Polarization and Distillation of HD
for solid HD targets

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- Introduction to solid HD target Polarization
- Distillation apparatus at Orsay
- First test run and results
- Summary and outlooks

HD solid state polarized targets offer:

High Dilution Factor: All nucleus are polarizable
Long relaxation time: Nuclear spin – lattice coupling switch off

How to polarized HD target?

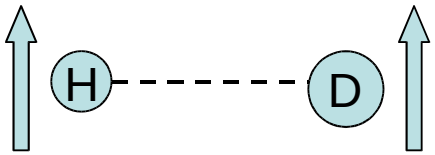
Static polarization.
Dynamic Nuclear Polarisation

What quality of HD do we need?

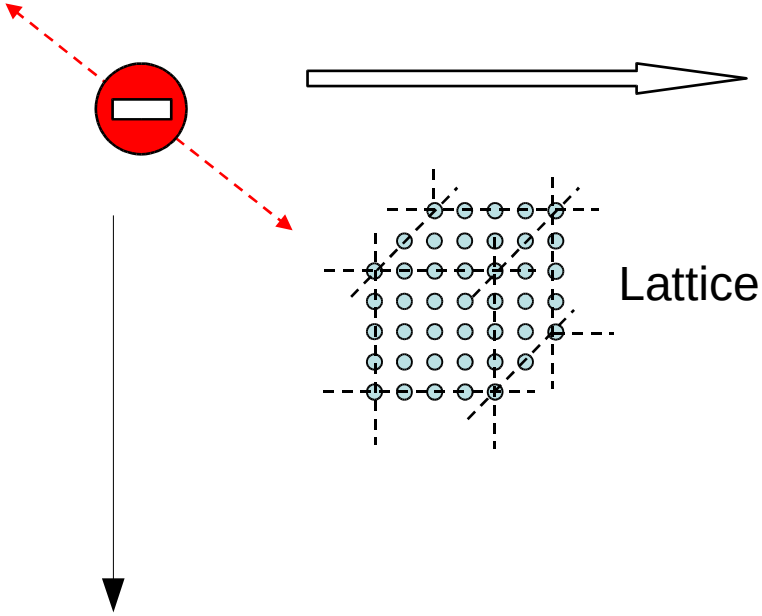
Static Polarization of HD targets

Solid HD at low temperature

(Honig, 1967)



Polarized HD Molecule



High relaxation time at ~1K temperature

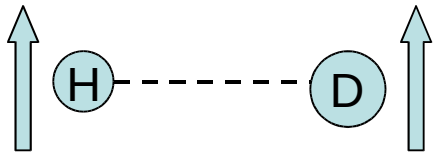
Lattice of the HD crystal

Good for DNP.

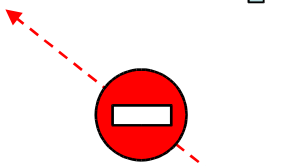
Problematic for Static polarization (target unpolarizable)

Static Polarization of HD targets

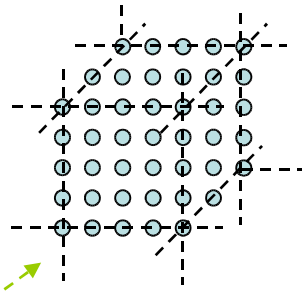
(Honig, 1967)



