

Status of the Distillation Apparatus

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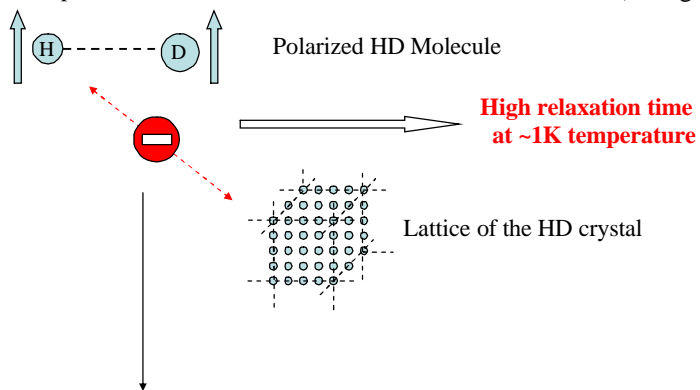
- o Introduction to solid HD target Polarization
- o Distillation apparatus at Orsay
- o First test run and results
- o Improvement of the Apparatus

Polarized Nucleon Targets for Europe, Rech Germany, February 2006

Static Polarization of HD targets

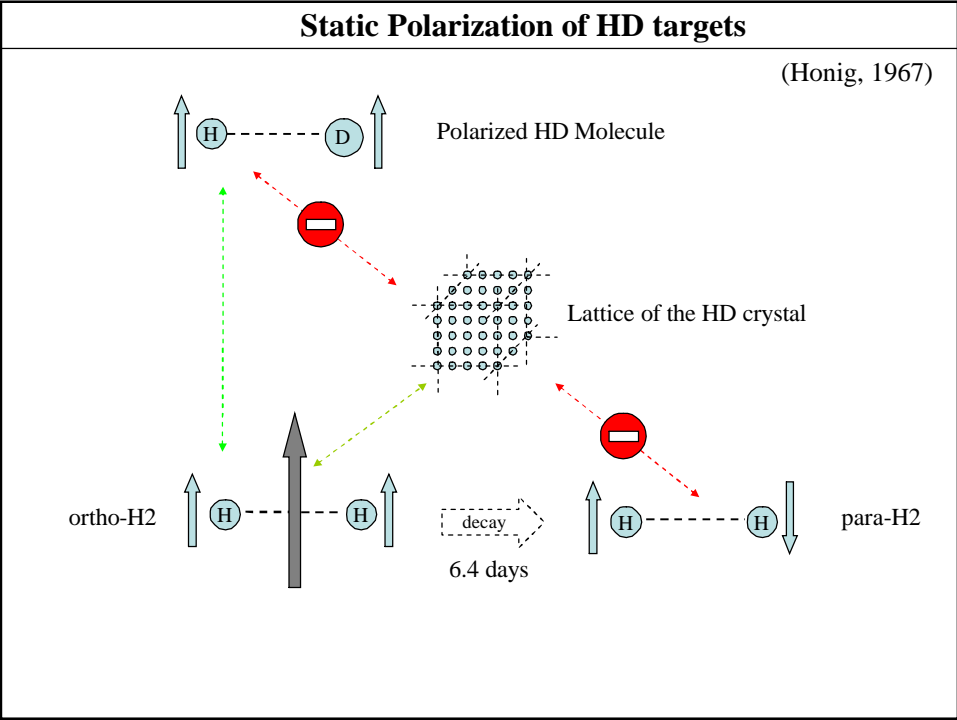
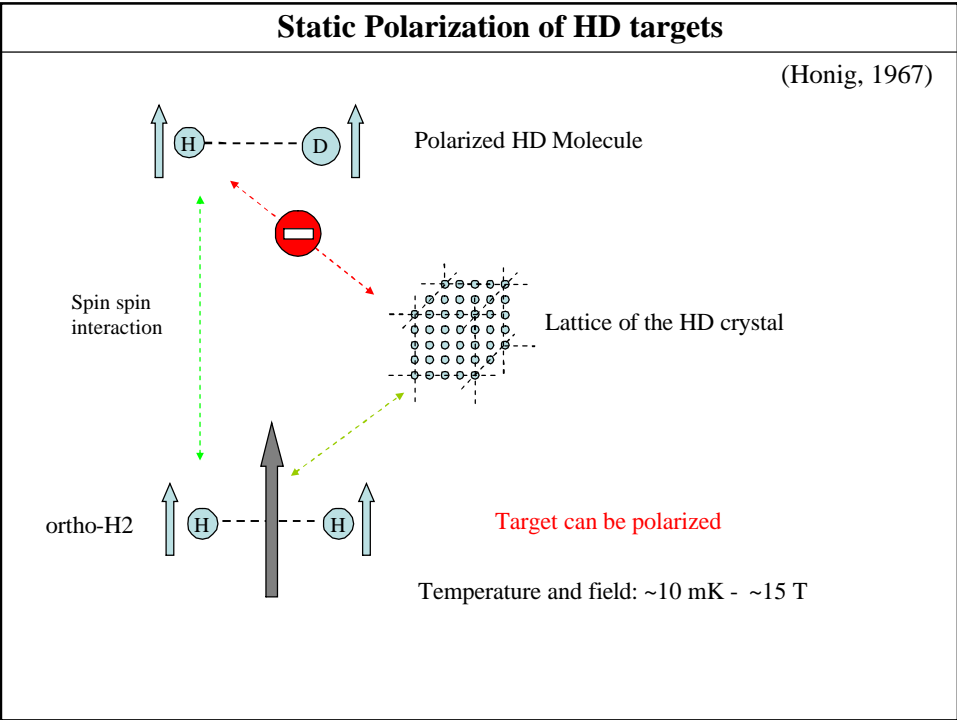
Solid HD at low temperature

