiting step is oxygen diffusion in the alloy
 - Capacity : 0.234 g(O)/g alloy

C. Latgé, S. Sellier

"Oxidation of Zr-Ti alloys in liquid sodium : validation of a hot trap, determination of the kinetics" Material behaviour and physical chemistry in Liquid Metal Systems 2, Edited by H.U. BORGSTEDT, Plenum Press 1993



Operational feedback from Germany: Ex : ⁵⁴Mn, ⁶⁵Zn 50h in Na at 360°C 1mmNi : 83kBq/cm²

Also in France : loop TPL in SILOE, And in Belgium: BR2



Trap with Ni foil for ⁵⁴ Mn (tested at EBR2) (set up in a fuel assembly above pins, not necessary to equip all assemblies!). Less efficient for ⁶⁰Co



- → Reticulated vitreous carbon (RVC) traps : adsorption on RVC
 Efficient process ; operation at T around 200°C
 (possibility to reduce contamination by a factor 10 for each transfer through the trap)
 Applied to EBR2, BOR60, RAPSODIE, ...
- <u>Nota</u>: necessity to take into account delay before Na treatment and decay ¹³⁷Cs/ ²²Na (Feedback from RAPSODIE)
- 3 cartridges adsorbed about 0.49 TBq ¹³⁷Cs
- Will be used for PHENIX primary Na within the next months.







EBR2 : cold trap RVC

FFTF : cold trap RVC

RVC: Reticulated Vitreous Carbon trap







Oxygène-mètre Harwell







Currently, new developments in CEA

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O-meter: calibration

Calibration curve in Na Oxygène-mètre Harwell



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H-METER (NI MEMBRANE + MASS SPECTROMETER)



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ELECTRO-CHEMICAL H-METER (COURTESY IGCAR INDIA)



ELECTROCHEMICAL H-METER

COURTESY IGCAR

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HYDROGEN-METERS (CABR2-CAHBR) (IGCAR) TESTED IN PHENIX




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Thank you for your kind attention !

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