A Solid Polarized Target Development Facility at Jefferson Lab

James Brock

Presented on the behalf of
The Jefferson Lab Polarized Target Group

U.S. DEPARTMENT OF ENERGY

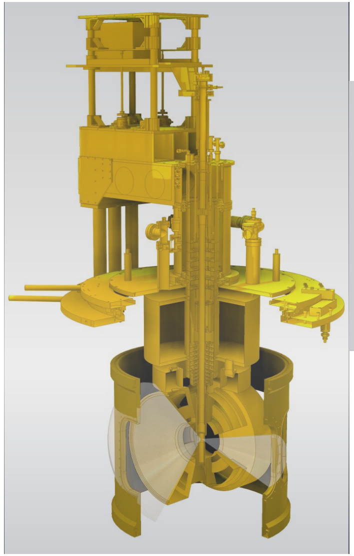
24st of September, 2024

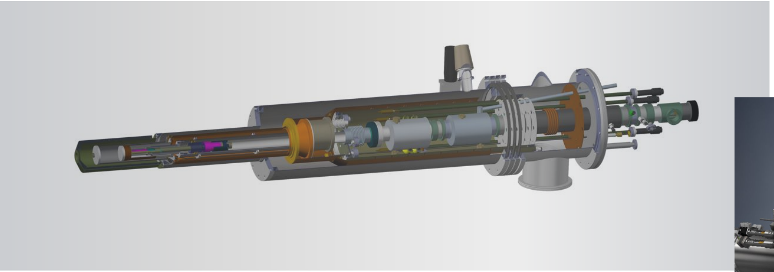
Jefferson Lab
Thomas Jefferson National Accelerator Facility

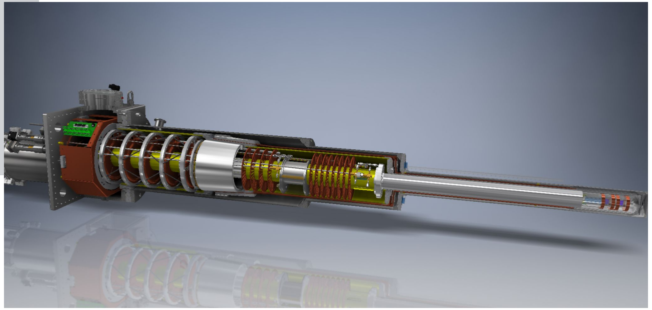
A Solid Polarized Target Development Facility at Jefferson Lab

JLab @ 6 GeV Era (1998 - 2012) 12 polarized solid targets were used in experiments

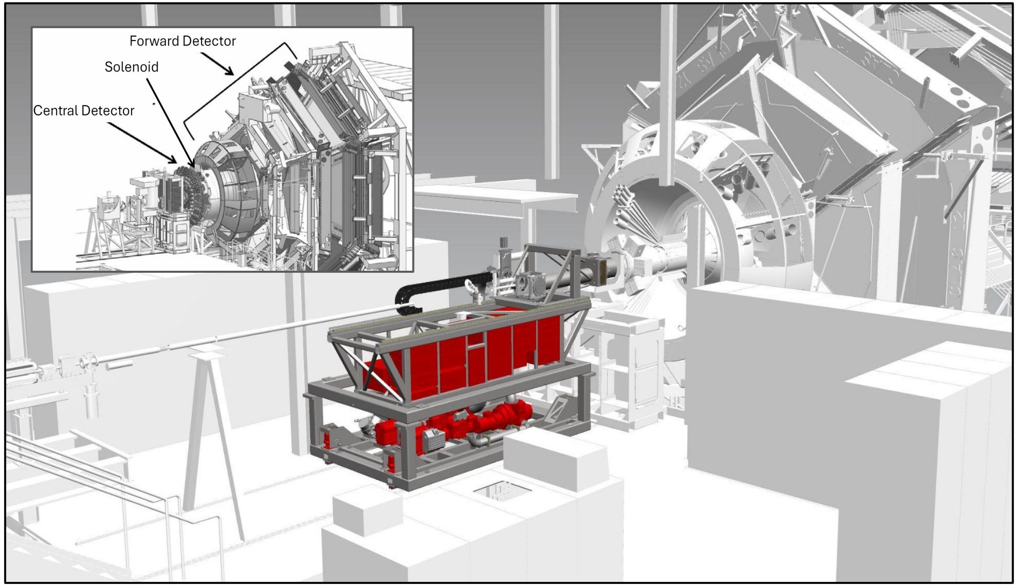
JLab @ 12 GeV Era (2016 - _____) >12 experiments have been approved to use Polarized Targets