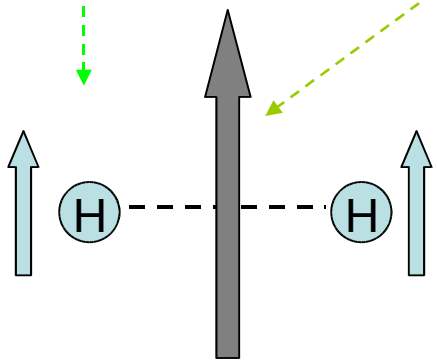
Polarized HD Molecule



Spin spin interaction



Lattice of the HD crystal



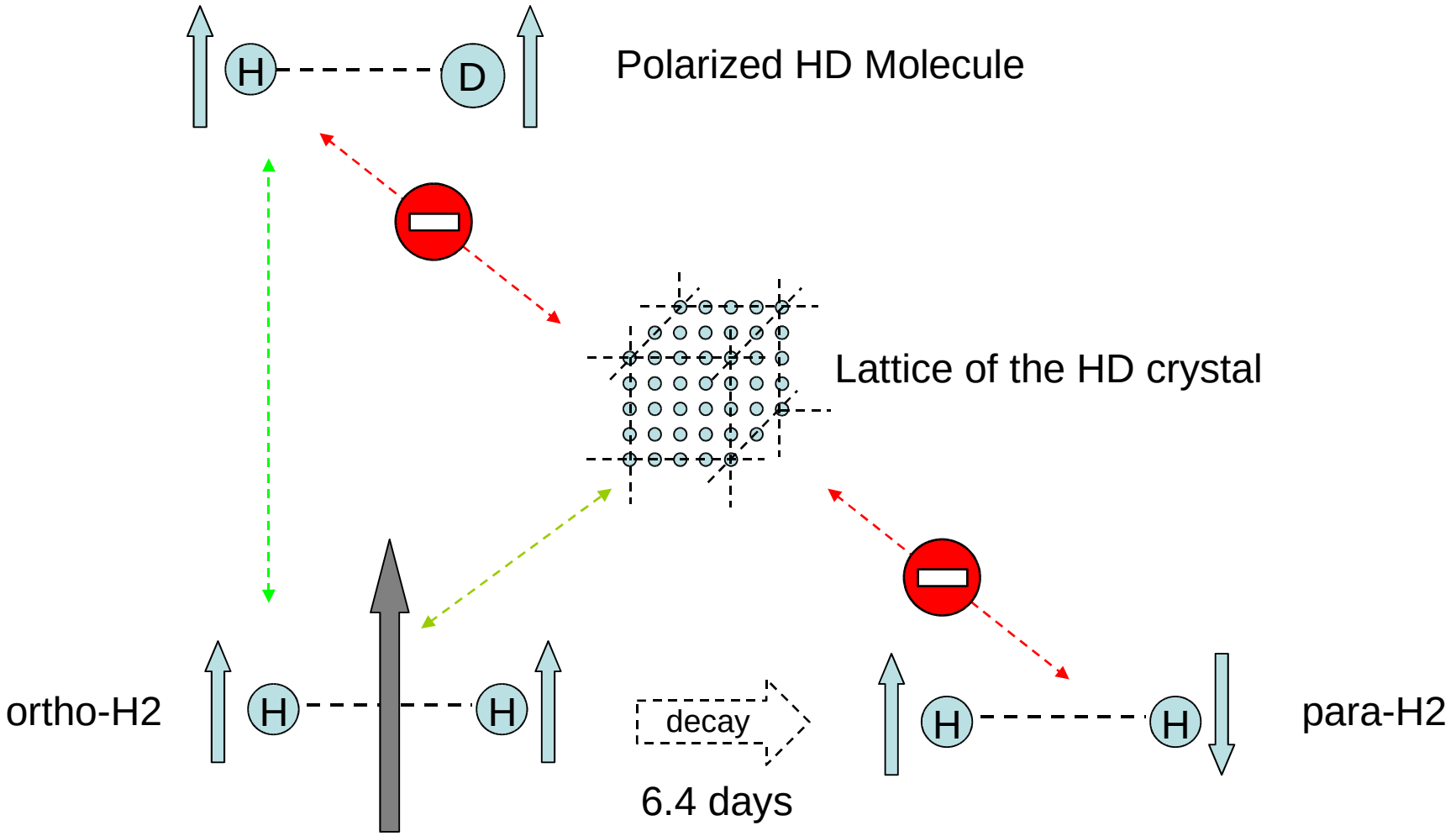
ortho-H2

Target can be polarized

Temperature and field: ~10 mK - ~15 T

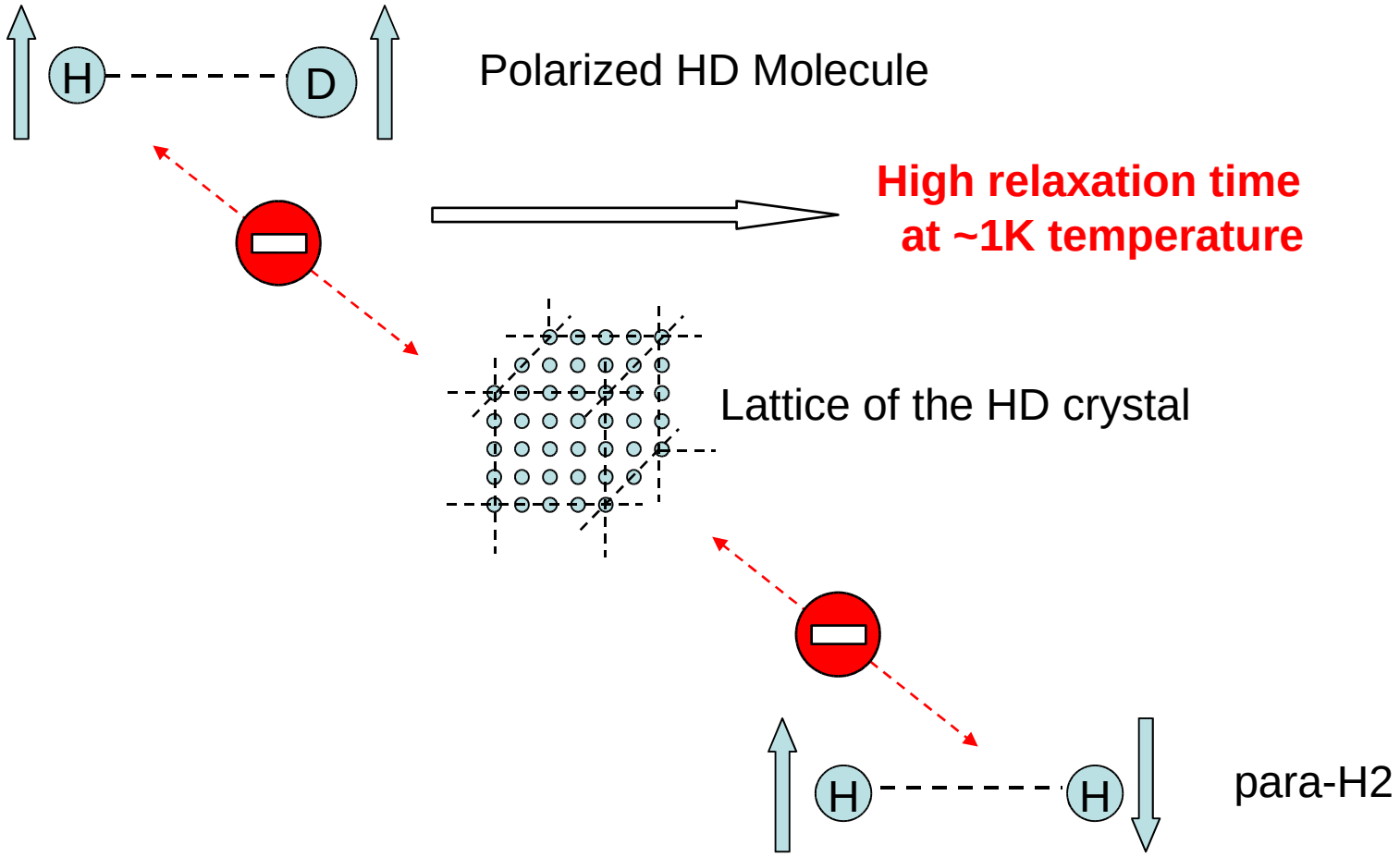
Static Polarization of HD targets

(Honig, 1967)



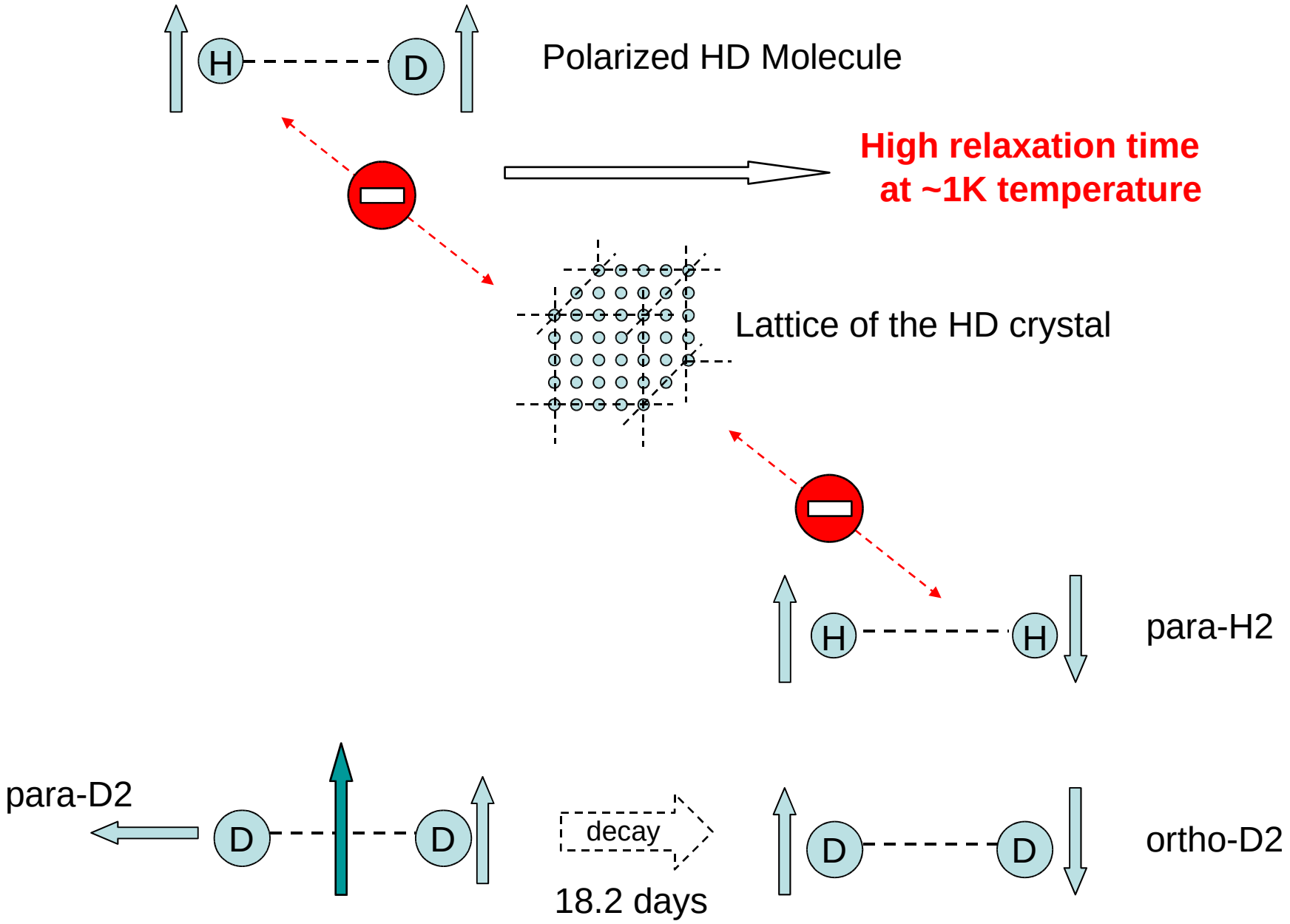
Static Polarization of HD targets

(Honig, 1967)