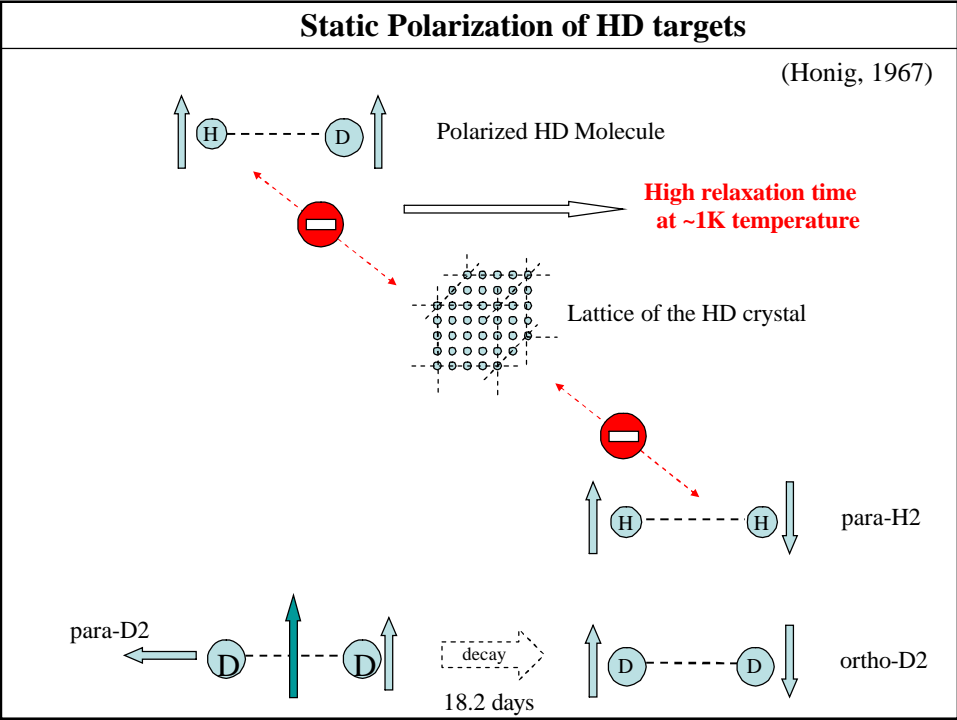
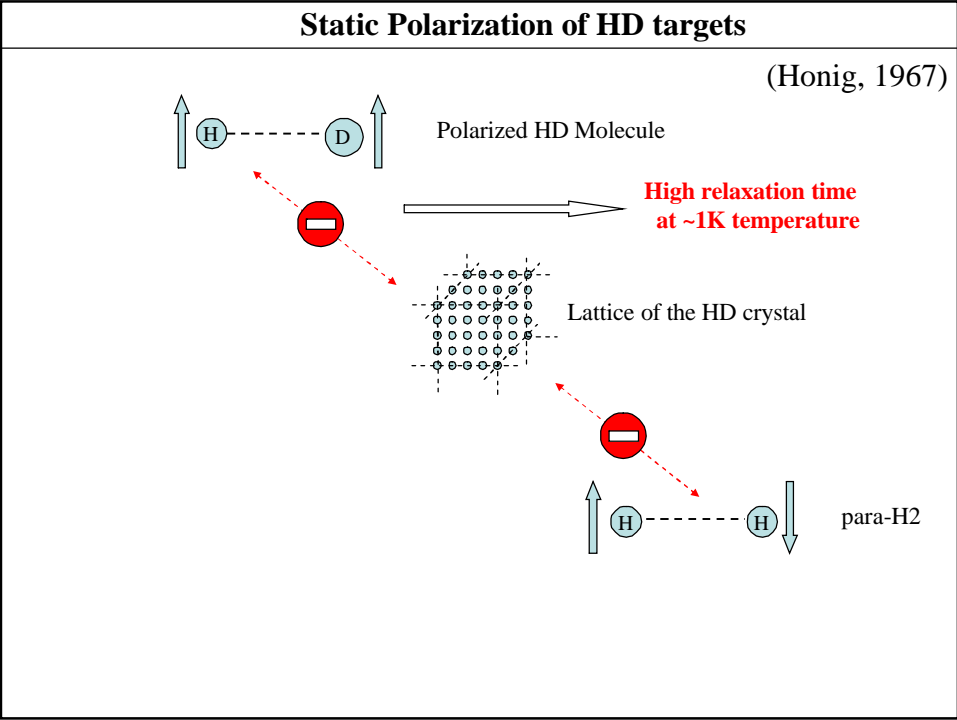
(Honig, 1967)