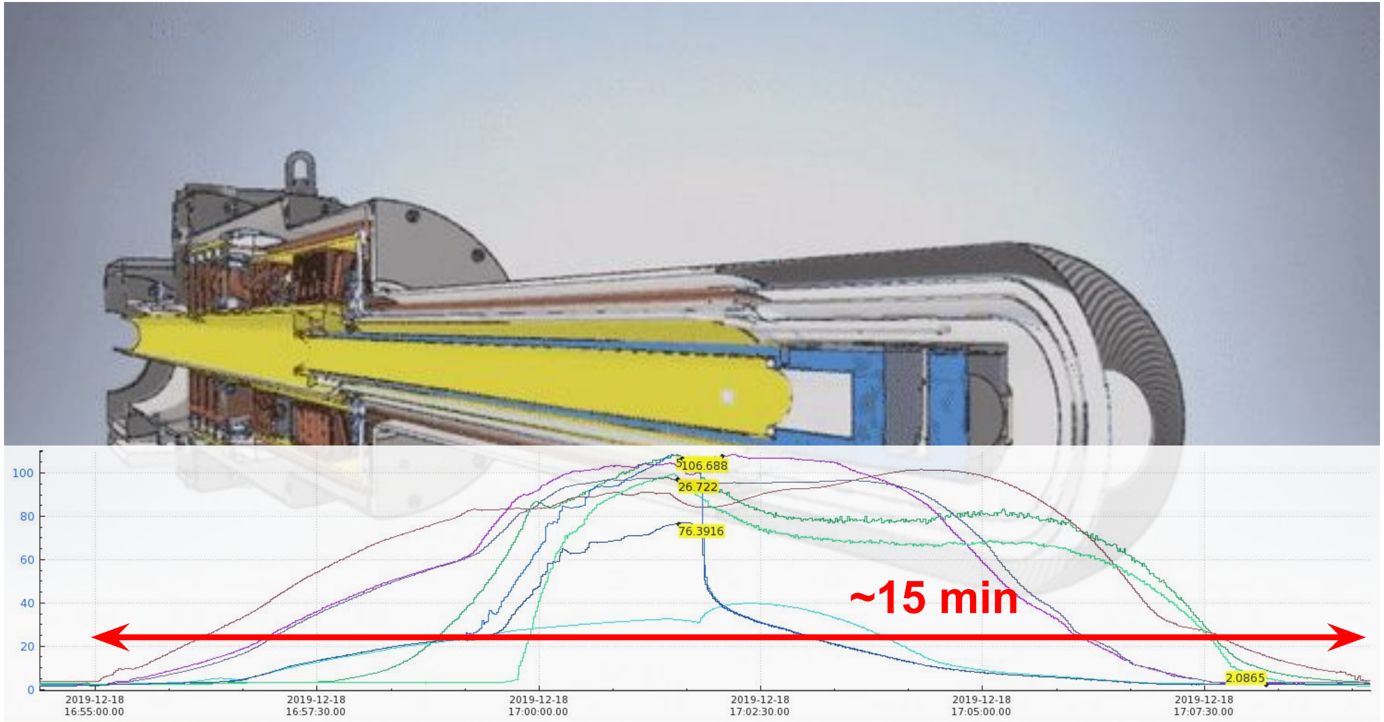


The 1st solid polarized target in the 12 GeV Era ran from July 2022 - March 2023 in Hall B for Run Group C



Refrigerator base temperature, 0.9 K, cooling power 1 W @ 1.08 K, liquid helium consumption, 25 L/day.

The actual time for swapping the Target Cell is **~15 min**. Additional time is need for other tasks associated with Target changes account for the remainder of the 1.5 hr avg estimated time Controlled Access



https://youtu.be/22PTbdC2sjw

The Target Cell has been changed **75 time**, alternating in no particular order between; NH_3, ND_3, CH_2, CD_2, C, Empty and Optical Targets

With the traditional method of inserting the target in a horizontal refrigerator using a stick would require a factor of **2 × personnel** and **5 × down time**. (this is a conservative estimate!)

Avg. Target swap time ~1.5 hrs × 75 = 112.5 hrs vs. Est. Target swap time ~7.5 hrs × 75 = 562.5 hr

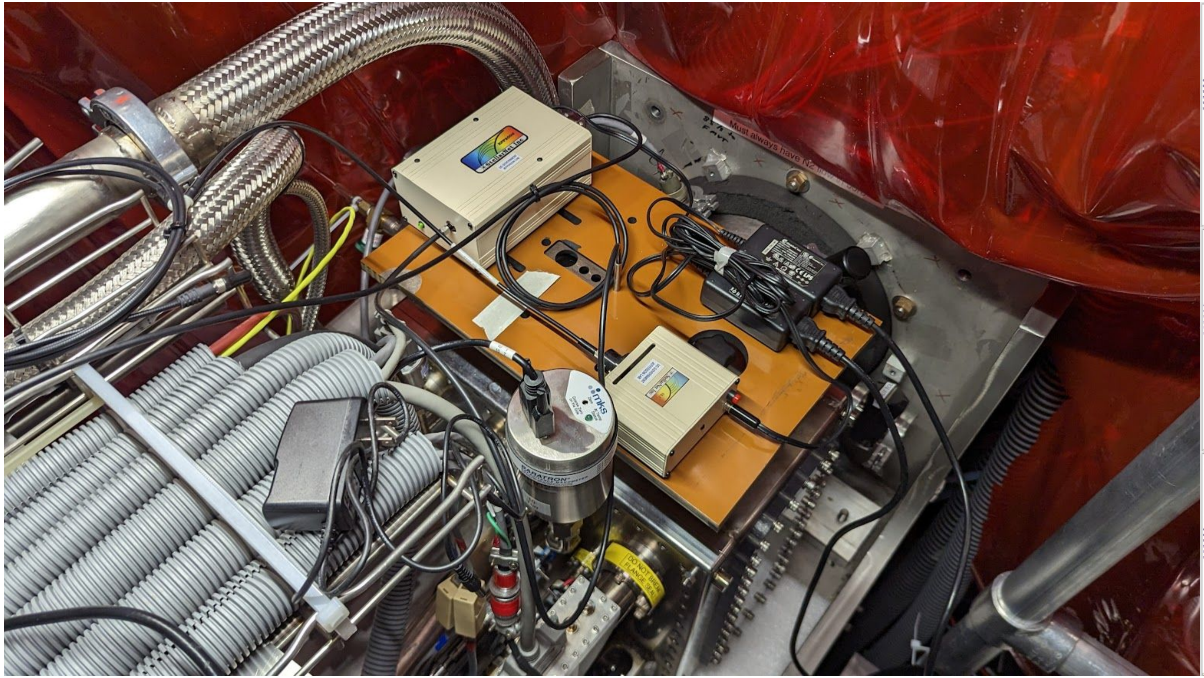
The Retractable 1 K Bath, Target Cell Cartridges, and installation/retrieval process has, over this experiment, **saved a minimum of ~ 18.75 days of downtime**

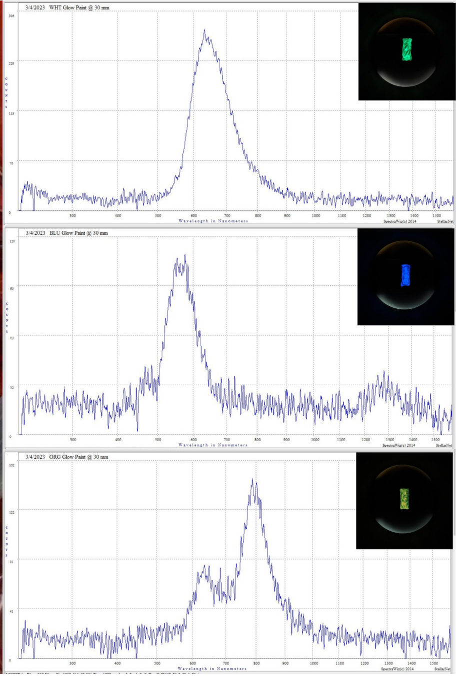
~10% of the allotted run days

https://youtu.be/22PTbdC2sjw

DNP Targets - Longitudinally Polarized Target for CLAS12

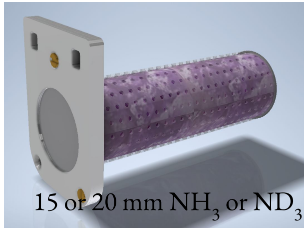
https://www.youtube.com/watch?v=x-8wJdM5egk