Static Polarization of HD targets

(Honig, 1967)



Static Polarization of HD targets

The three steps for static polarization

I

Dilution refrigerator

10 mK
13-15 Tesla

Static polarization
+ Aging (oH2 -> pH2)

II

Transport cryostat

2 – 4 K
1 - 2 T

Transportation

III

In Beam Cryostat

0.2 - 1.5 K
1.5 Tesla

On site exploitation

Initial concentration Needed

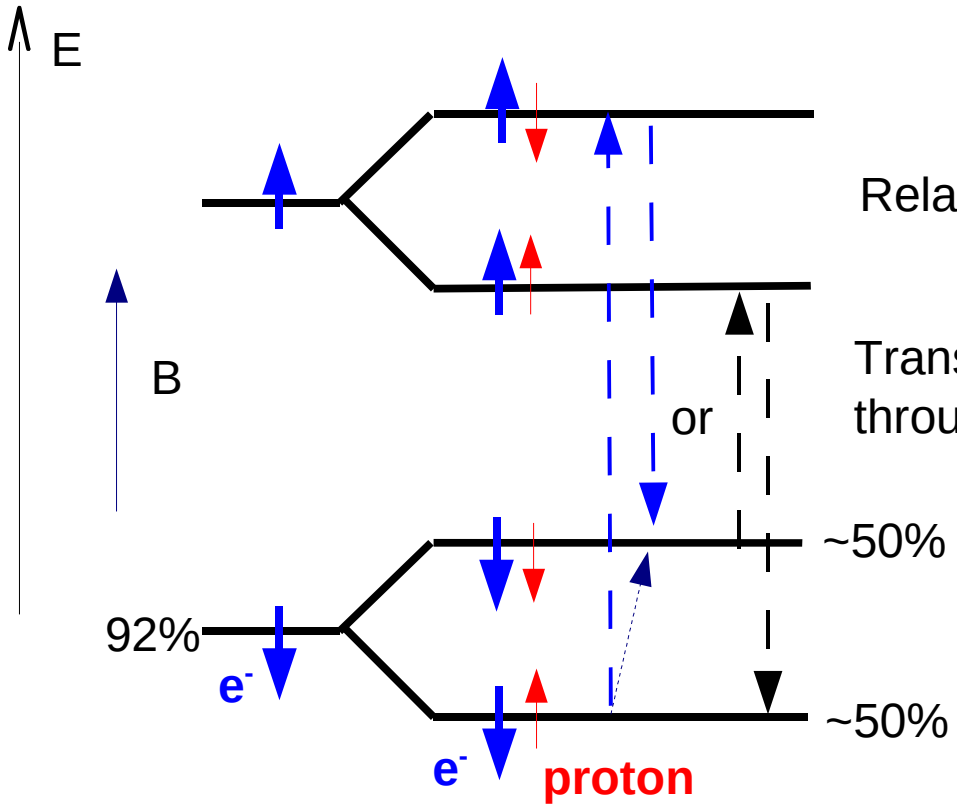
H2: 0.1 0.2 %
D2: < 0.01%

Need to purify HD

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

Dynamic Polarization of HD targets

Adding impurity: **free electrons**. For B=2.5 T and T = 1 K, e^- polarization = 92%



