The Development of the Dilution Insert

and

some Tunneling Experiments

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Development of the dilution insert:

- at the ILL,
- in Garching (WMI, FRM II)

Tunneling experiments with the dilution insert

- CH_4
- H trapped by interstitials (O, N and C) in Nb

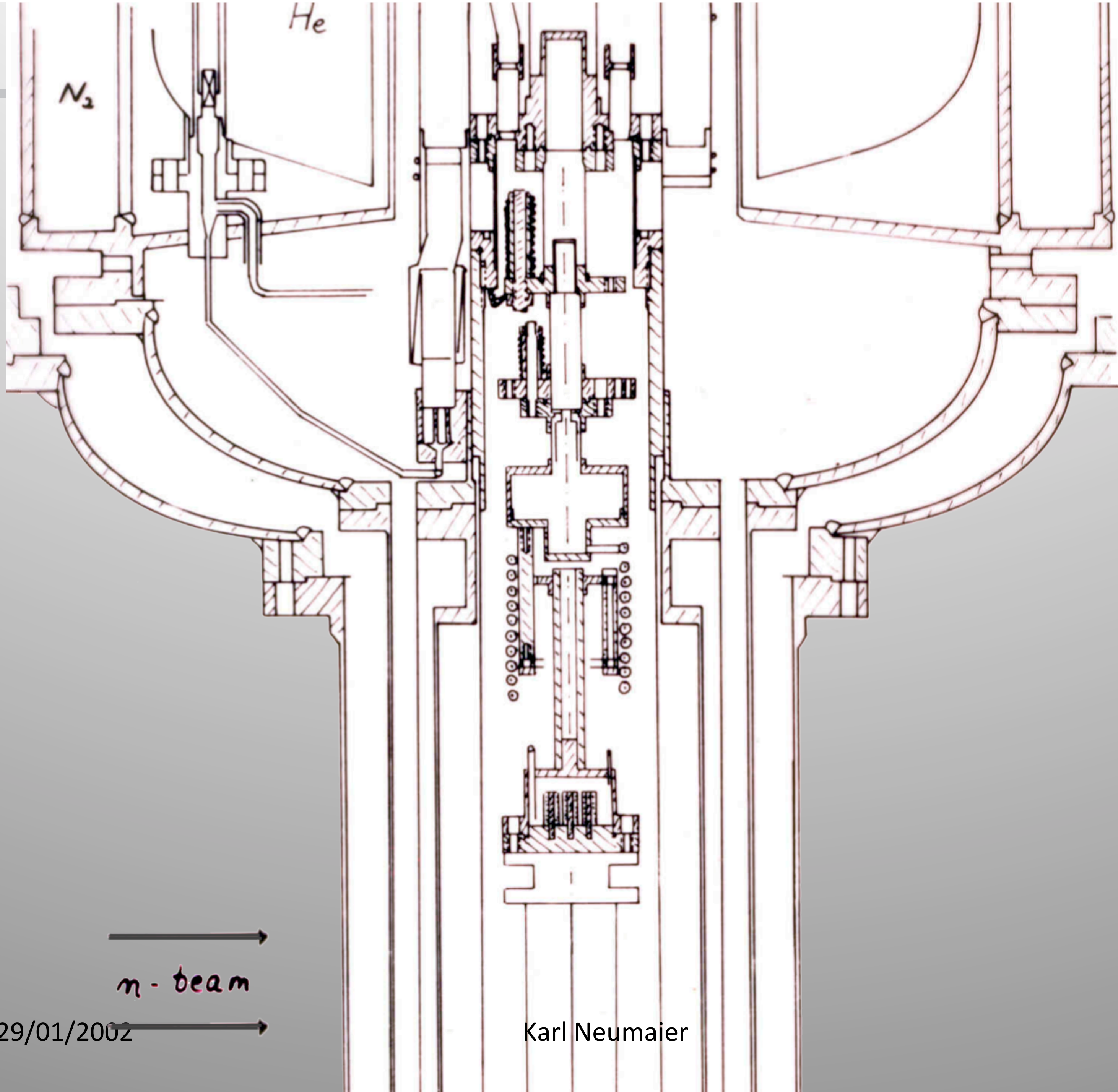
Starting point for the development of the dilution insert:

Experiment at IN13 (1981) with old Oxford dilution fridge (Lu: H-Tunneling): Preparation time: 1 week

Idea: replacement of the sample stick of an orange cryostat by a dilution insert

Advantage : no extra cryostat necessary

Uncertainty: cooling power and thermal contact at 1.5K sufficient to condense He-mixture?


He
N₂
n - beam

Bottom of a dilution insert

Flow rate: 30 µmol/s
Cooling power at 0.1 K:
24 µW
T_{min} = 19 mK
Time from RT to 30 mK
~ 6 h
Max. sample size:
Ø = 38 mm, l = 50 mm
Filling lines for gaseous
samples and He
Vacuum can:
upper part: Cu
lower part: Al