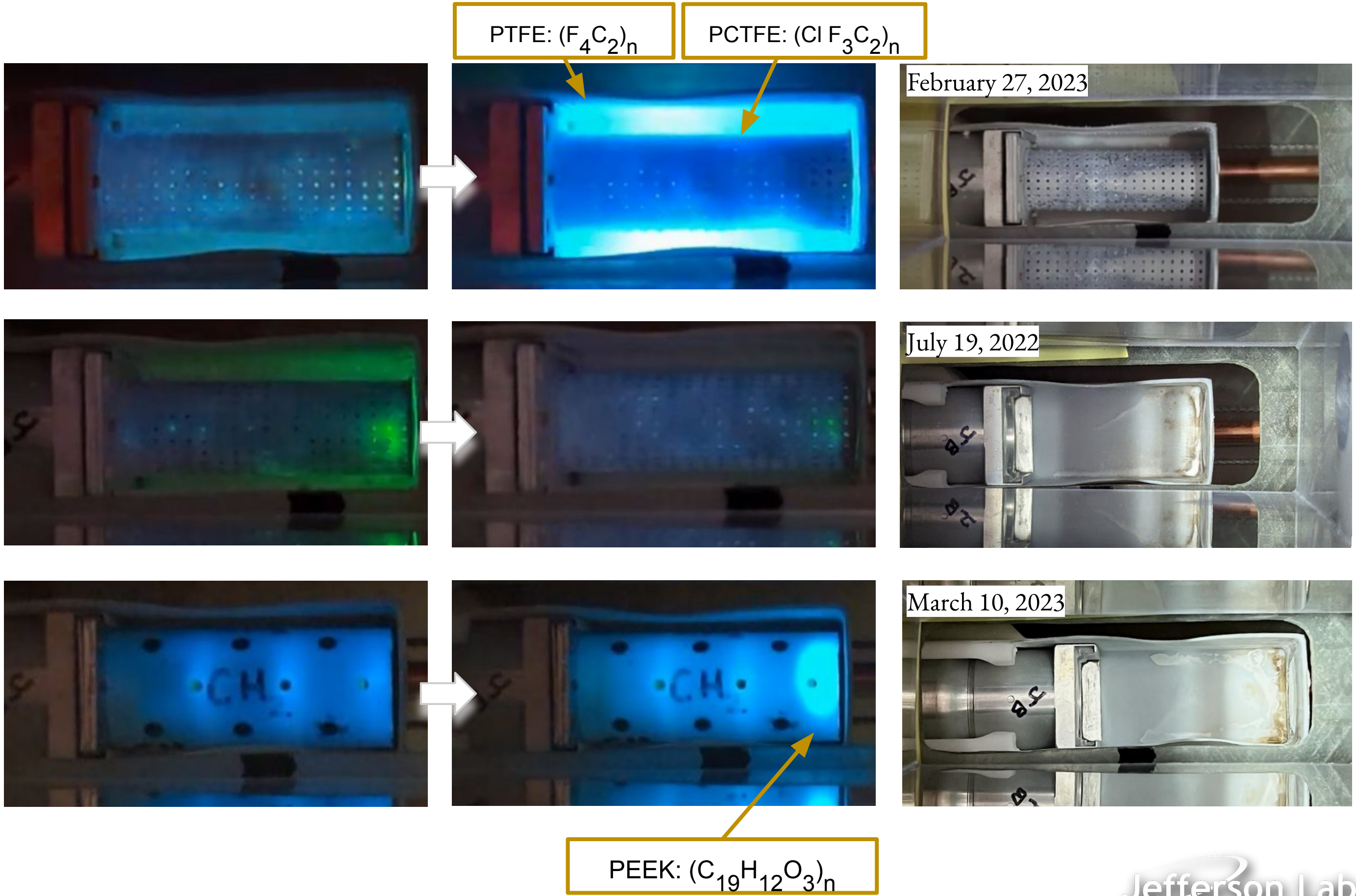


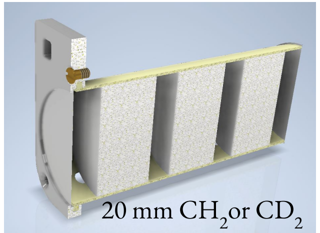


Attempt at Recording a Spectrum

DNP Targets - Longitudinally Polarized Target for CLAS12







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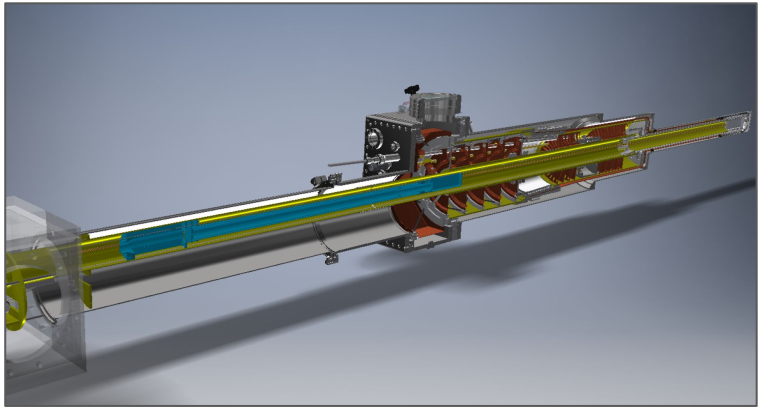

