Distillation of Hybrid Deuterium
for solid HD targets
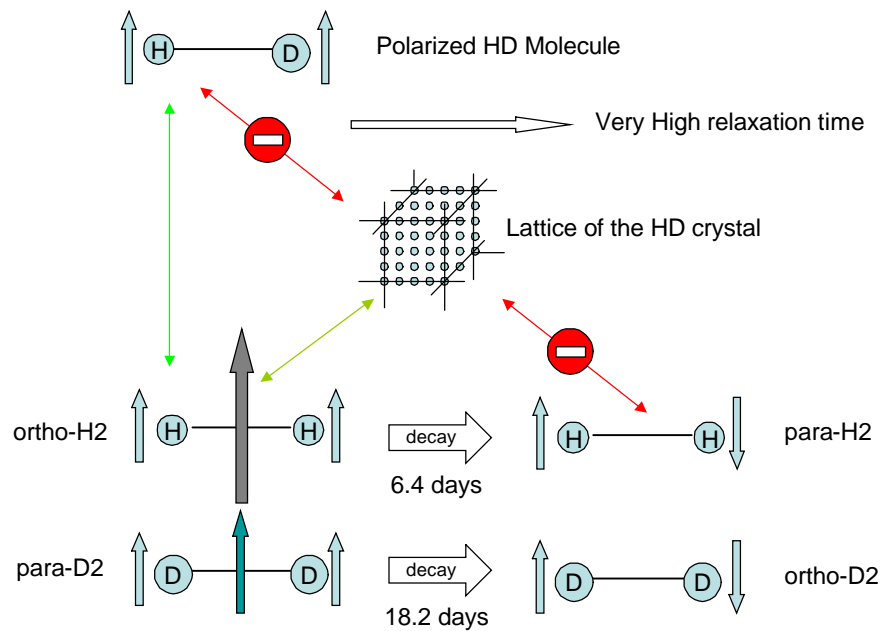
S. Bouchigny, J.P. Didelez, G. Roullie
IPN Orsay – I3HP

o Introduction to solid HD target Static Polarization

o Distillation apparatus at Orsay

o First test run and results

o Summary and outlooks

Polarized Targets for Europe, Miltenberg June 2005

Static Polarization of HD targets

(Honig, 1967)

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Static Polarization of HD targets

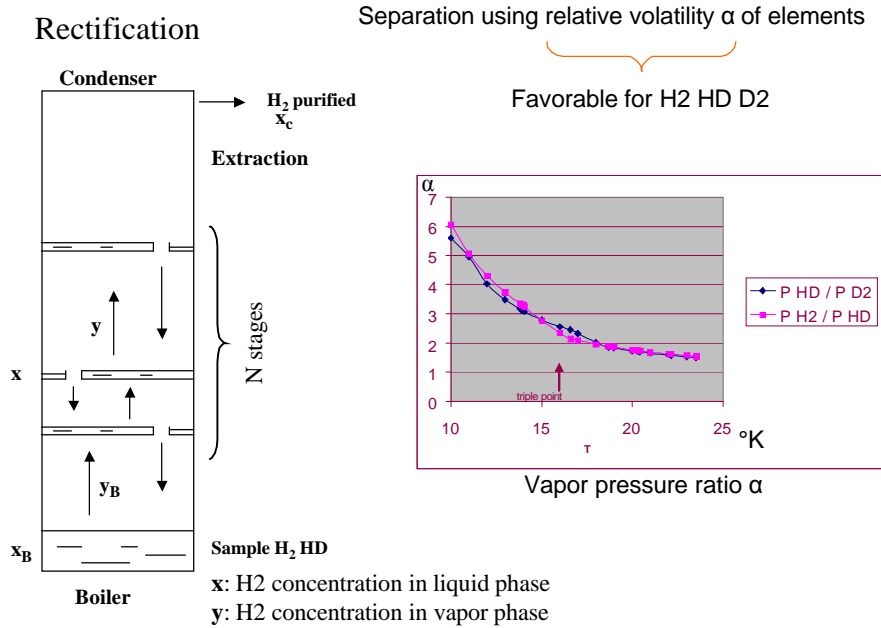Initial concentration needed to polarized an HD target

H2 :   0.2%   0.5 %
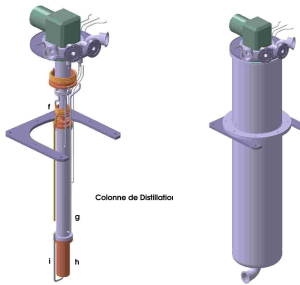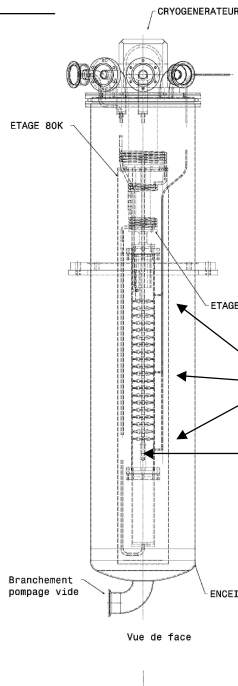
D2 :   < 0.1 % pD_2 decay constant too long