<- Silicon rubber seal
Thread for vac. can

<- Flow impedance
$6*10^{11}$ cm^{-3}

<- Still

<- Concentric tube
heat exchanger

<- Cu mixing chamber
Surface ~ $2m^2$

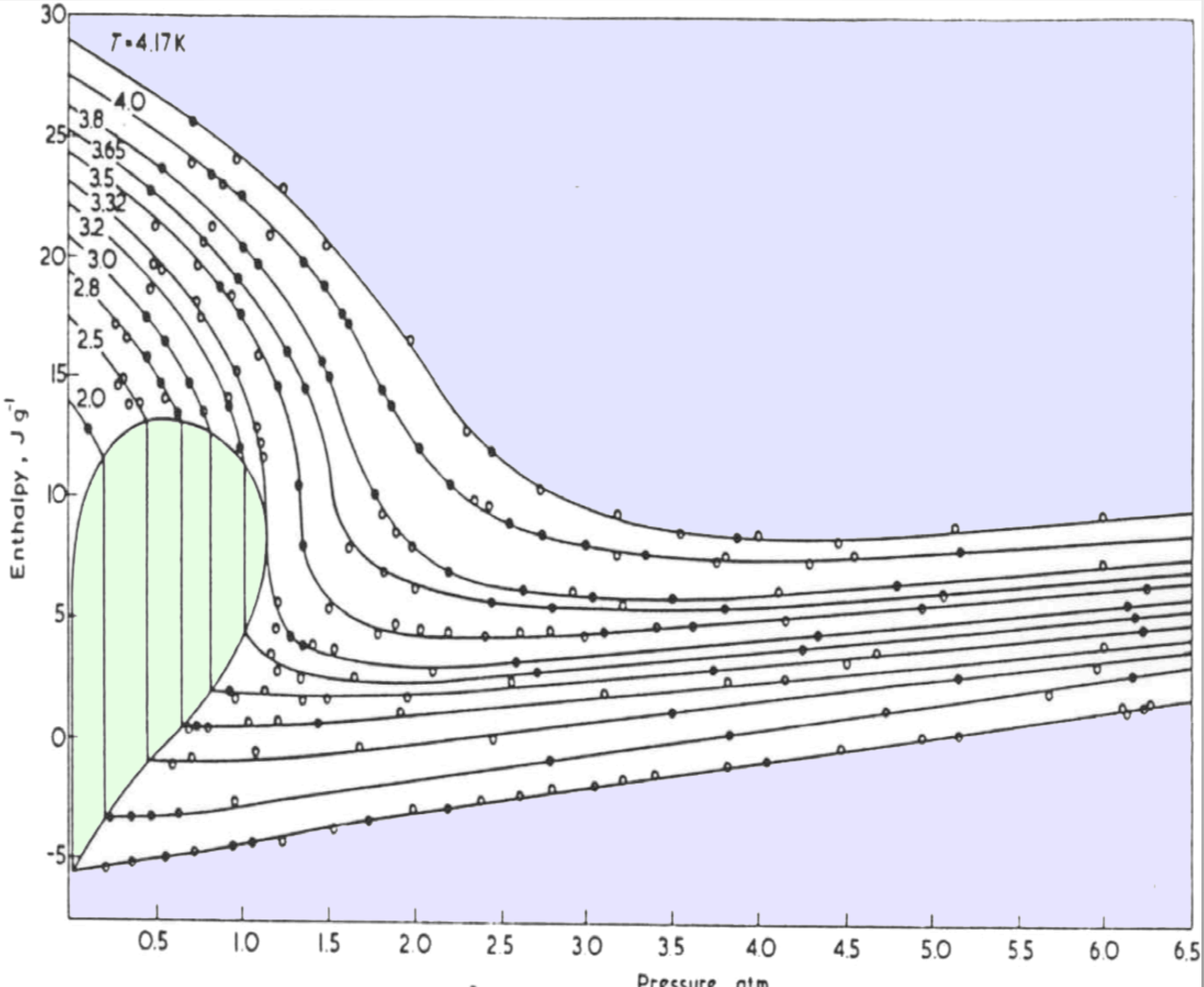
Logbook of the dilution insert

29/7/82	leak, very large
11/9/82	T =0.5 K, leak, large
10/11/82	T = 0.09 K, no leak detectable
14/11/82	T = 0.06K
18/11/82	CH_4 at 40mK
Jan. 83	CH_4 on IN5, T = 100mK
End of Jan. 83	NbO_xH_y on IN12
Sept.83	Mn acetate on IN13
Dec .83	NbO_xH_y on IN6
..	
2002	workhorse for mK-experiments

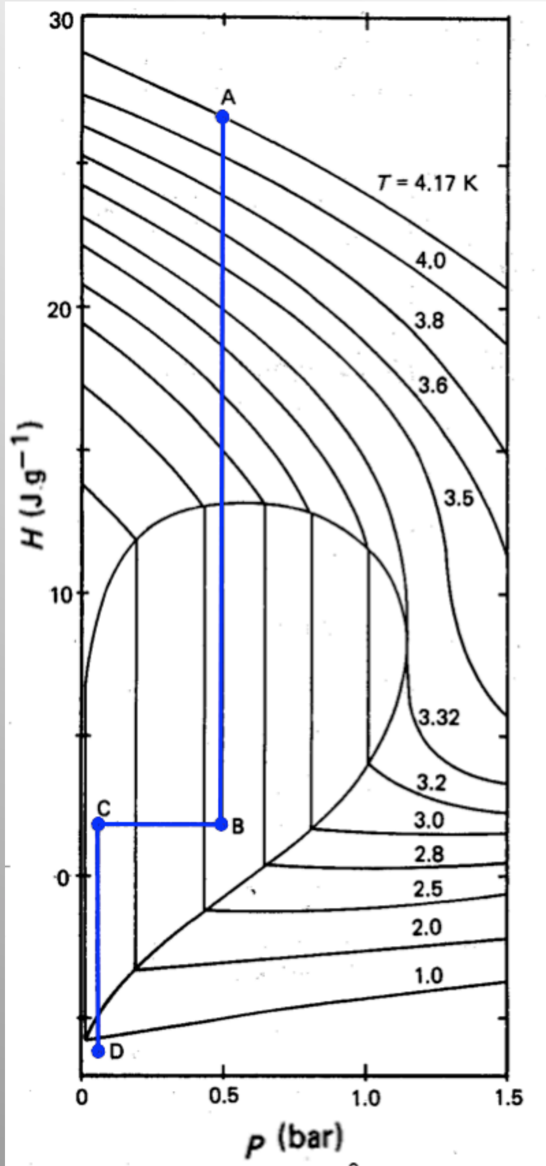
Dilution inserts at the Walther-Meissner-Institute

Need: facility for experiments at mK-temperatures in a high
field superconducting magnet (dewar with liquid He):
modified dilution insert

choice between: 1.) suppl. 1 K pot with extra He-pump
or 2.) internal condensation stage
(Kraus, deWaele 1977)
outgoing He-mixture (p~0) precools
incoming He-mixture (p~0.5bar)
in a heat exchanger above the still