Relaxation time proton >> electron

Transitions made possible
through microwave excitation: ~70GHz

Initial concentration Needed

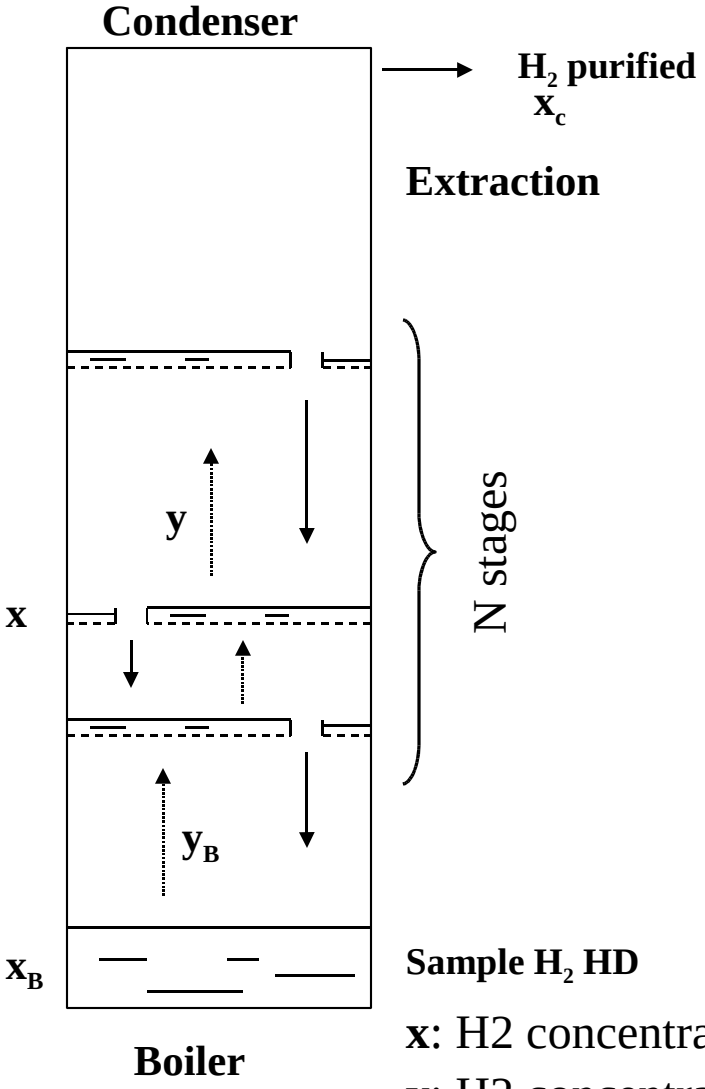
H2: < 0.05 %
D2: < 0.05%

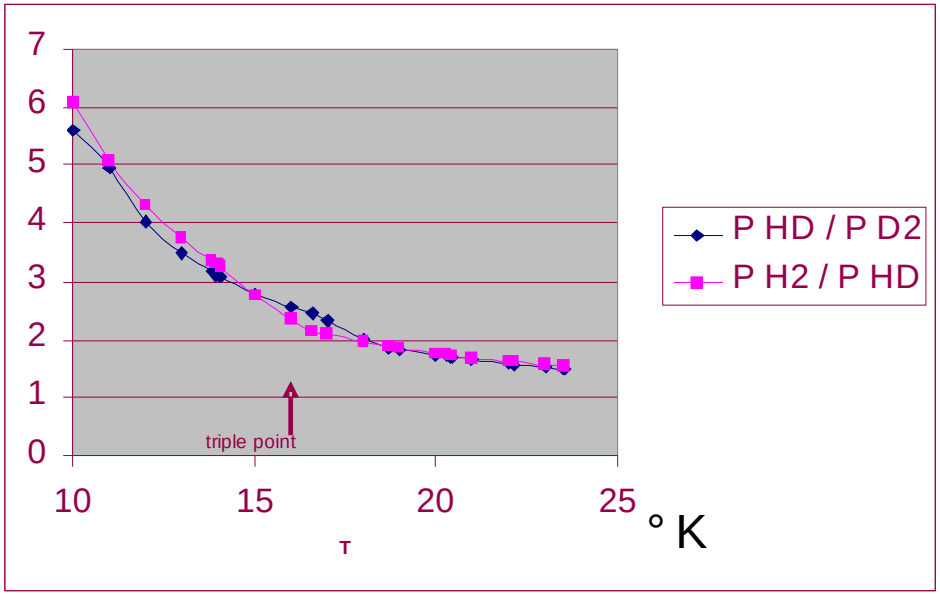
Need to purify HD

Rectification

Separation using relative volatility of elements

Favorable for H2 HD D2



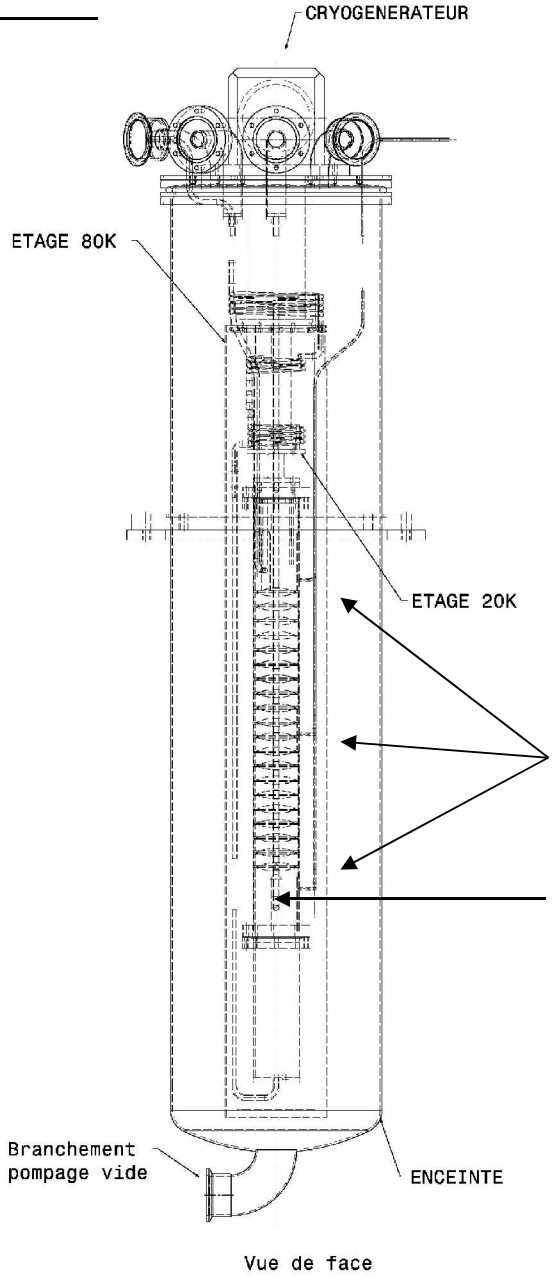


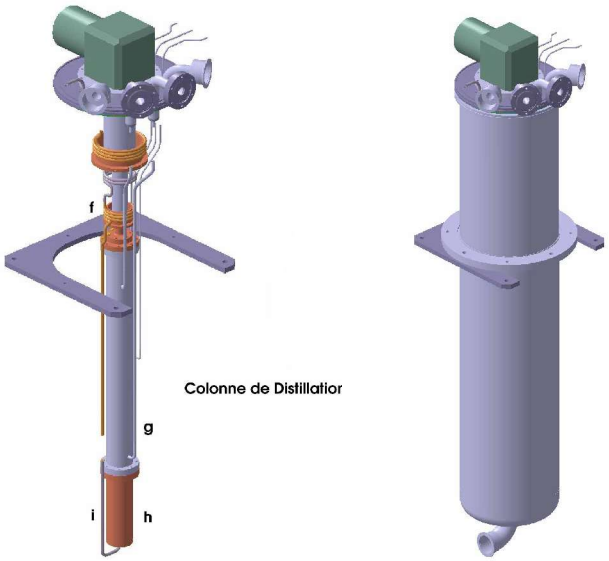
Vapor pressure ratio

x: H2 concentration in liquid phase

y: H2 concentration in vapor phase

Orsay Still





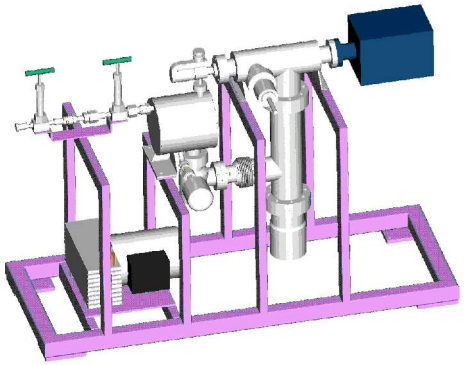
Three extraction points ⇒ To the mass spectrometer or extraction tanks