Good for DNP.

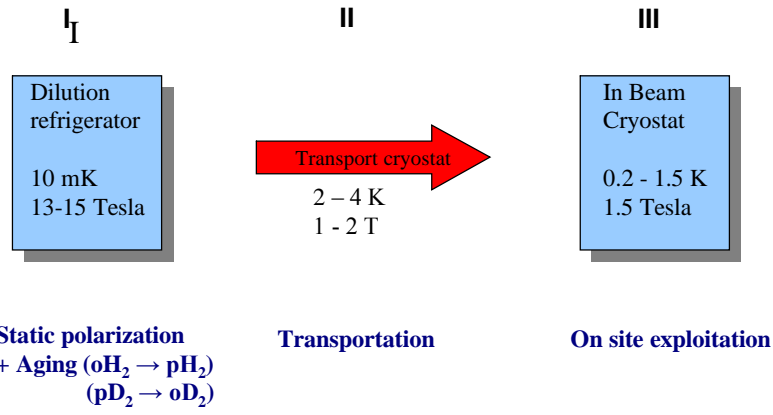
Problematic for Static polarization (target unpolarizable)





Static Polarization of HD targets

The three steps for static polarization



Initial concentration Needed

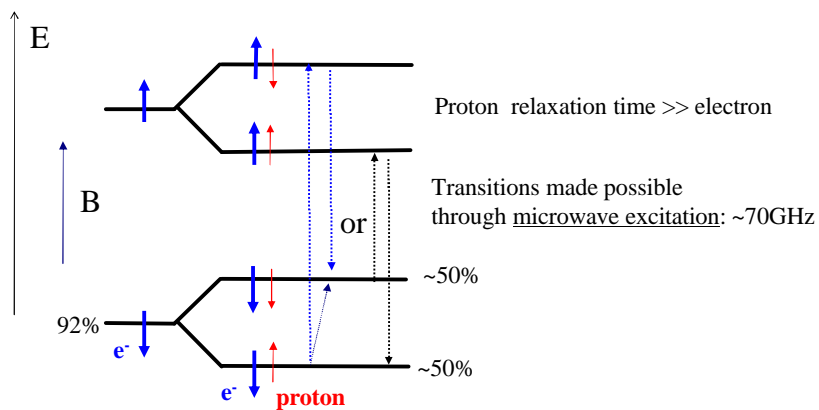
H2: 0.1 - 0.2 %
D2: < 0.01%

Need to purify HD

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

Dynamic Polarization of HD targets

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



Initial concentration Needed

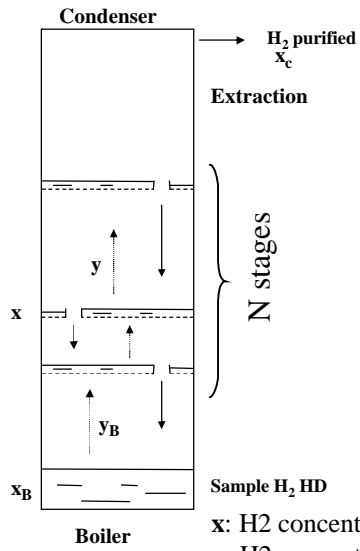
H2: < 0.05 %
D2: < 0.05%

Need to purify HD

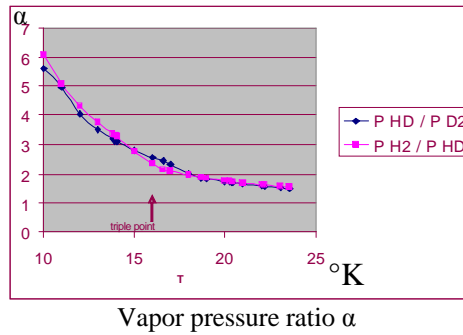
Rectification techniques

Rectification

Separation using relative volatility of elements



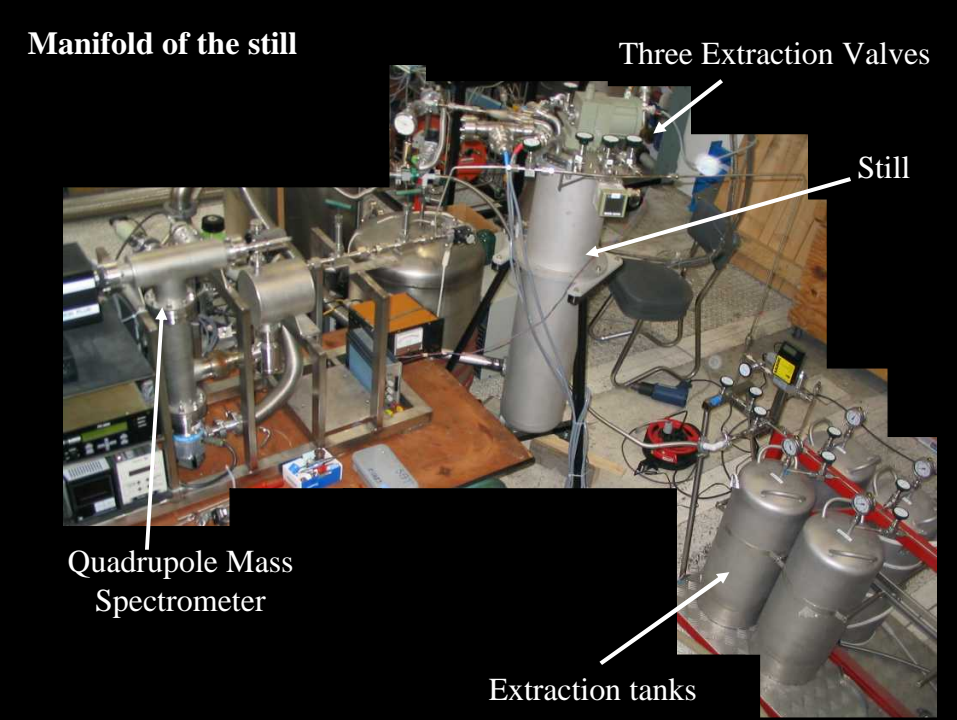
Favorable for H₂ HD D₂



Orsay Still

Colonne de Distillation