Enthalpy–pressure (H–p) diagram of He^3



A-B: precooling of ^3He

B: liquid-gas mixture

B-C: expansion in prim. flow imped.

from 0.5 to 0.05 bar

C-D: liquefication in the still heat

exchanger (T_{still} = 0.75 K)

expansion from 0.05 – 0 bar

in the concent. tube exchanger

carefull choice of flow

impedances mandatory

Bottom of a dilution insert with internal condensation

P_{cond} = 0.5bar

B = 0 T T_{min} = 20 mK
B = 15 T T_{min} = 30 mK
with rot.pump (20 m³)

workhorse at WMI

<- 4.2K exchanger

<- silicon rubber seal
thread for vac. can

<- J-T exchanger

<- prim. impedance
$2*10^{13}cm^{-3}$

<- still

<- sec. Impedance
$10^{12}cm^{-3}$

<- concentric tube
exchanger

<- Ag-mixing chamber
surface ~5m²

Project: dilution insert for FRM II

FRM II: low temperature experiments

-> with pulse tube refrigerator (PTR)

no cyclically moving pistons

small heat load due to vibrations

large cooling power:

1. stage: 30 W at 60 K
2. stage: 0.5 W at 4 K

T_{min} ~ 2.4 K

-> cryoliquidfree orange cryostat

goal: dilution insert in combination with a dry split coil magnet

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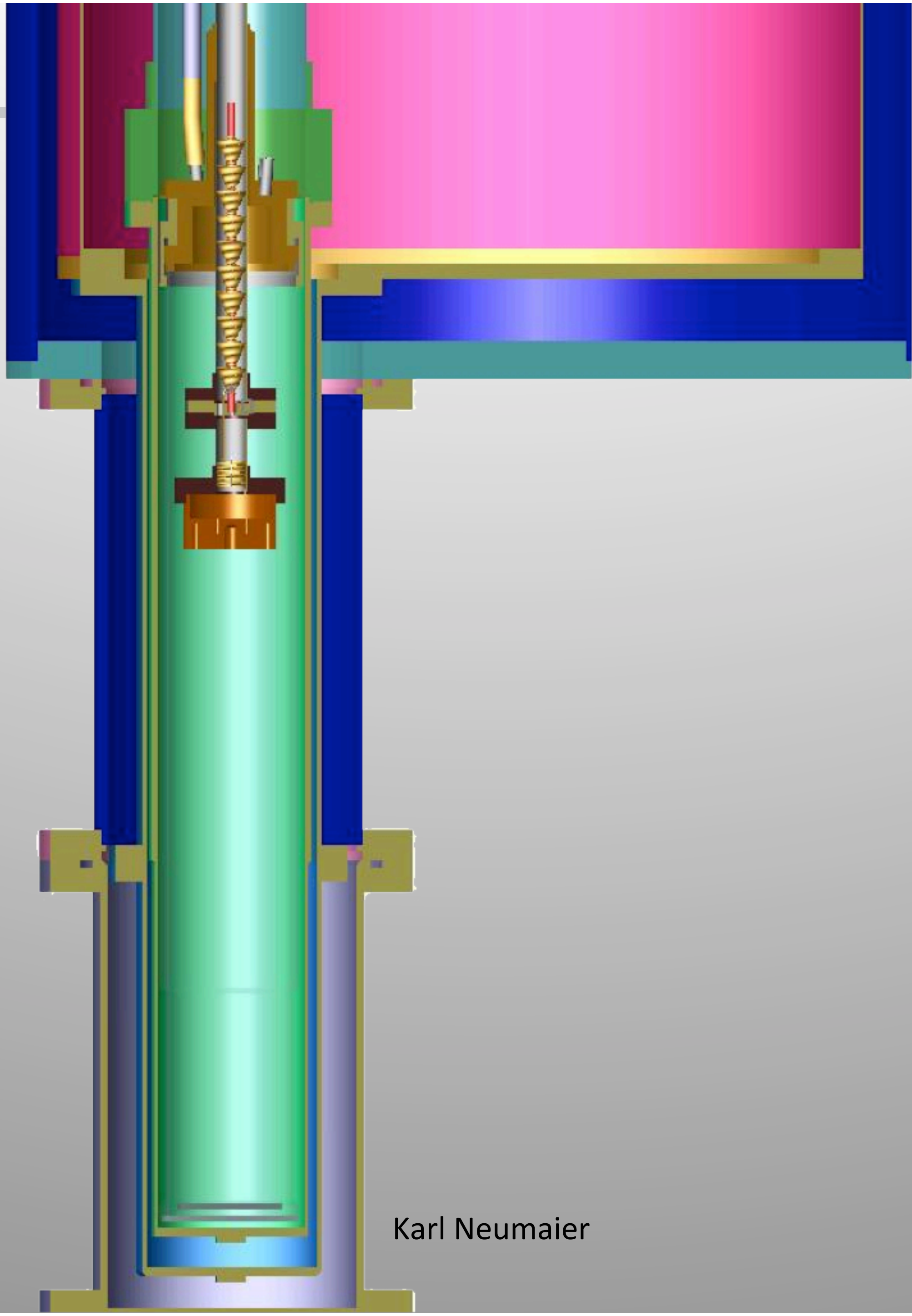
Pulse tube refrigerator with ^3He insert at the FRM II