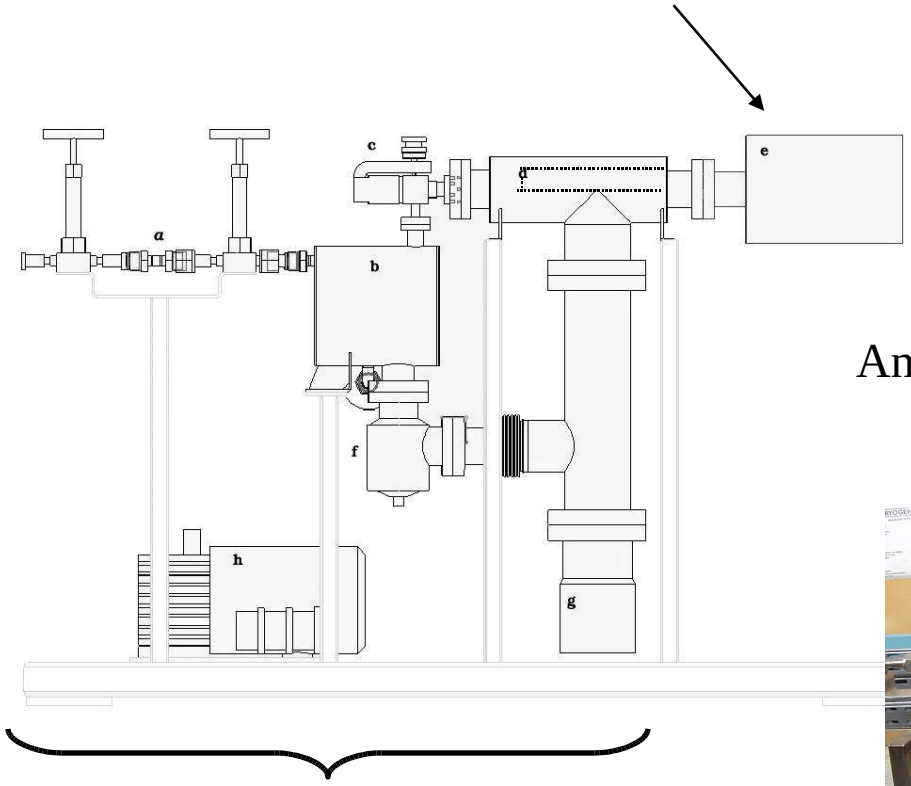
Three temperature probes

Stainless steel column with Stedman packing

Concentration Measurements

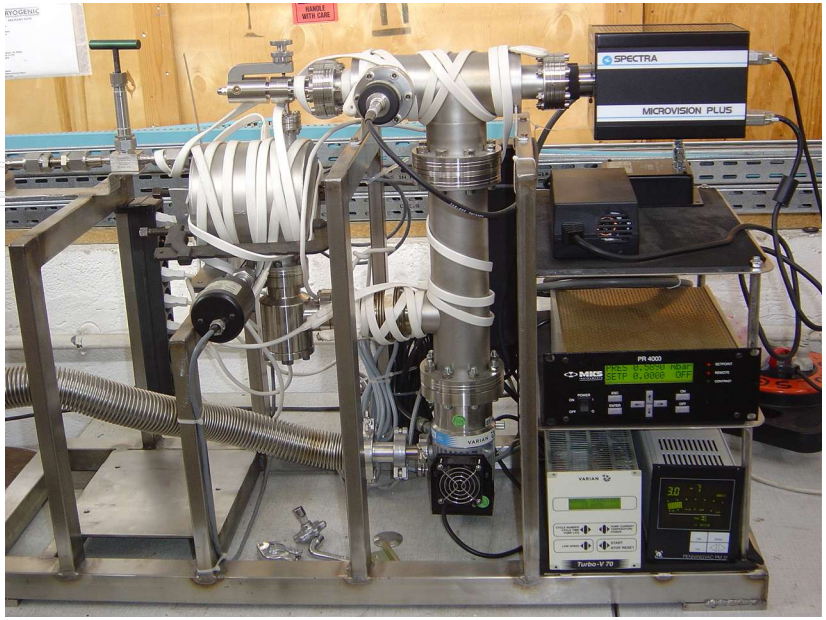
MKS Microvision Plus Quadrupole Mass Spectrometer





Analyse Mass from 1 to 6

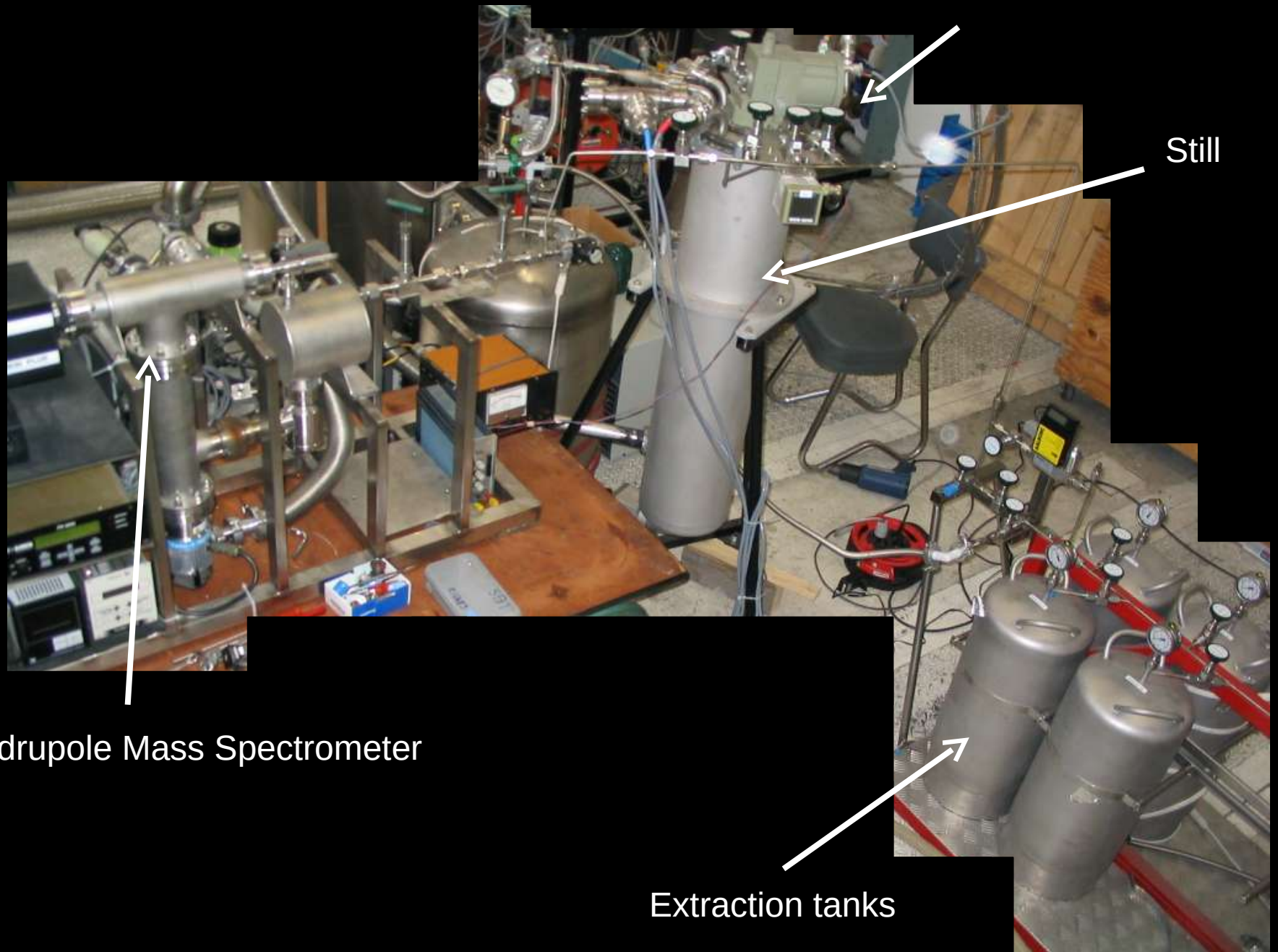
Gaz Input Manifold



Measure [H2] down to $2\ 10^{-4}$

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

Manifold of the still



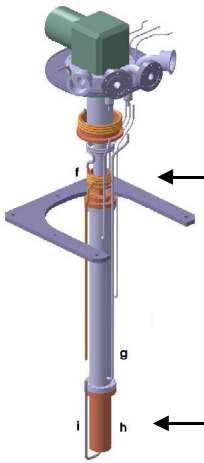
Still at Work

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



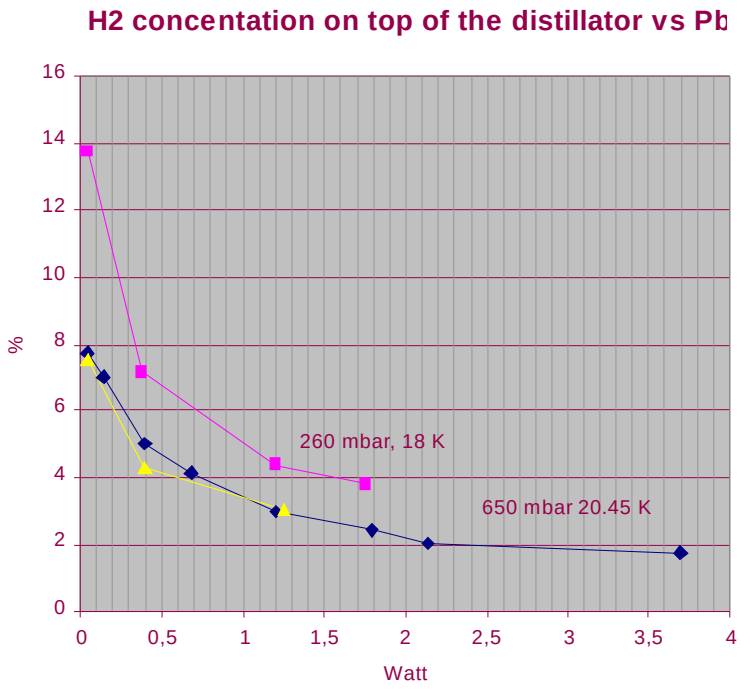
← Pc = Power in the condenser ⇨ Pc + Pb control the operating temperature