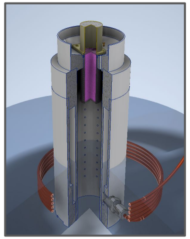

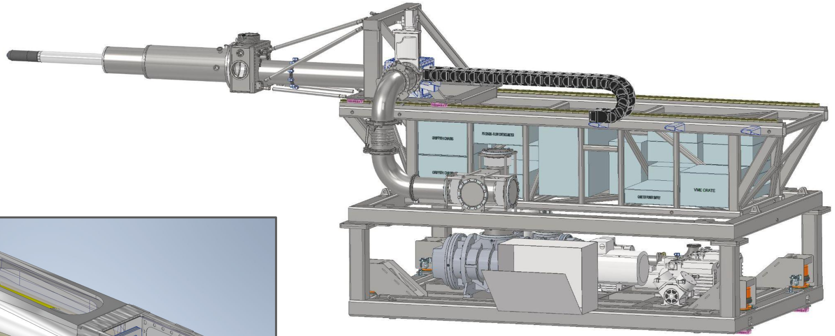

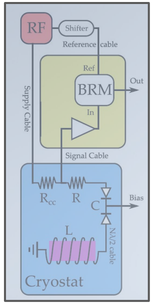

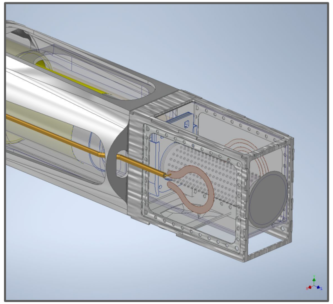

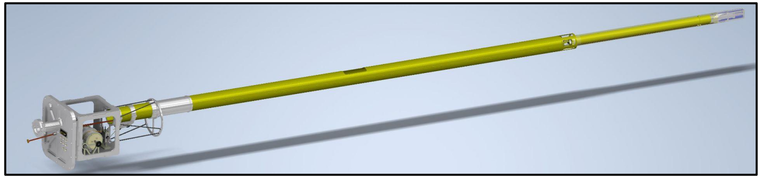

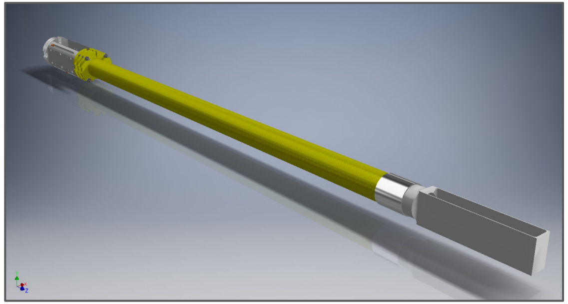

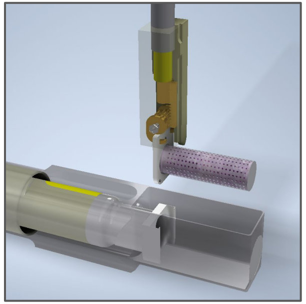

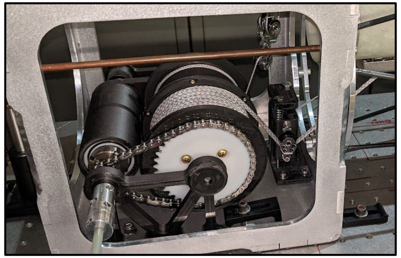

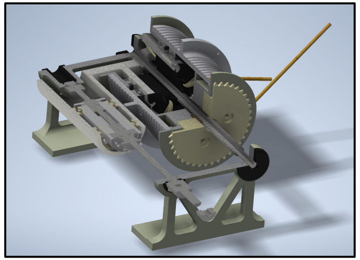

Table 1 Polarized target materials commonly used in particle scattering experiments

Materials and chemical composition	Dopant[a] and method	Polarizable nucleons % by weight	B/T Tesla/K	Polarization %	Radiation characteristic flux[b] 10^{14} particles/cm^2
LMN $La_2(Co, Mg)_3$ $(NO)_3 . 24H_2O$	Neodymium Ch	3.1	2.0/1.5	±70	~0.01
1,2 Propanediol $C_3H_6(OH)_2$	Cr(V) Ch	10.8	2.5/0.37	+98 −100	~1
1,2 Ethanediol $C_2H_4(OH)_2$	Cr(V) Ch	9.7	2.5/0.5	±80	~2
Butanol C_4H_9OH	EHBA Cr(V) Ch	13.5	2.5/0.3	±93	3–4
EABA $C_2NH_7BH_3NH_3$	EHBA Cr(V) Ch	16.5	2.5/0.5	+75 −73	7(+), 3.5(−)[c]
Ammonia $^{14}NH_3$, $^{15}NH_3$	NH_2• Ir	17.5, 16.6	5.0/1.0	+97 −100	70, 175[d]
d-Butanol C_4D_9OD	EDBA Ch	23.8	2.5/0.3	±50	Not measured
d-Ammonia $^{14}ND_3$, $^{15}ND_3$	ND_2• Ir	30.0, 28.6	3.5/0.3	+49 −53	130(+), 260(−)
Lithium deuteride ^6LiD	f-center Ir	50	6.5/0.2	±70	400

[a]Ch: chemically doped, Ir: doped through irradiation.
[b]The radiation dose which reduces the polarization by e^{-1} of its value.
[c]For positive and negative polarizations, respectively.
[d]In NH_3 there are two distinct regions of decay.