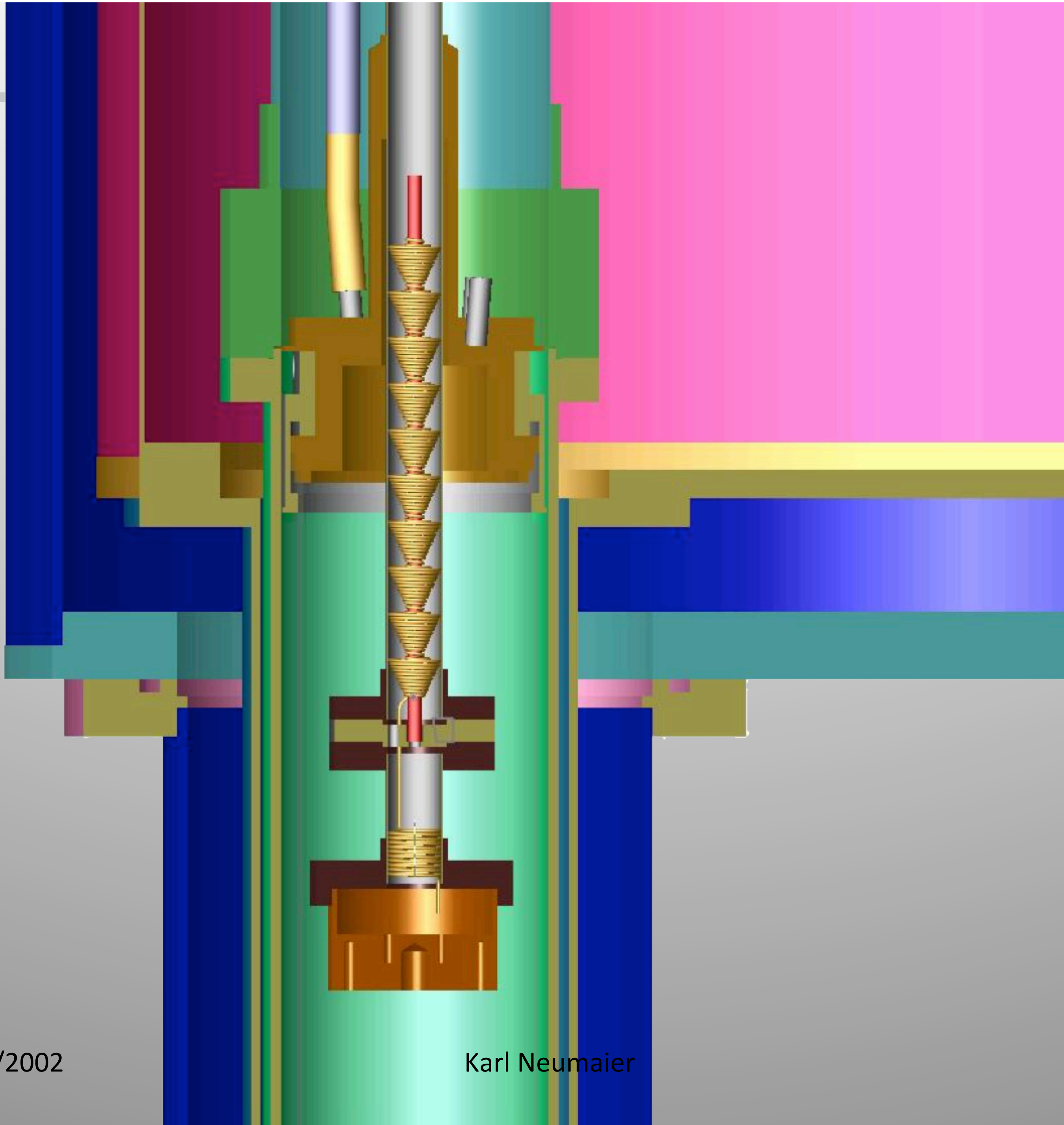
24 Nov. 2002

T_{min}= 0.45 K

Flow rate 100 µmol/s

J.Peters, H.Kolb





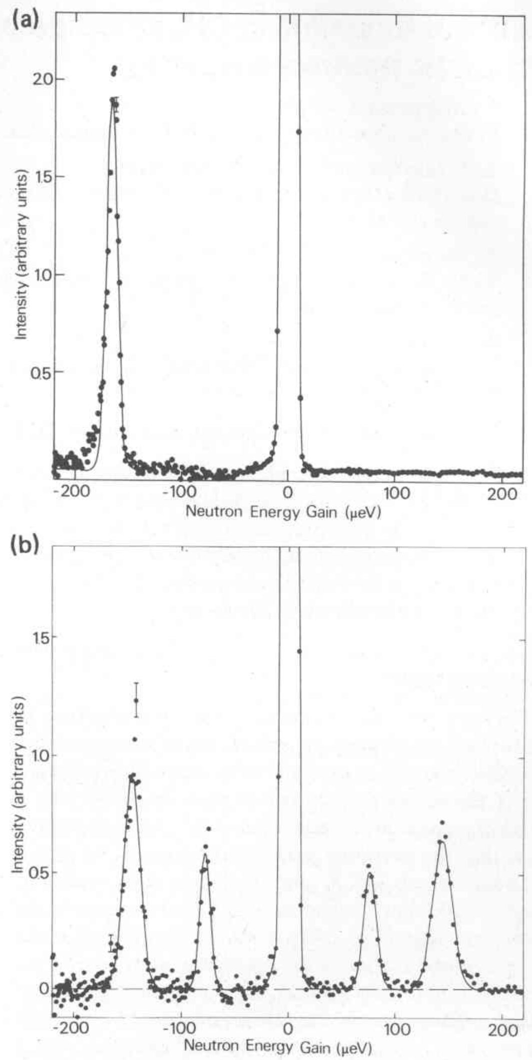


FIG. 3. Energy spectra of neutrons scattered in CH_4 with the spin system in thermal equilibrium; wavelength 13 Å; spin temperatures: (a) $T_S = <0.2$ K with a concentration of the A molecules greater than 99.8%; (b) $T_S = 5$ K.