Maximum purity of commercial HD ~0.6 % for both H2 and D2

Need to produce ultra pure HD

Rectification techniques

Rectification

Condenser

H_2 purified
x_c

Extraction

N stages

x

y

y_B

x_B

Sample H_2 HD

Boiler

x: H2 concentration in liquid phase
y: H2 concentration in vapor phase

Separation using relative volatility α of elements

Favorable for H2 HD D2

Vapor pressure ratio α

Orsay Still

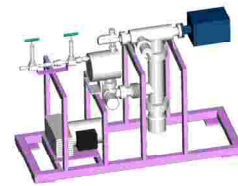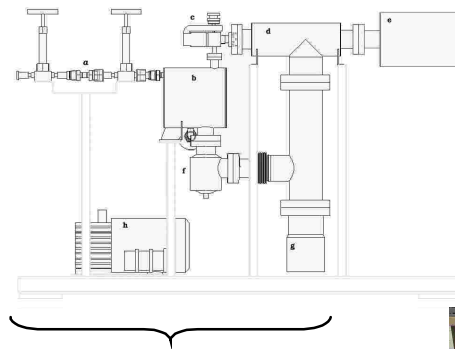CRYOGENERATEUR

ETAGE 80K

ETAGE 20K

Colonne de Distillation

Three extraction points ⇒ To the mass spectrometer
Three temperature probes

Stainless steel column with Stedman packing

Branchement
pompage vide

ENCEINTE

Vue de face

Concentration Measurements

MKS Microvision Plus Quadrupole Mass Spectrometer

yse Mass from 1 to 6

Gaz Input Manifold

Measure [H2] down to 5×10^{-4}

Measure [D2] down to 10^{-5}

Manifold of the still

Three Extraction Valves

Still

Quadrupole Mass Spectrometer

Extraction tanks
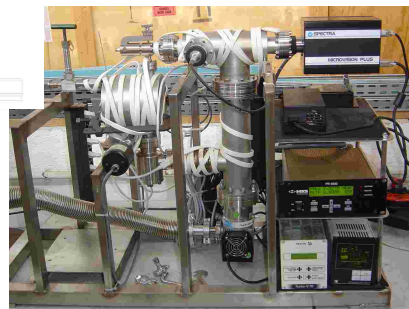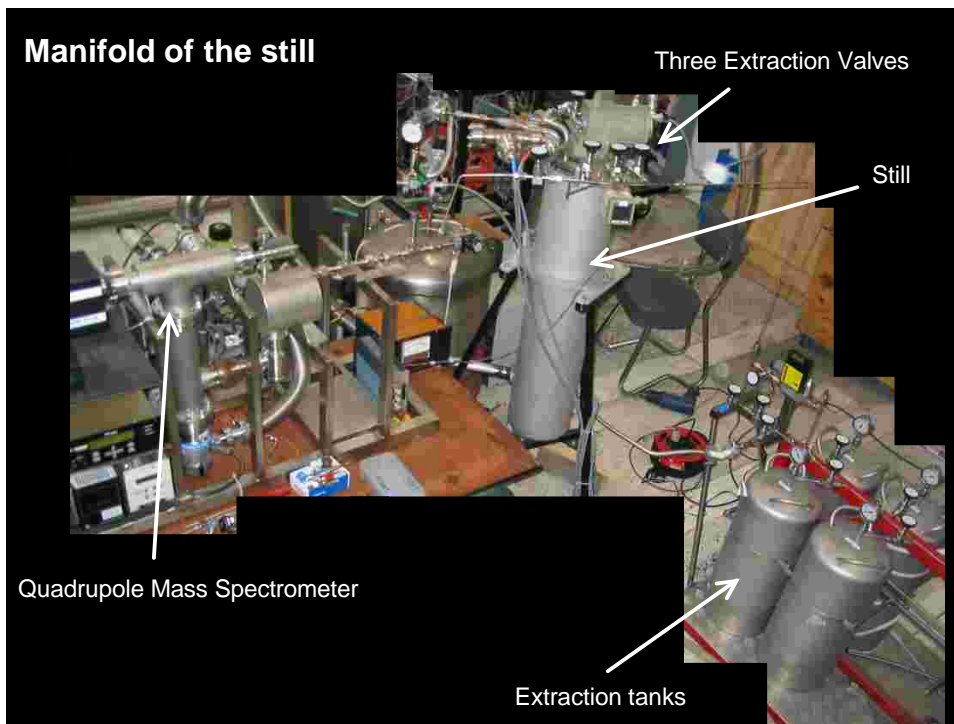
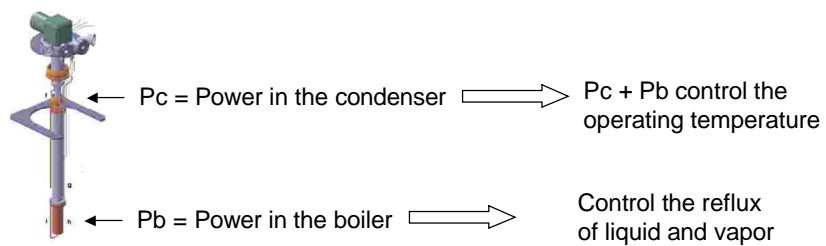**Still at Work**