Irradiated Ammonia NH_3, ND_3

Tempo Doped Butanol $C_2H_4(OH)_2$

D.Crabb, W.Meyer / Annu. Rev. Nucl. Part. Sci. 1997. 47:67–109

DNP Targets - Materials - NH_3 & ND_3

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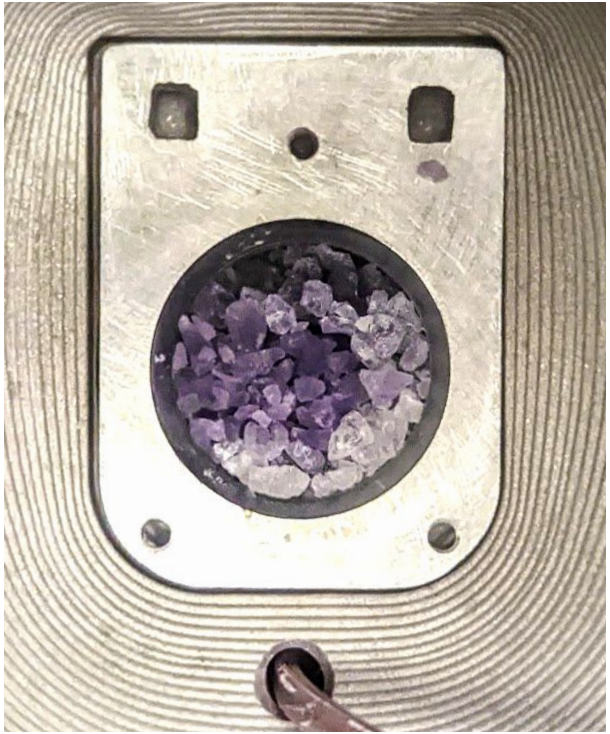
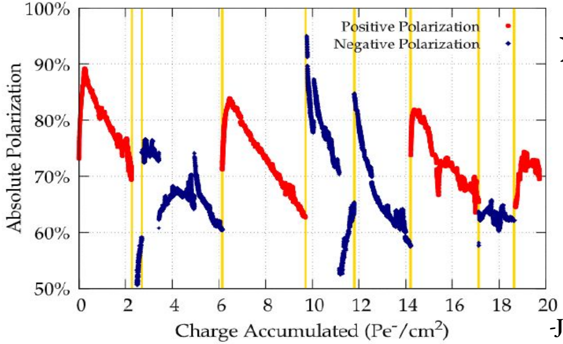


D.Crabb, W.Meyer / Annu. Rev. Nucl. Part. Sci. 1997. 47:67–109

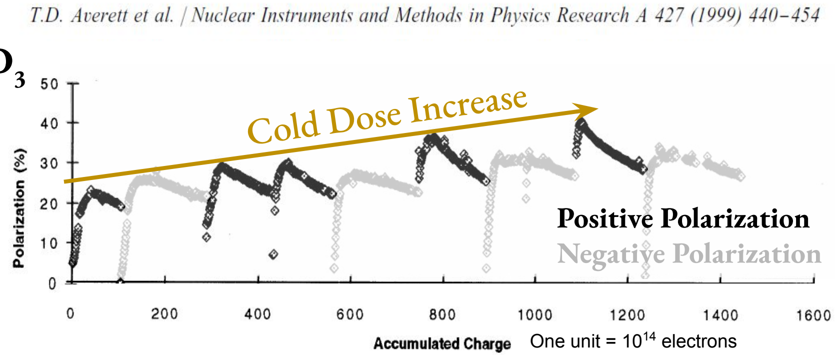
DNP Targets - Effects of Material Dosing