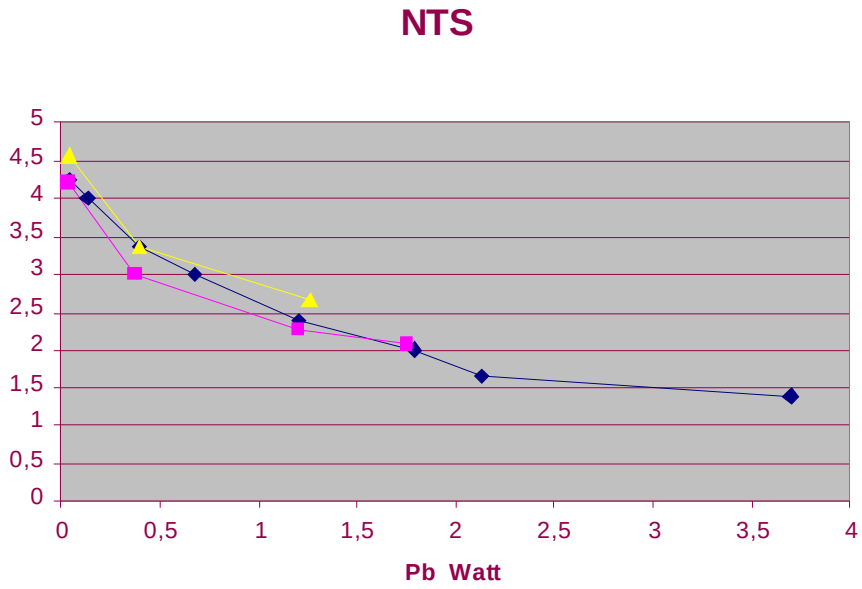
← Pb = Power in the boiler ⇨ Control the reflux of liquid and vapor

Still at Work

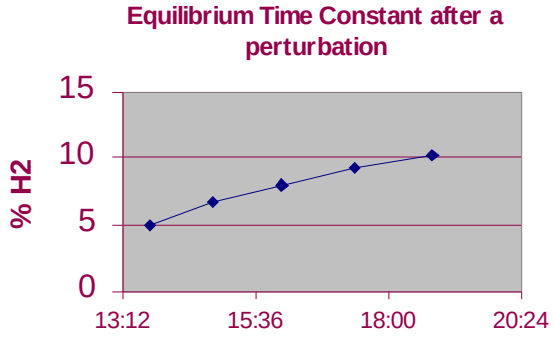
Initial commercial HD sample: 6.5 moles [H2] = 0.5 % [D2] = 0.65 %

I Influence of Pb at various operating temperature

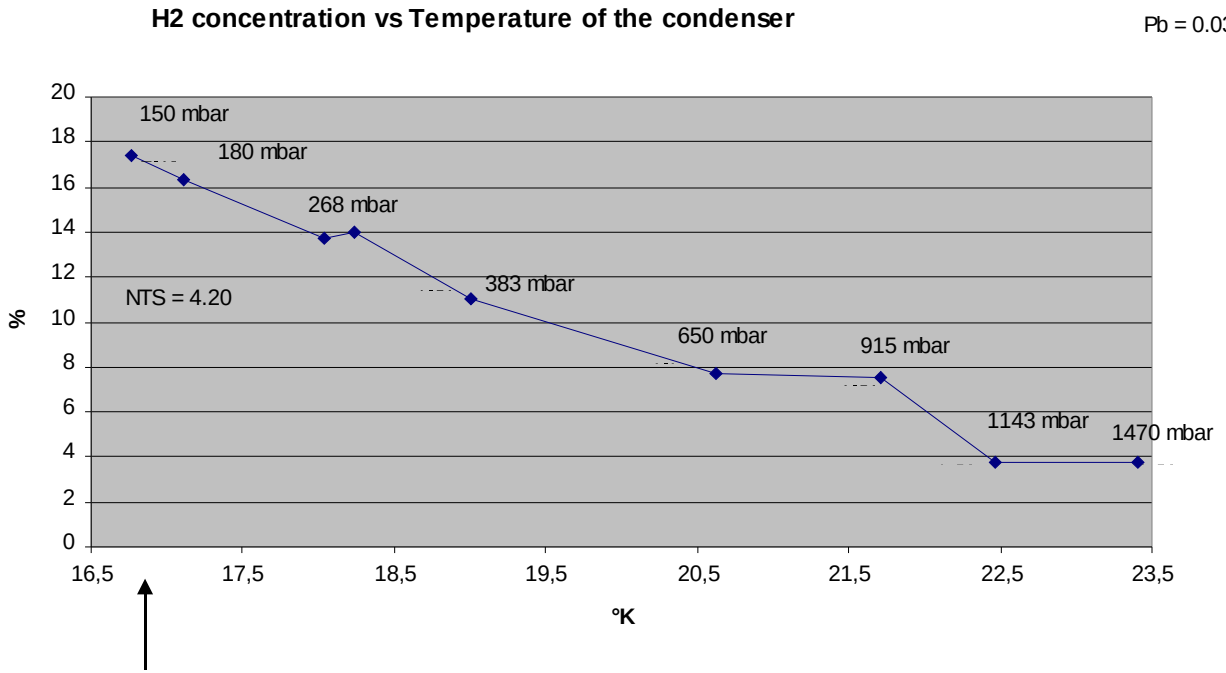




⇨ Lowest boiling rate:



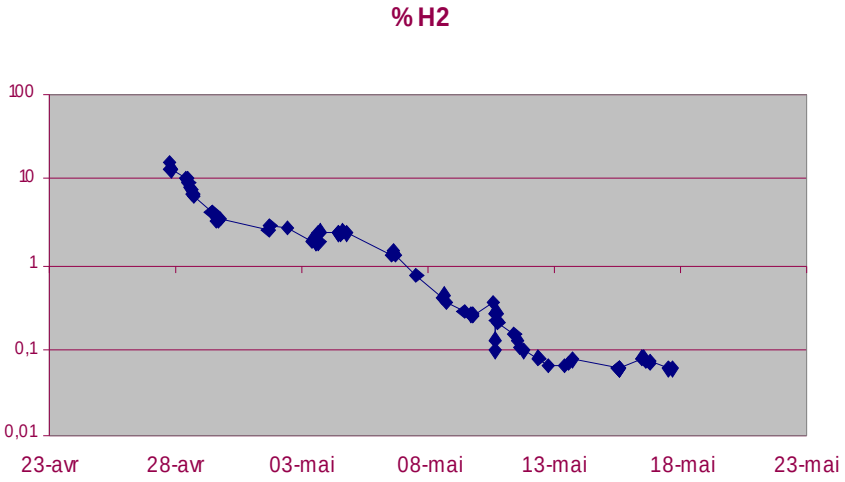
II Influence of Temperature (Pc+Pb) at lowest boiling rate