with T -> 0
tunneling frequencies increase
inelastic lines narrow

for T_{min}
intrinsic linewidth finite
asymmetric line shape of
inelastic peaks

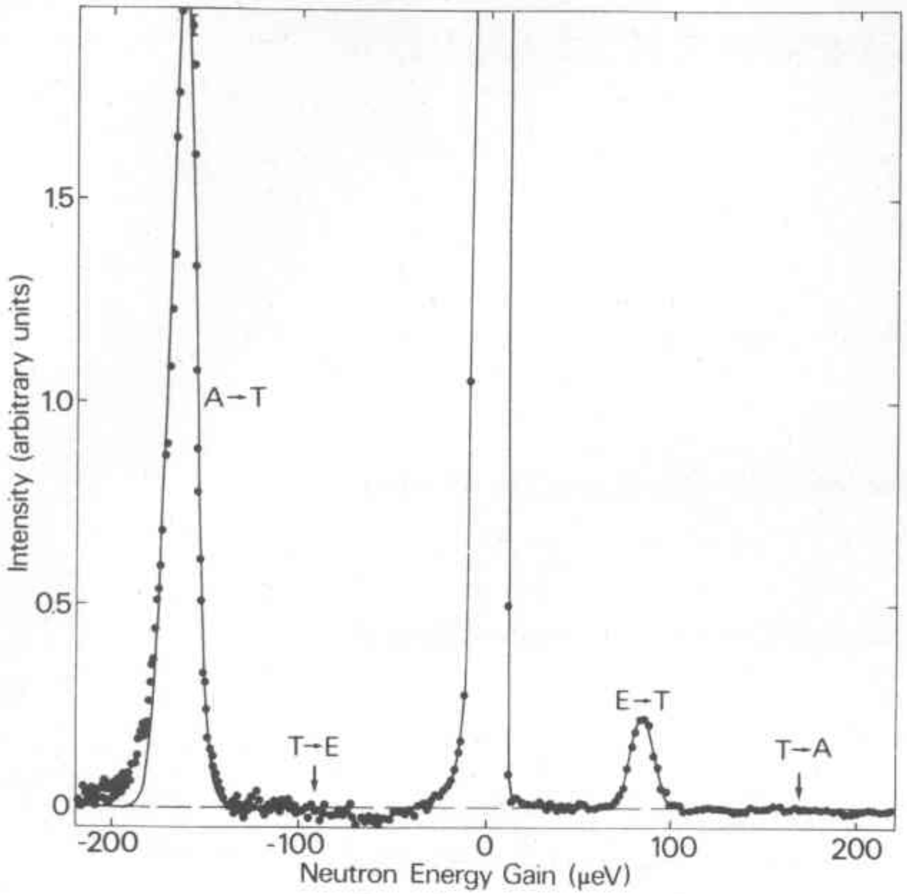


FIG. 1. Energy spectrum of neutrons scattered in "quenched" CH_4; wavelength 13 Å; temperature of the sample cell 0.25 K; concentration of the spin species: $c_A = 96.5\%$; $c_T < 0.1\%$; $c_E = 3.5\%$.

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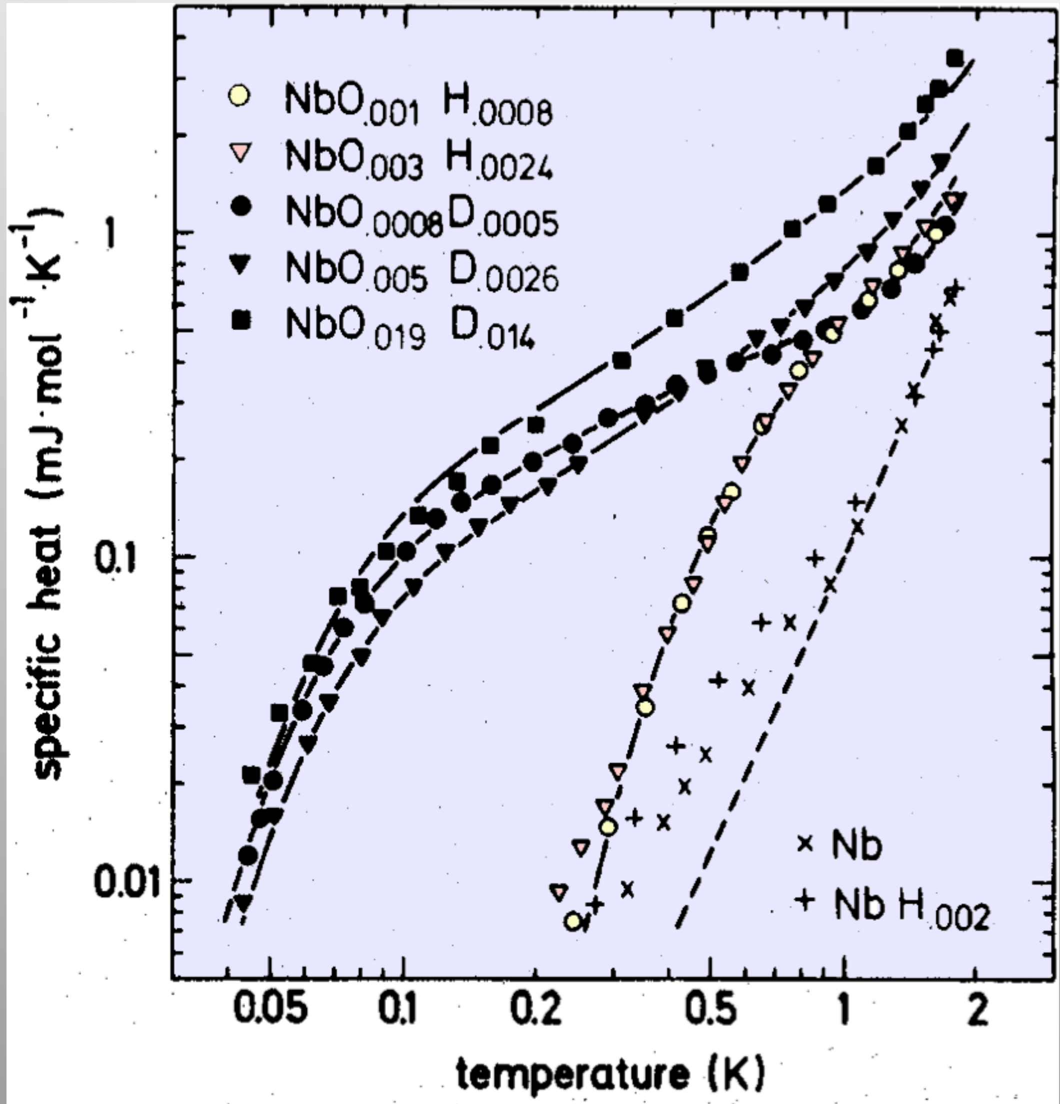