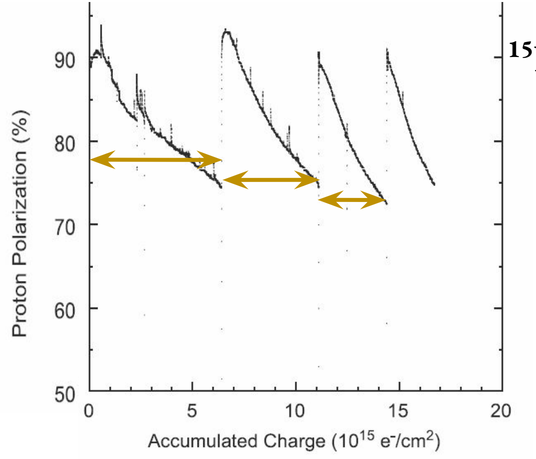


NH$_3$

-J.D. Maxwell

ND$_3$

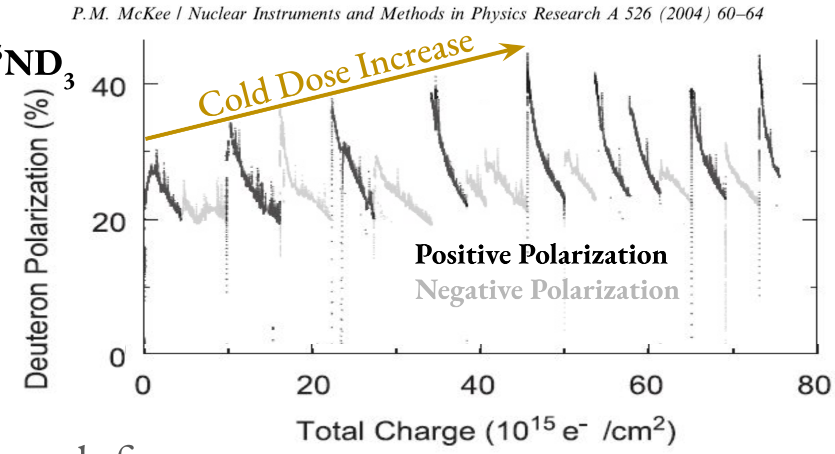




$^{15}NH_3$

-P.M. McKee

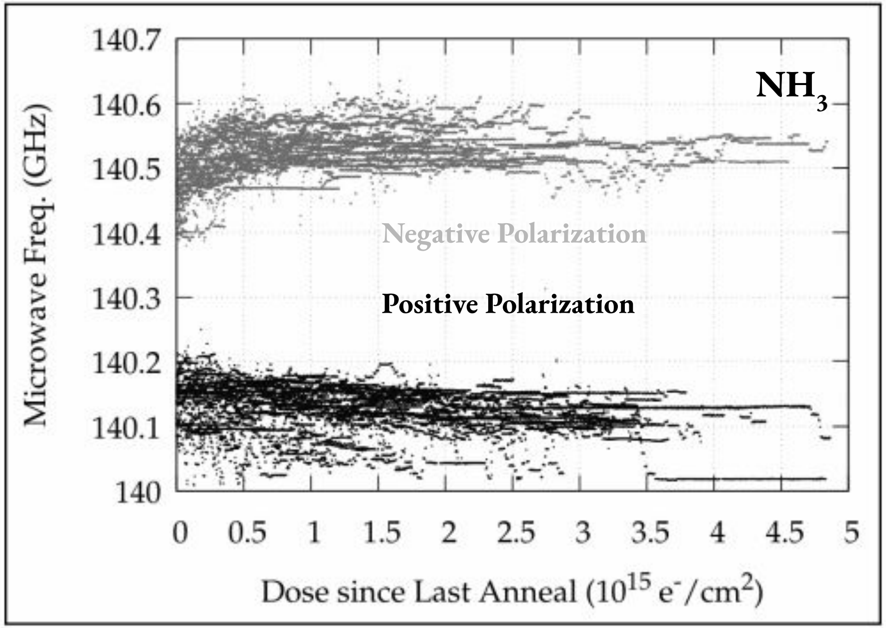
$^{15}ND_3$



Annealing trends for NH$_3$ and ND$_3$

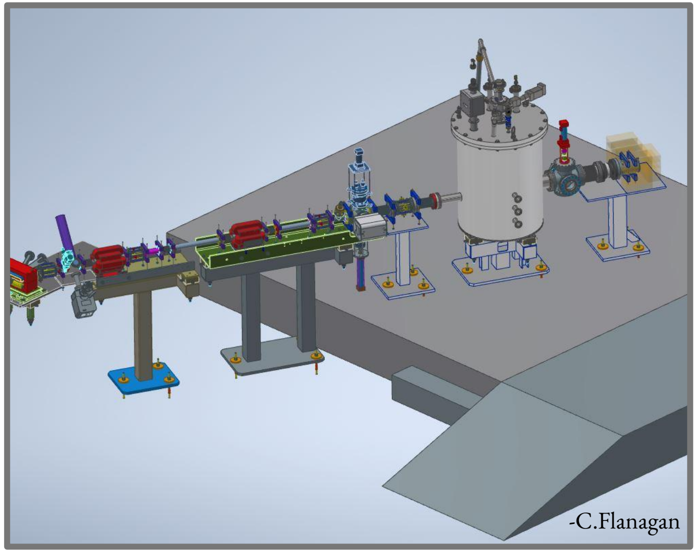
As the material accumulates dose the microwave frequency must be adjusted to achieve maximum polarization.

This evolution follows predictable trends and is well documented.



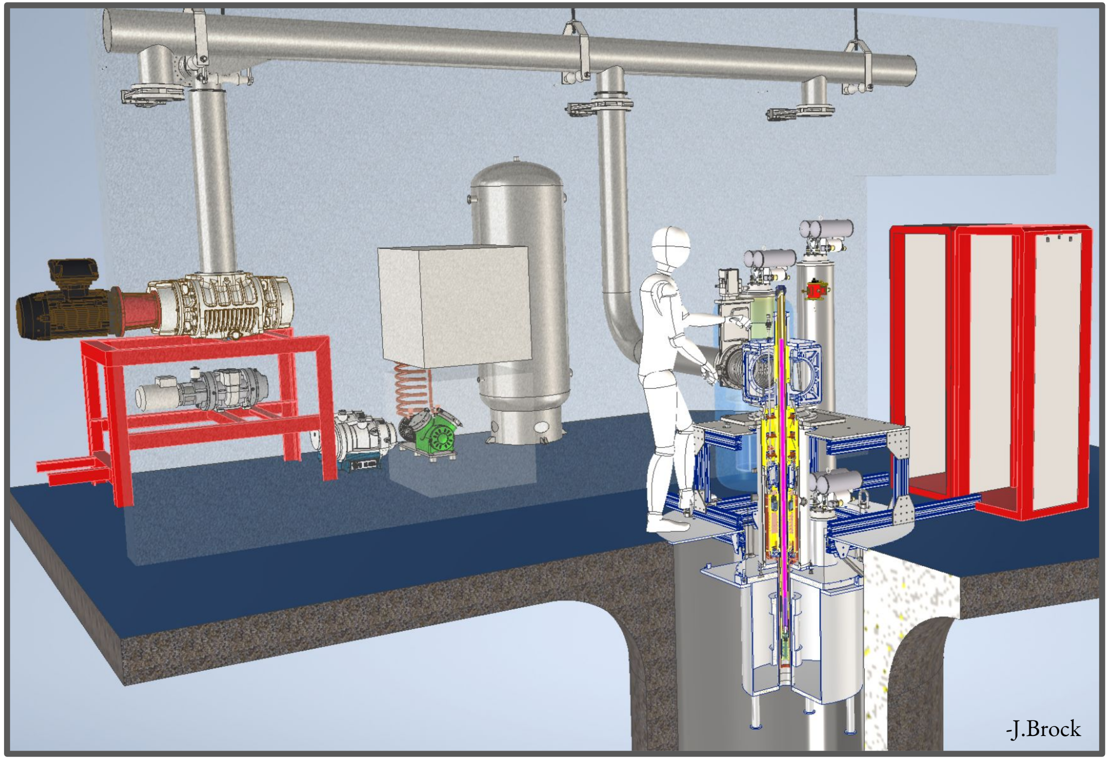
-J.D. Maxwell, Nucl. Instrum. Meth. A 885, 145–159 (2018)

In the "Middle" of the "Beginning"...



Irradiation Cryostat (JLab Injector)

Polarized Target Group - D.Akers, J.Brock, C.Flanagan, C.Keith*, D.Meekins, ...
Injector Group - M.Polker*, ...
Hall B - P. Dobrenz, Xiangdong Wei*, ...
Cryo Group - B.Mastracci, ...



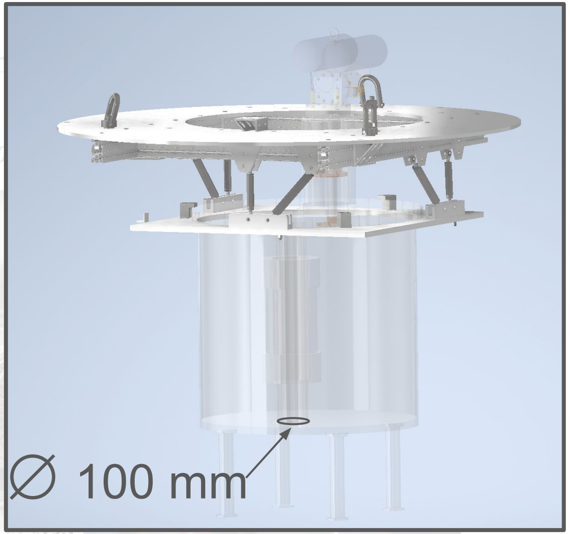
DNP 1 K Test Refrigerator

Polarized Target Group - J.Brock, C.Keith*, J.Maxwell, D.Meekins,...