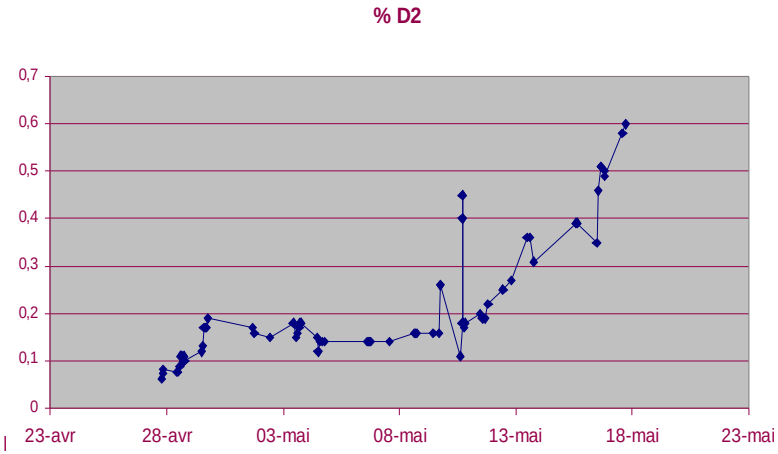


Concentration of H2 x32

⇒ Working at lowest boiling rate and lowest temperature

III Extracting the HD





15 days 7 days

1 2 3

H2 rich *H2 poor* *D2 rich*

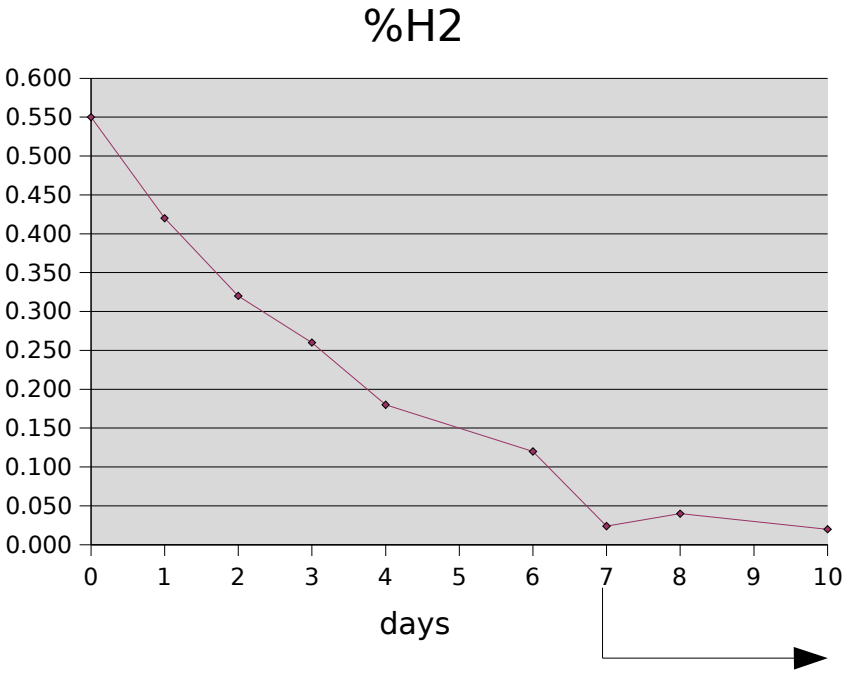
1 1.44 moles extracted: [H2] = 2.46 % [D2] = 0.157 %

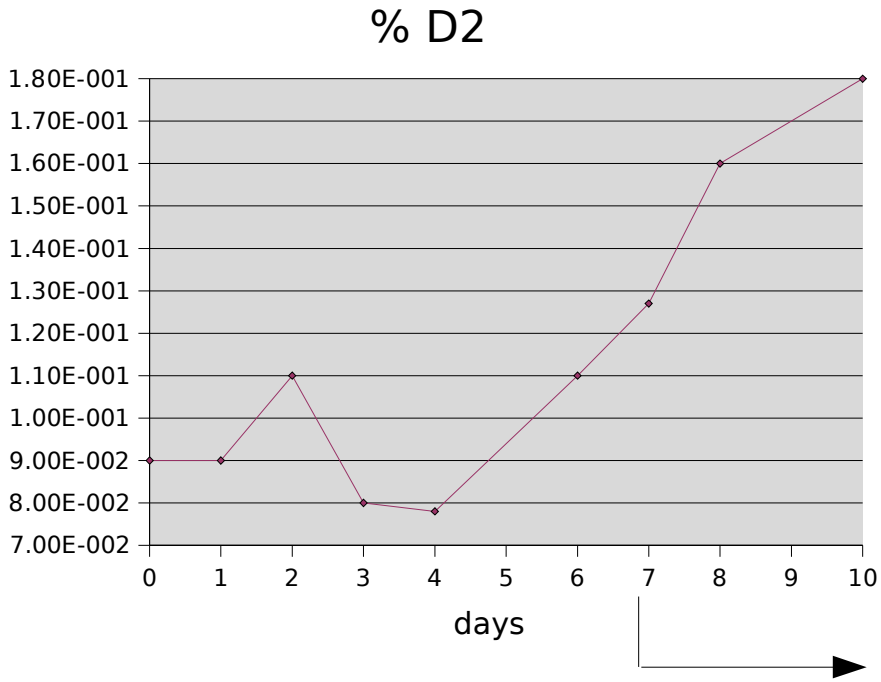
2 3.5 moles extracted: [H2] = 0.08 % [D2] = 0.49 % ⇨ 2nd distillation

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

Still at Work

IV Double Distillation



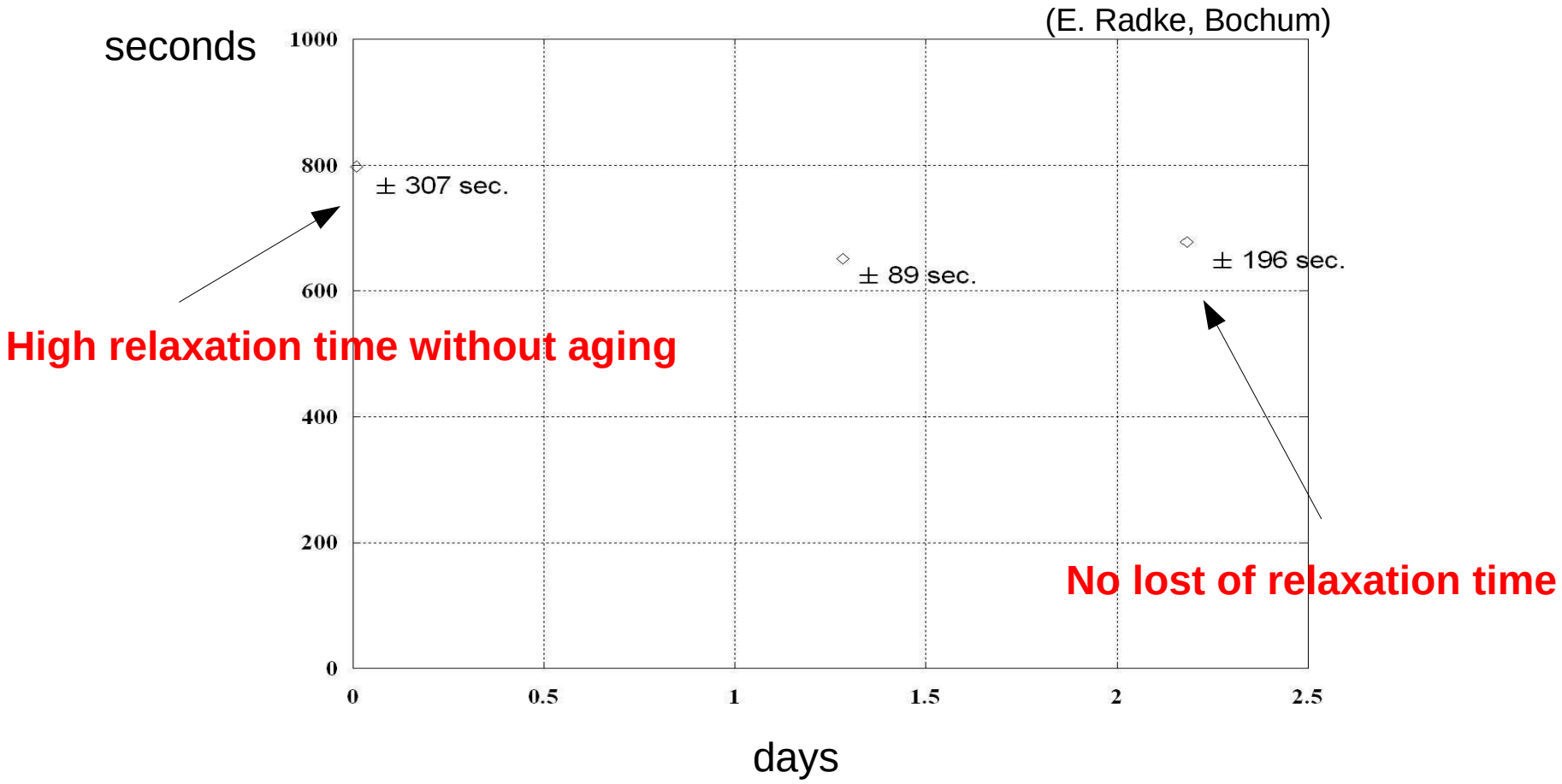


4 334 mmoles extracted: [H2] < 0.02 % [D2] = 0.17 %

DNP sample

Relaxation Time

Relaxation time was measured in Bochum in July 2005 under radiation



Optimum radiation dose in 7 days

Summary and Outlooks

0ne distillation has been done and gives good results.