Three extraction points → **To the mass spectrometer or extraction tanks**
Three temperature probes
Stainless steel column with Stedman packing



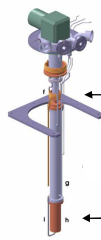
Still at Work

- Efficiency of the column is given by:

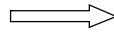
Number of Theoretical Stages: **NTS**

$$\text{NTS} = \frac{\ln(x_c(1-x_b)/x_b(1-x_c))}{\ln(\alpha)} - 1 \quad (\text{Fenske Relation})$$

- Control of the distillation

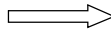


← Pc = Power in the condenser



Pc + Pb control the operating temperature

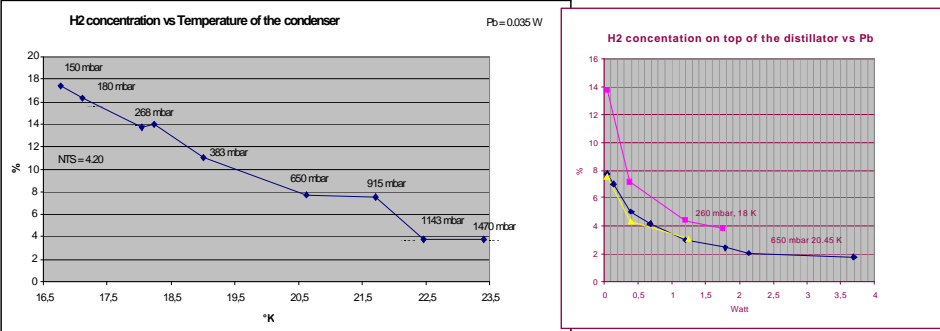
← Pb = Power in the boiler



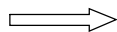
Control the reflux of liquid and vapor

Still at Work

Sample: [H2] = 0.5 % [D2] = 0.6 %



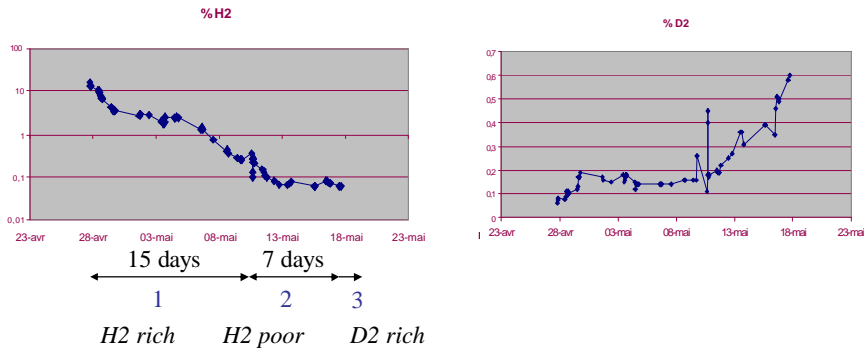
Working at lowest boiling rate and lowest temperature