DNP Test Refrigerator - Overview

- **Cryomagnetics Cryo-Free 5 T Warm Bore Magnet**
- **Insulating Vacuum Can w/ Alignment**
- **Heat Shield**
- **G10/316 SS Laminated He Pumpin**
- **1 K Refrigerator**

Shorter Vertical Versio



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Shorter Vertical Version of RCG Fridge

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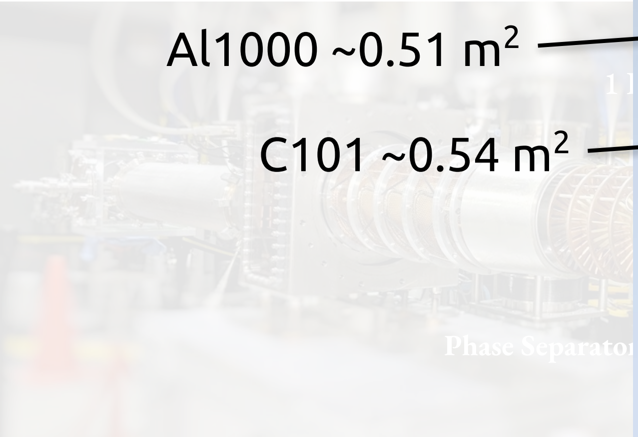

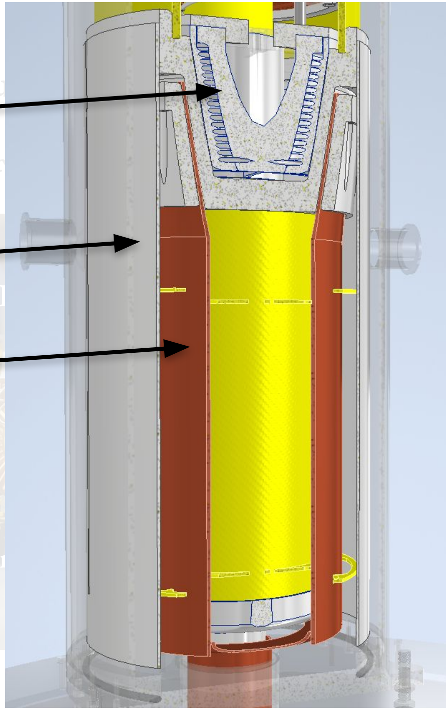

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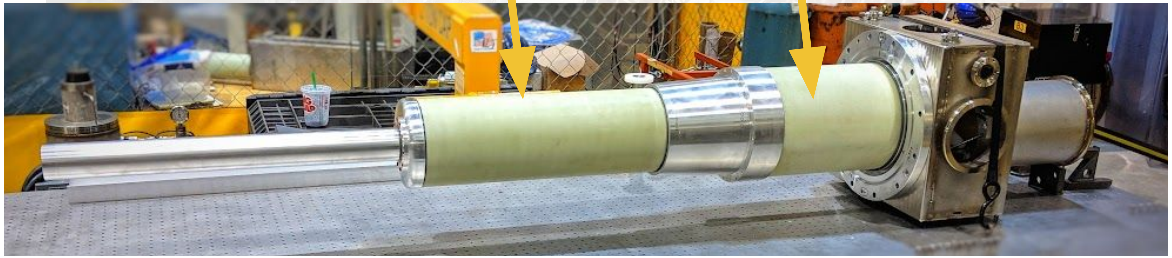
Shorter Vertical Version of RCG Fridge

G10 vs 316SS: thermal load reduction
~3.6 (T = 290 to 40 K) & ~3.4 (T = 40 to 1K)



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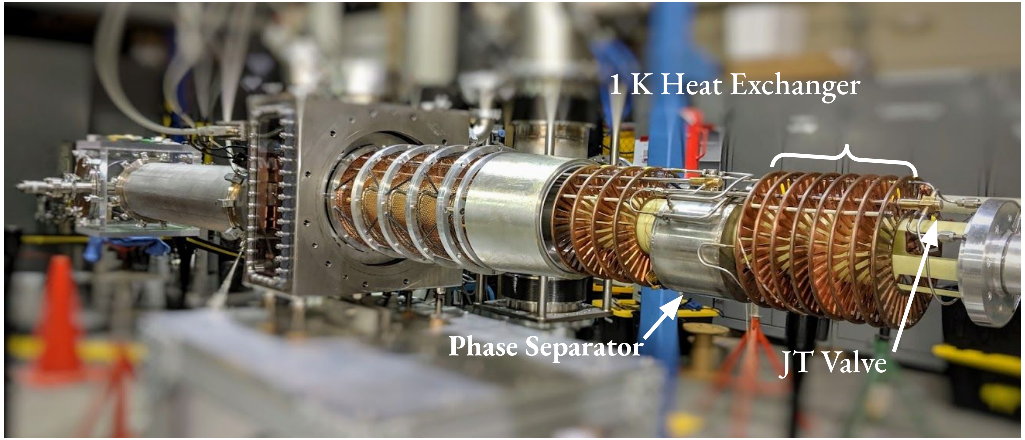

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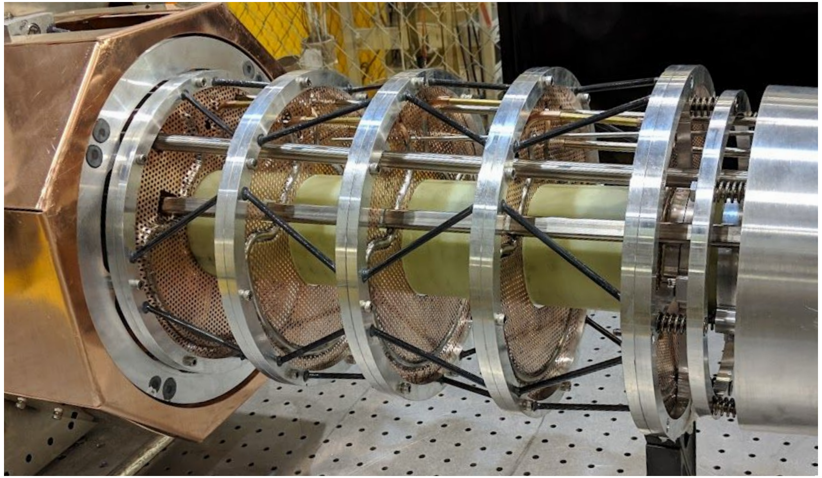