More than 1 month of aging saved for static polarization

Promising results on relaxation time for Dynamic polarization.

Next Highlights:

Test of new configuration of the column packing

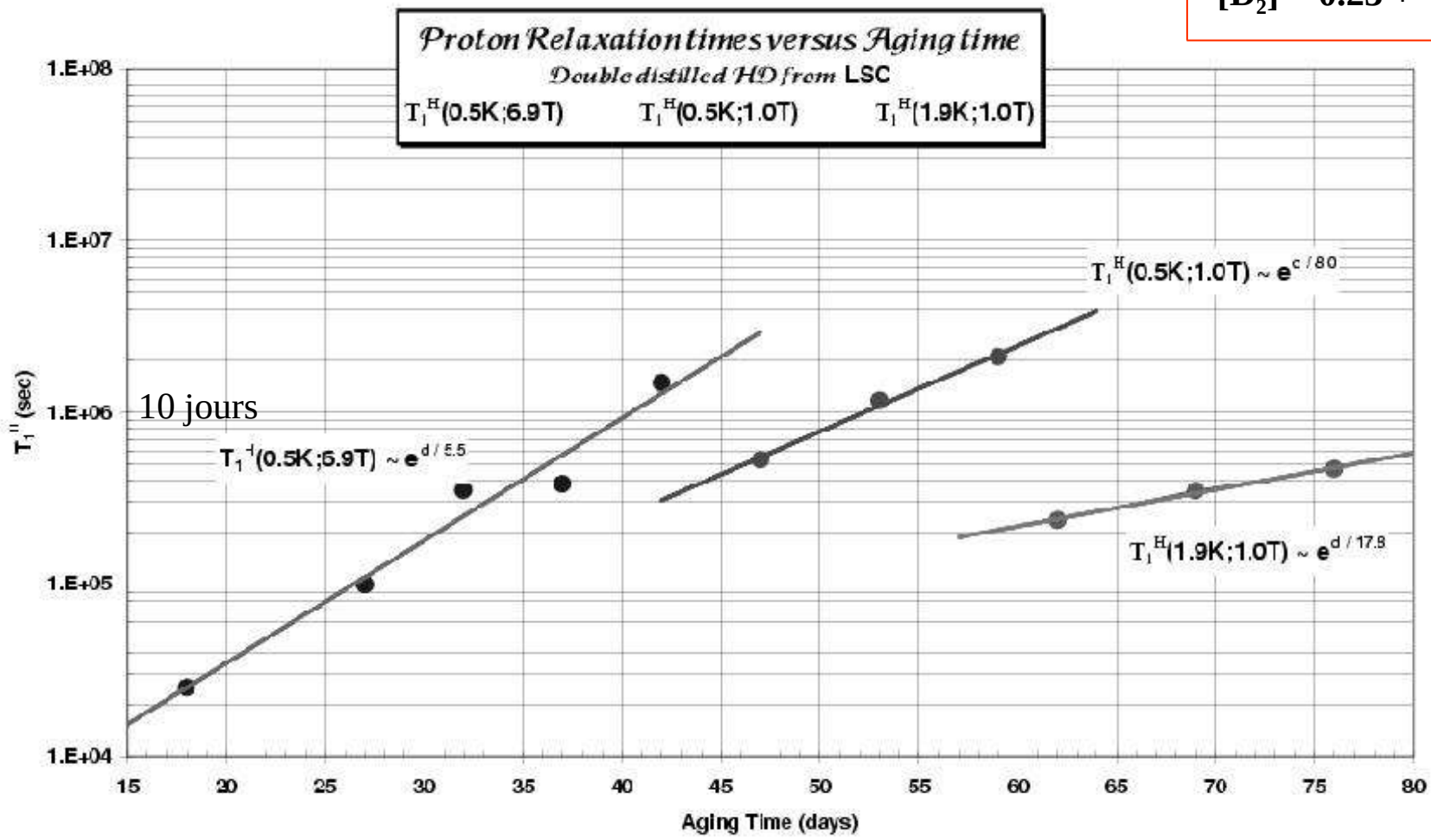
Systematic measurements of relaxation time vs. initial concentration of H_2, D_2 and aging

Pure HD Target HYDILE

relaxation time

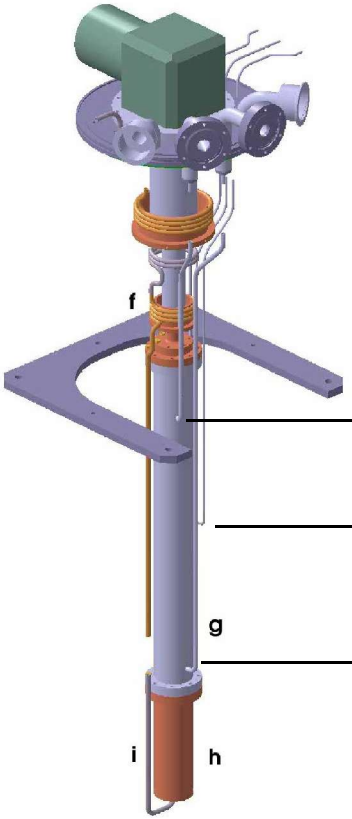
$[H_2]$ = 0.26 +- 0.02

$[D_2]$ = 0.23 +- 0.02



Improvement of the Still

Increasing the efficiency of the column:



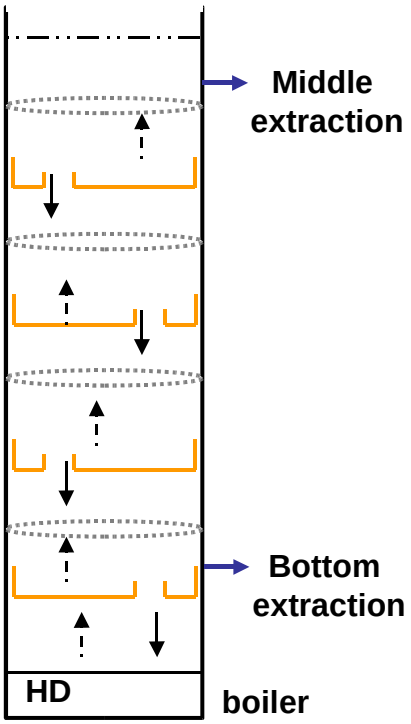
Typical results

NTS = 4

NTS = 0.3

NTS = 0

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



⇨ Need to change the design of the column

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


SPECTRA
MICROVISION PLUS
PR 4000
MKS
Turbo-V 70
VARIAN


AALBORG
MASS
FLOW
CONTROLLER
GFC