Concentration of H2 **x32**

Still at Work

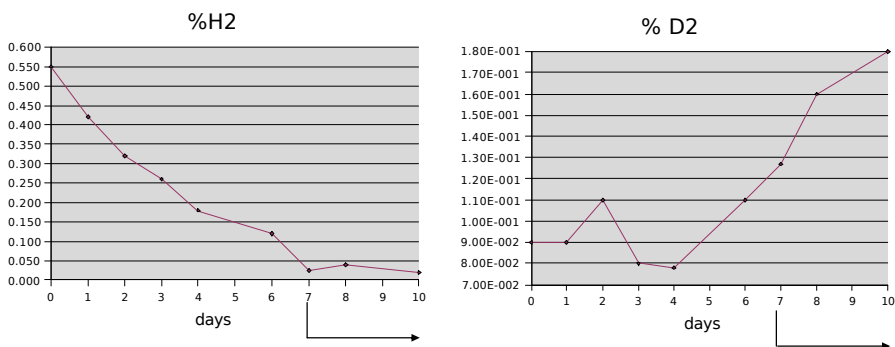
III Extracting the HD. Initial sample: [H2] = 0.5 % [D2] = 0.6 %



- 1 1.44 moles extracted: [H2] = 2.46 % [D2] = 0.157 %
- 2 3.5 moles extracted: [H2] = 0.08 % [D2] = 0.49 % \implies 2nd distillation
- 3 1.5 moles extracted: [H2] = 0.02 % [D2] = 2.52 %

Still at Work

IV Double Distillation

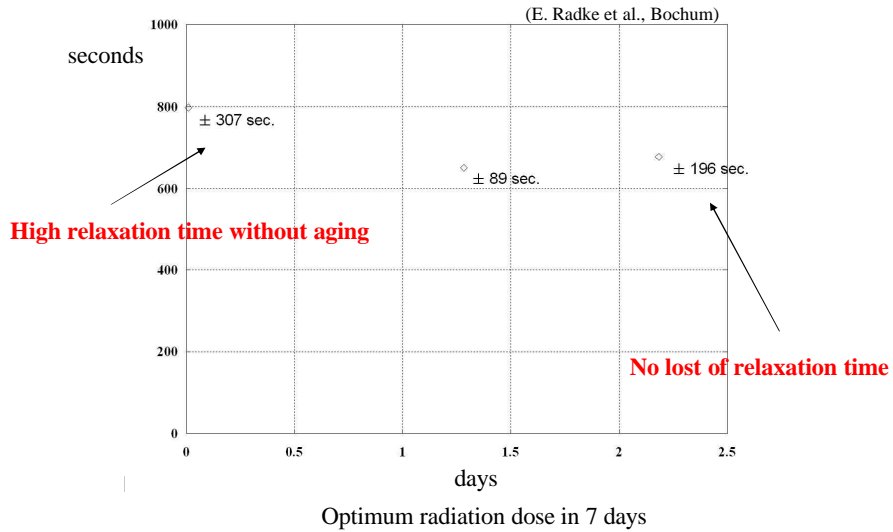


- 4 334 mmoles extracted: [H2] < 0.02 % [D2] = 0.17 %


DNP sample

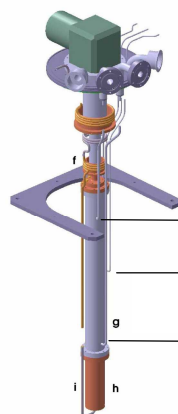
Relaxation Time of HD Sample

Relaxation time was measured in Bochum in July 2005 under radiation



Improvement of the Still

Increasing the efficiency of the column:



Typical results

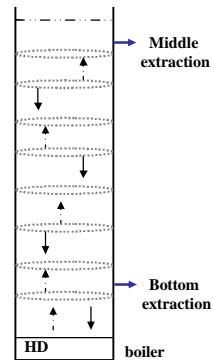
NTS = 4

NTS = 0.3

NTS = 0

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

First version

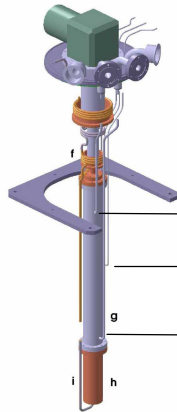


Need to change the design of the column

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

Improvement of the Still

Increasing the efficiency of the column:



Typical results

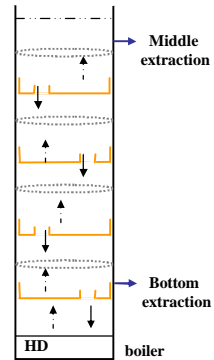
- NTS = 4.6
- NTS = 1.35
- NTS = 0

NTS = 3.2

Distillation between the bottom and the middle part of the still but less efficient on top