Tunneling of H interstitials in Nb trapped by impurity atoms (O,N,C)

Tunneling energies:

J(OH) ~ 230 μeV

J(NH) ~ 170 μeV

J(CH) ~ 160 μeV

J(OD) ~ 20 μeV

J(ND) ~ 14 μeV

J(CD) ~ 12μeV

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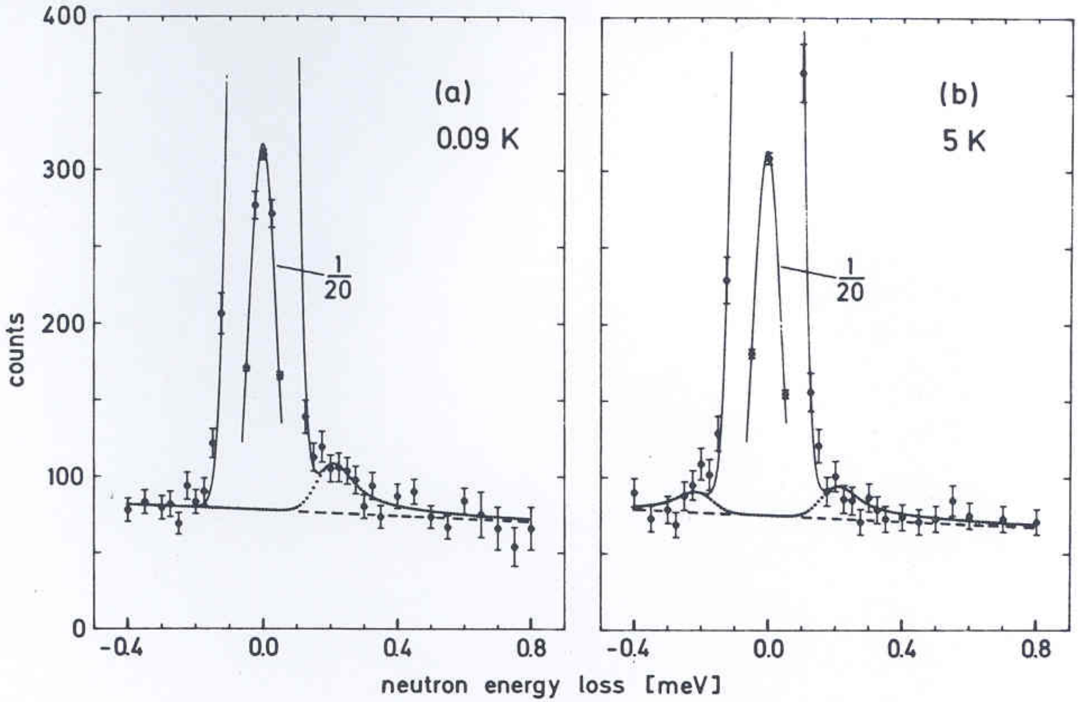

FIG. 1. Inelastic neutron spectra of $NbO_{0.013}H_{0.016}$ at (a) 0.09 K and (b) 5 K. The counting time is 84 min (the counts and standard deviations for data points measured with different counting times are appropriately corrected). The full, dotted, and broken lines indicate fits explained in the text.