➢Efficiency of the column is given by:

Number of Theoretical Stages: NTS

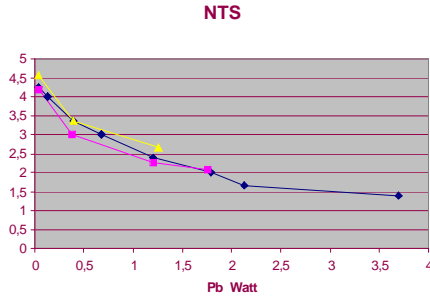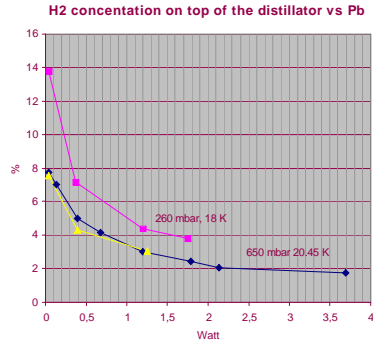$$NTS = \frac{\ln(x_c(1-x_b)/x_b(1-x_c))}{\ln(\alpha)} - 1 \qquad \text{(Fenske Relation)}$$
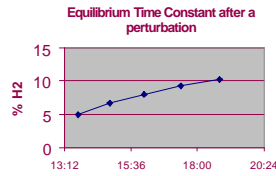
➢Control of the distillation

← Pc = Power in the condenser ⟹ Pc + Pb control the operating temperature

← Pb = Power in the boiler ⟹ Control the reflux of liquid and vapor

Still at Work

Initial commercial HD sample: 6.5 moles   [H2] = 0.5 %   [D2] = 0.65 %

I   Influence of Pb at various operating temperature

H2 concentation on top of the distillator vs Pb

260 mbar, 18 K

650 mbar 20.45 K

Watt

NTS

Pb Watt

⟹ Lowest boiling rate:

Equilibrium Time Constant after a perturbation

% H2

13:12   15:36   18:00   20:24

Still at Work

II   Influence of Temperature (Pc) at lowest boiling rate

H2 concentration vs Temperature of the condenser   Pb = 0.035 W

150 mbar

180 mbar

α=2.12

α=2.06

268 mbar

α=1.90

NTS = 4.20

α=1.85

383 mbar

650 mbar

α=1.71

915 mbar

α=1.65

1143 mbar

1470 mbar

α=1.59

α=1.55

%

°K
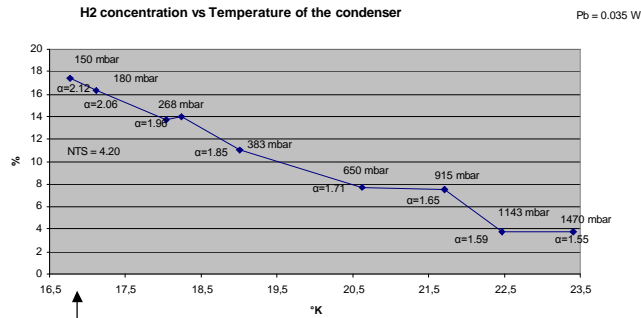
Concentration of H2 x32

⟹   Working at lowest boiling rate and lowest temperature

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III   Extracting the HD

% H2

% D2

15 days   7 days

1   2   3

H2 rich   *H2 poor*   *D2 rich*
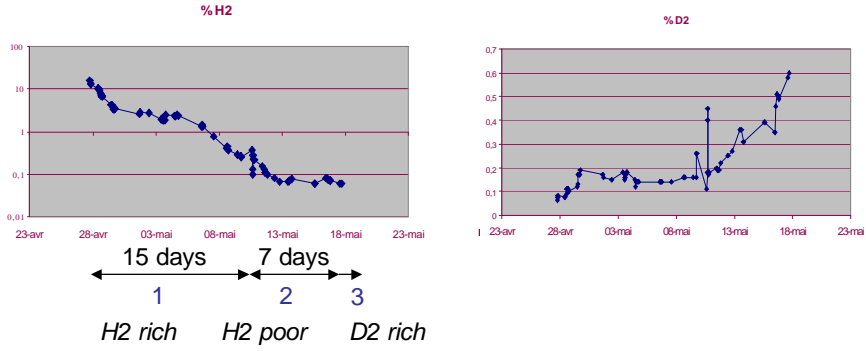
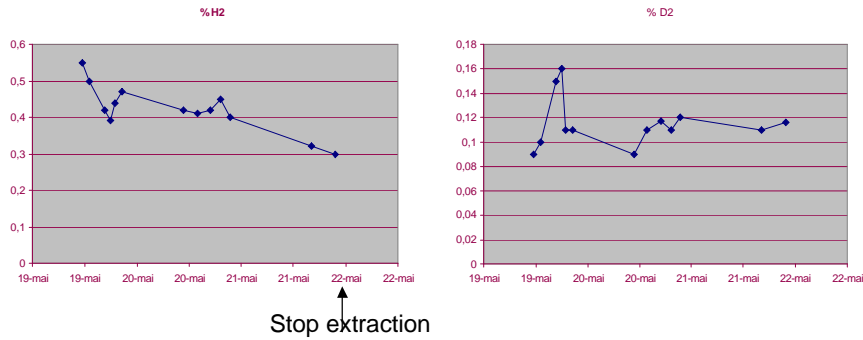1   1.44 moles extracted: [H2] = 2.46 %   [D2] = 0.157 %

2   3.5   moles extracted: [H2] = 0.08 %   [D2] = 0.49 % ⟹ 2nd distillation

3   1.5   moles extracted: [H2] = 0.06 %   [D2] = 2.52 %

IV   Double Distillation

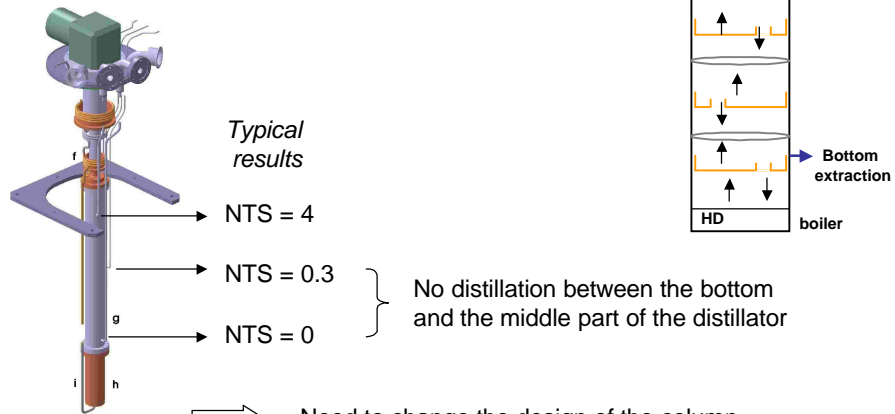**% H2**

% D2

Stop extraction

4   300 mmoles extracted: [H2] = 0.39 %   [D2] = 0.11 %

One month of aging is already saved for static polarization

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Improvement of the Still

Increasing the efficiency of the column:

Typical results

→ NTS = 4

→ NTS = 0.3

→ NTS = 0

No distillation between the bottom and the middle part of the distillator

Middle extraction

Bottom extraction

HD   boiler

⟹ Need to change the design of the column

Test of classical plates in the bottom part
to retain more liquid in the column

Summary and Outlooks

0ne distillation as been done and gives good results.

Ready for the next step:
        Test of new configuration of the column packing
                Effect of Stedman Packing density
                Test of classical plates in the bottom part of the column

Next Highlights:

Systematic measurements of relaxation time vs. initial
concentration of H_2, D_2 and aging

Test of reverse conversion oD2 → pD2 at room temperature

relaxation time

$[H_2] = 0,26 +- 0,02$

$[D_2] = 0,23 +- 0,02$

Proton Relaxation times versus Aging time
Double distilled HD from LSC

$T_1^H(0.5K;5.9T)$ $T_1^H(0.5K;1.0T)$ $T_1^H(1.9K;1.0T)$

$T_1^H(0.5K;1.0T) \sim e^{t/80}$

10 jours

$T_1^{-1}(0.5K;5.9T) \sim e^{d/5.5}$

$T_1^H(1.9K;1.0T) \sim e^{d/17.9}$

T_1^H (sec)

Aging Time (days)

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