+ 20 % efficiency

Second version

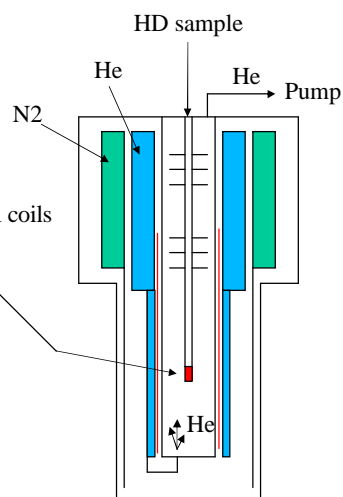


Improvement of the Apparatus

Installation of an NMR apparatus in Orsay using a variable temperature cryostat.

- Cryostat can run from 1.5 K to 30 K
- Field up to 2 Tesla
- We made a new insert equipped with NMR coils contains 200 mmoles of HD.

NMR System is ready.



Summary and Outlooks

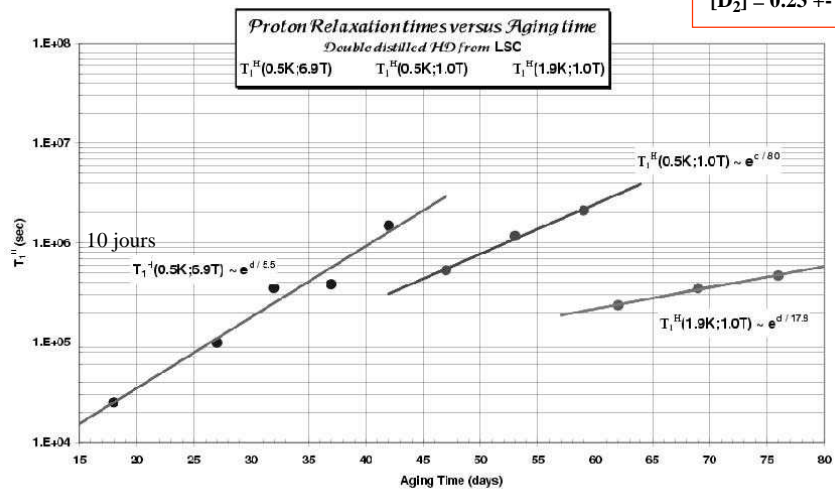
- ❖ More than **1 month of aging saved** for static polarization
- ❖ **Promising results** on relaxation time for Dynamic polarization.
- ❖ Test of new configuration of the column packings gives **+20%** efficiency
- ❖ Systematic measurements of relaxation time vs. initial concentration of H₂ and D₂ ready to start next week.

Pure HD Target HYDILE

relaxation time

$$[H_2] = 0.26 \pm 0.02$$

$$[D_2] = 0.23 \pm 0.02$$



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?

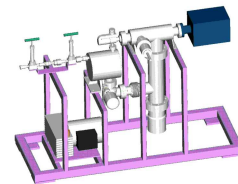
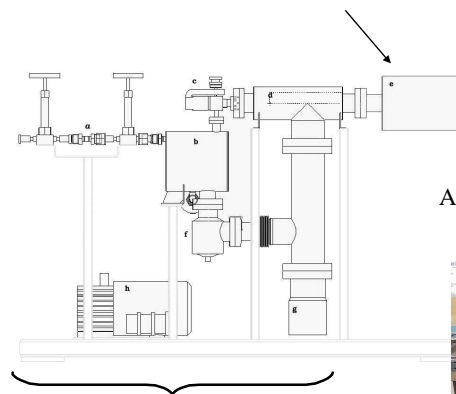
Static polarization.

Dynamic Nuclear Polarisation

What quality of HD do we need?

Concentration Measurements

MKS Microvision Plus Quadrupole Mass Spectrometer



Analyse Mass from 1 to 6

Gaz Input Manifold

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

Measure [D₂] down to 10^{-5}