c = 0.015

J(OH)~ 190μeV

Wipf, Magerl, Shapiro, Satija and Thomlinson

IN12

T = 0.1K

c = 0.002

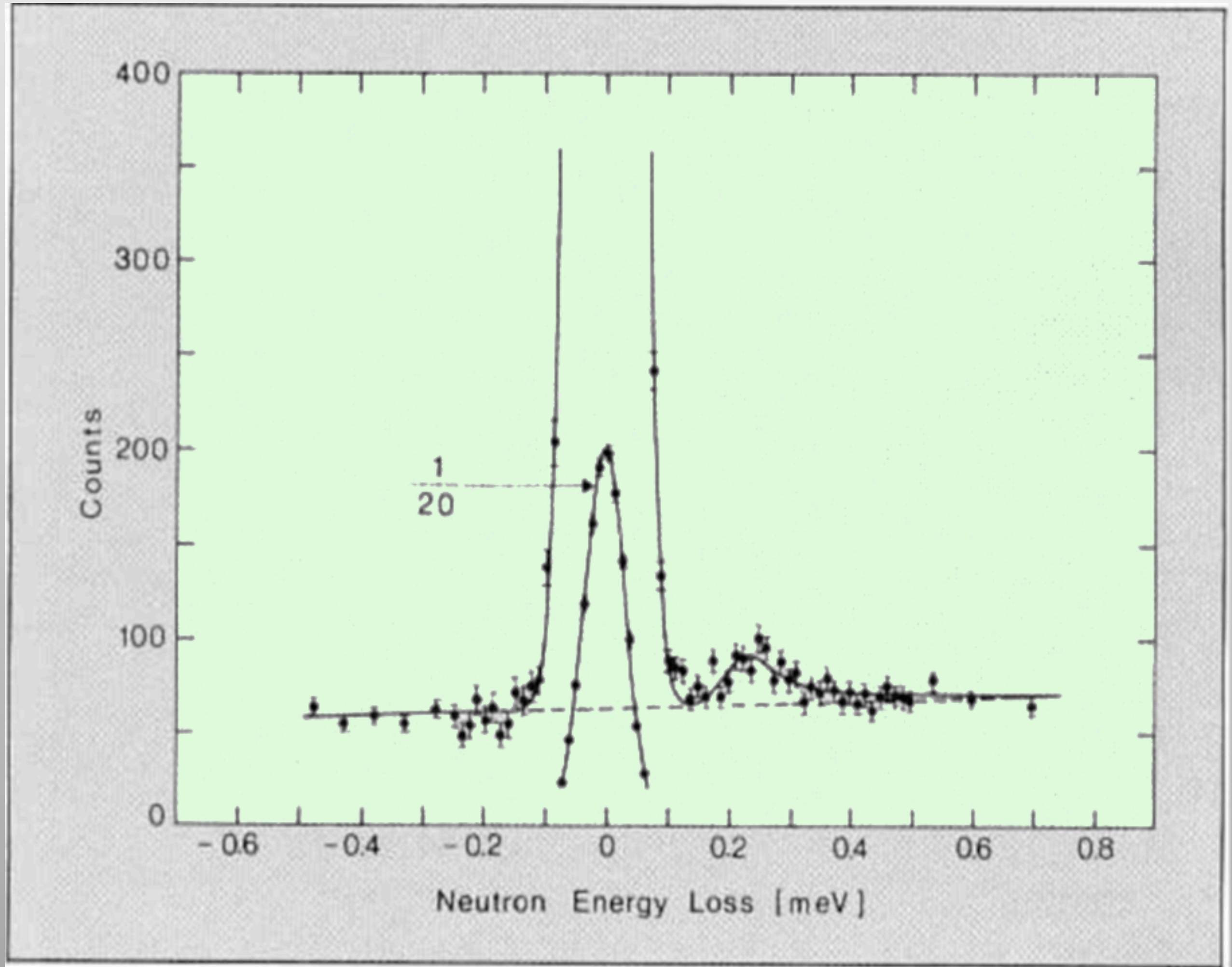

Figure 34: Neutron spectra of NbO_xH_x ($x = 0.002$) measured at 0.1 K. The energy resolution of the instruments is 0.07 meV.

ILL Ann. Rep. 1983

Wipf, Neumaier, Magerl, Heidemann and Stirling

T = 1.3 K

c = 0.01

c = 0.001

c = 0.00015

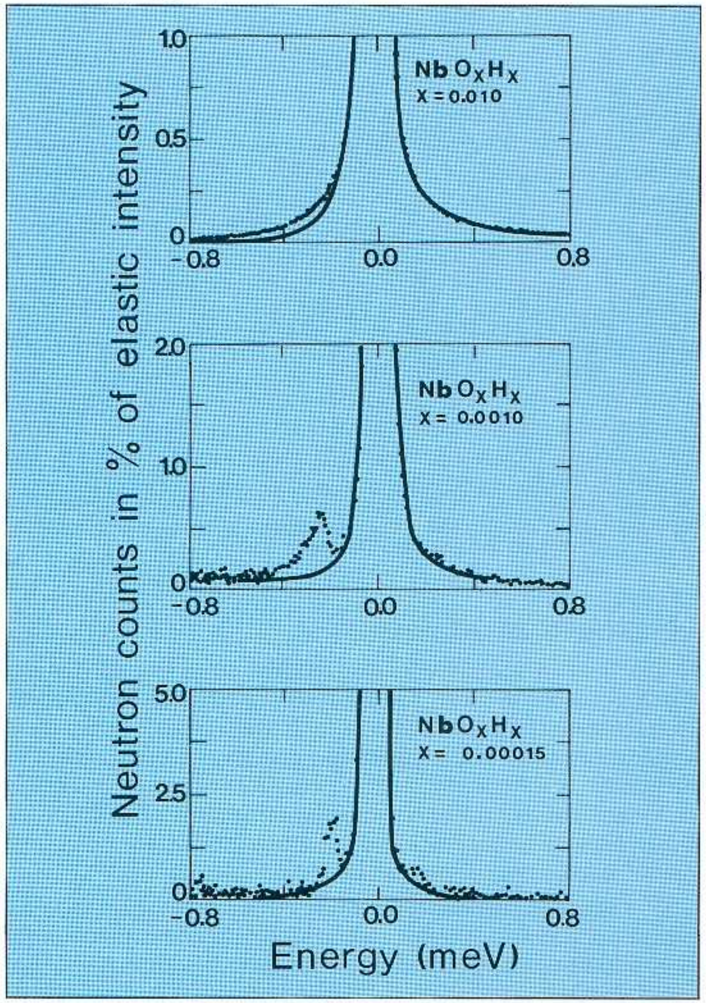

Figure 38: Inelastic scattering from the tunnel-split ground state for H in NbO_xH_x at various concentrations x. The measurements were performed at 1.3 K. The solid line gives the instrumental resolution.

IN6

ILL Ann. Rep. 1984

Magerl, Dianoux, Wipf, Neumaier and Anderson

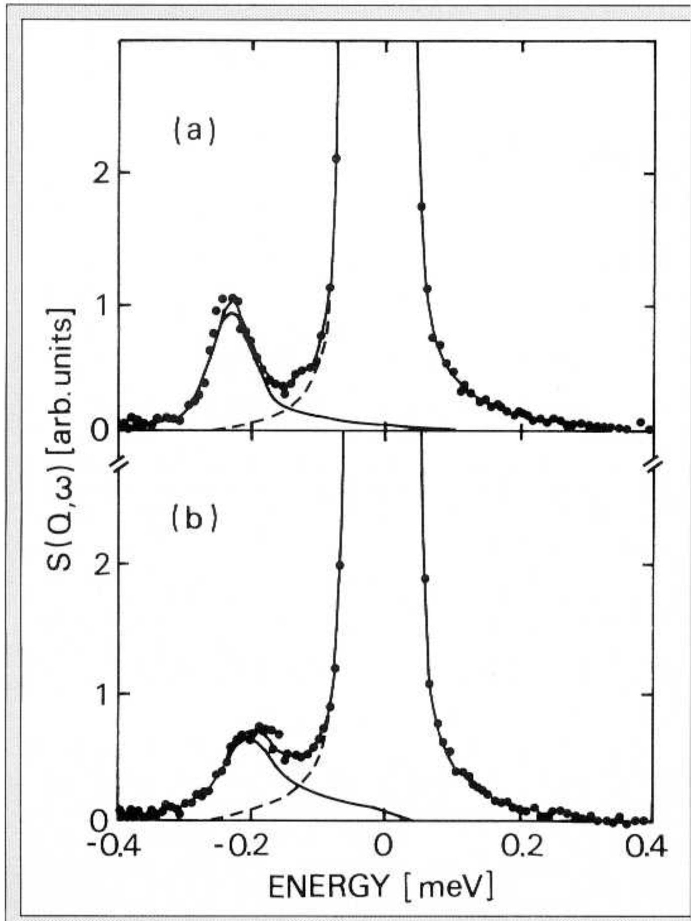

Figure 71: Neutron spectra measured at 0.1 K in the superconducting (part a) and in the normal conducting state (part b). The dashed line gives the elastic energy resolution of the spectrometer and the thin solid line shows the fitted inelastic scattering. The thick solid line is the sum of both scattering contributions. The data were taken with the time focusing time of flight spectrometer IN6 at the ILL.

Oxford Dilution Refrigerator
with dry split coil magnet
(built by H. Wipf)

c = 0.0002

0.0 T: J(OH) ~ 226 μeV
0.7 T: J(OH) ~ 209 μeV

ILL Ann.Rep 1986

Wipf, Steinbinder, Neumaier, Gutsmiedl, Magerl and Dianoux

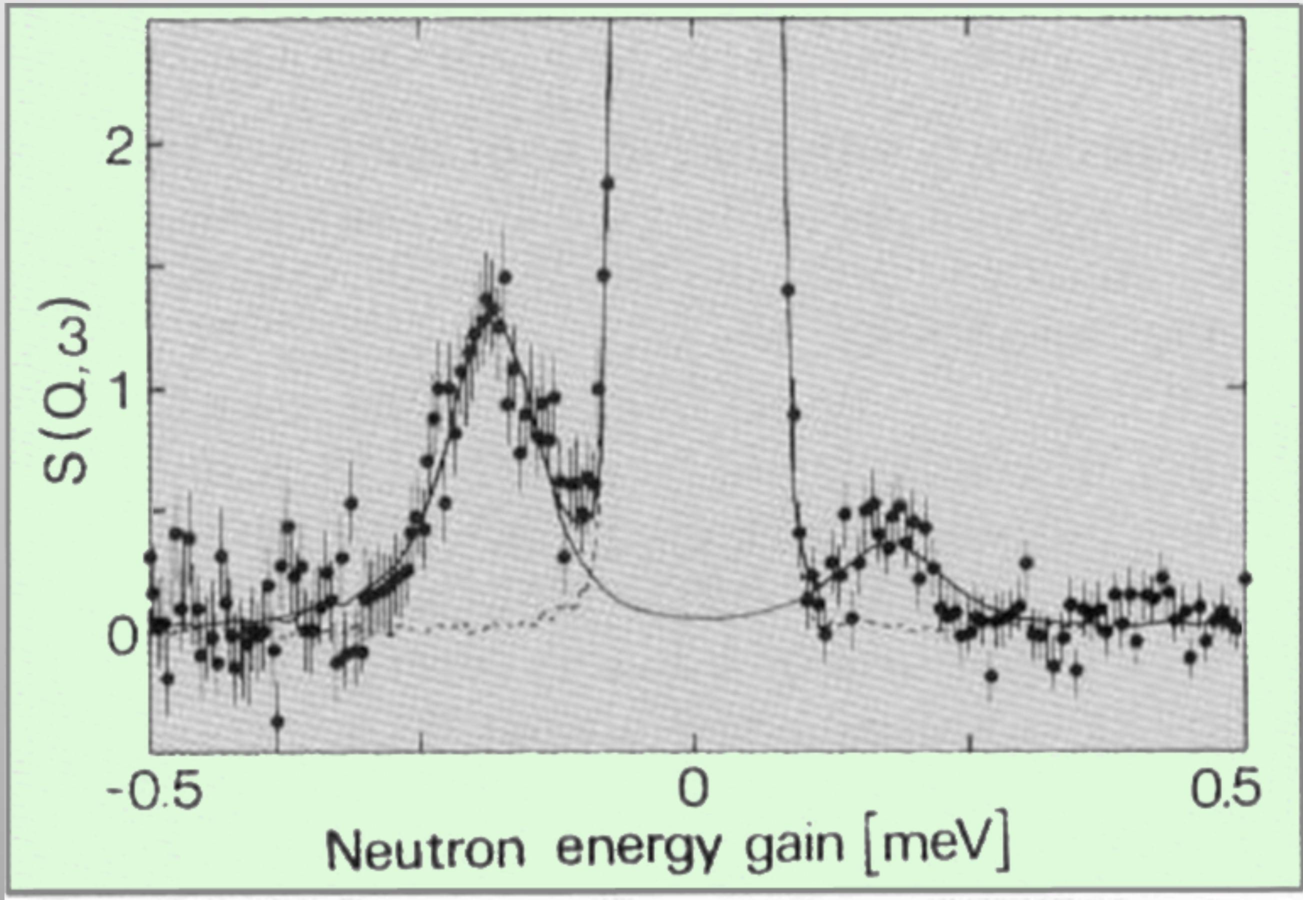

Figure 64 Hydrogen tunneling spectrum of $Nb(CH)_x$, where $x = 0.0002$ measured on IN5 at $T = 1.8$ K.

IN5

T = 1.8 K

c = 0.0002

J(CH) ~162 µeV

ILL Ann. Rep. 1988

Steinbinder, Wipf, Neumaier, Blank and Kearly

Walther Meissner Institute

Congratulation to the ILL cryogenic team

especially J.L. Ragazzoni for 20 years of efficient and reliable operation of the dilution inserts

My best thanks to Toni for our successfull

and friendly collaboration.

Without godfather Toni the dilution insert would not exist.