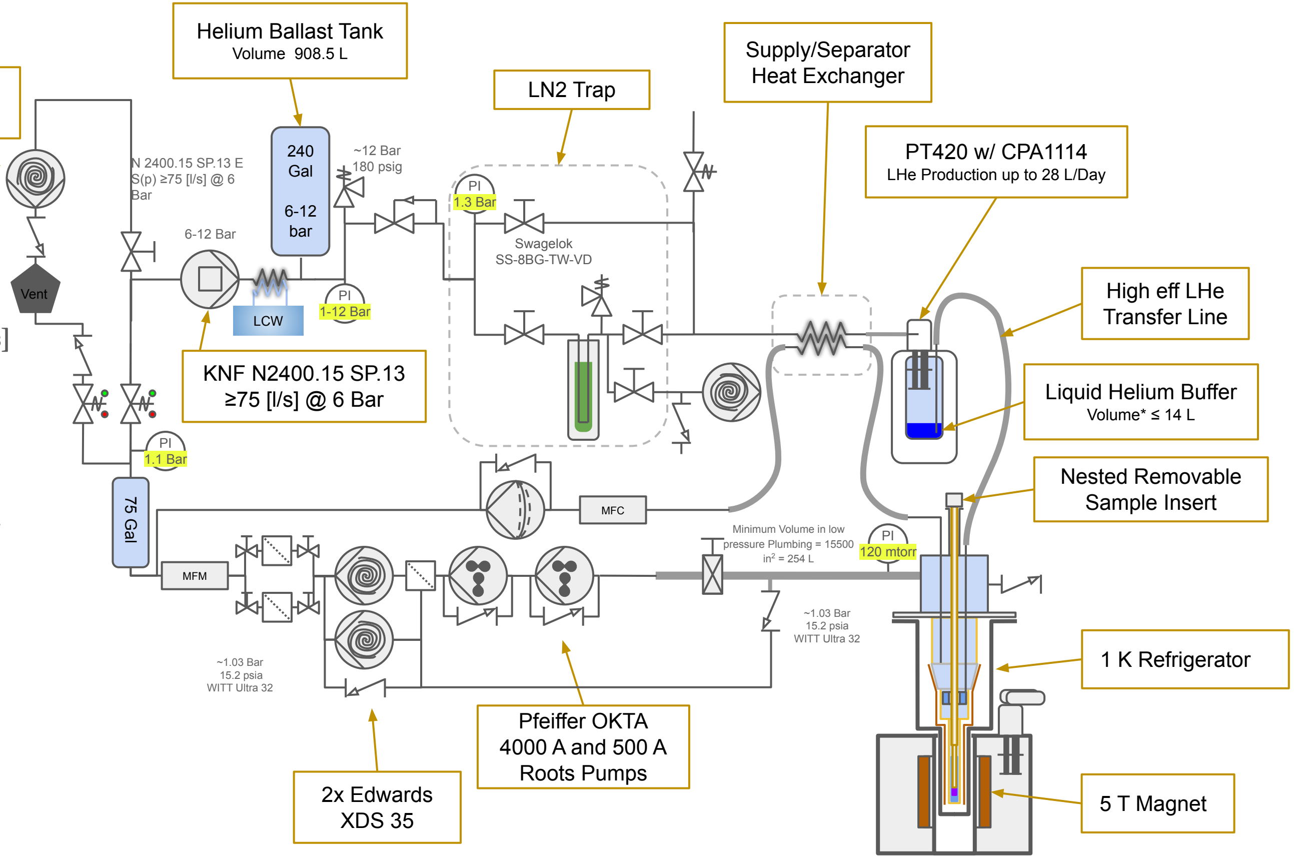
Est. Continuous Combined Flow Rate
10 - 14.5 SLPM (separator and bath)**, or**
19.3 - 28 LL He/day (RGC Fridge 25 LL He/day)

1 K Fridge Max Flow Rate ~32 SLPM [0.095 g/s]
(Limited due to dual Edwards XDS 35)

KNF Compressor ~75 SLPM @ 6 Bar
(Intermittent up 40 SLPM @ 12 Bar)

Liquid He Operational Buffer Volume 7 to 14* L
* Compressor max pressure dependent

Max Liquefaction Rate 0.039 g/s



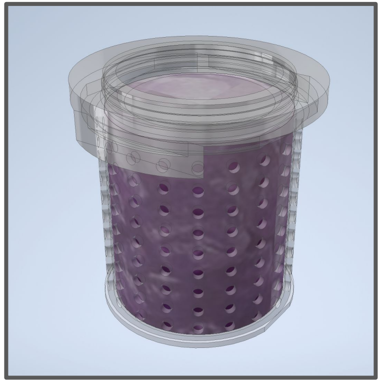
Lattice damage from radiation dosing causes chemical changes in ammonia affecting the local magnetic and electric field near the paramagnetic centers that are essential for DNP.

ESR Spectroscopy

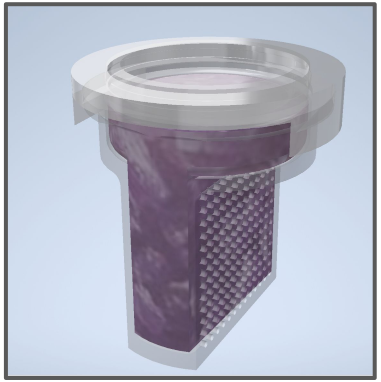
Investigate the ESR spectral structure of the paramagnetic radicals produced from irradiating NH_3 and ND_3

UV-Vis-NIR, Mid-IR Spectroscopy*

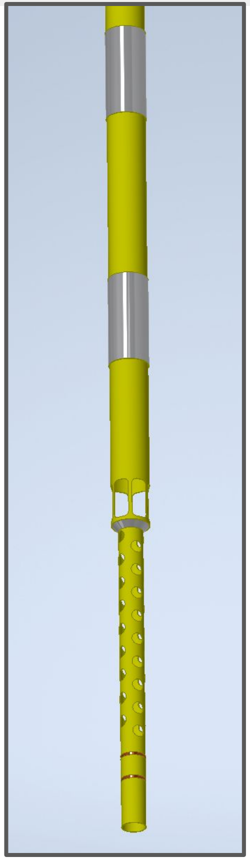
Absorption spectrums can identify the resulting chemical compounds eg. diimide (N_2H_2), hydrazine (N_2H_4), hydrazoic acid (HN_3), and aminos radicals (•NH_2)



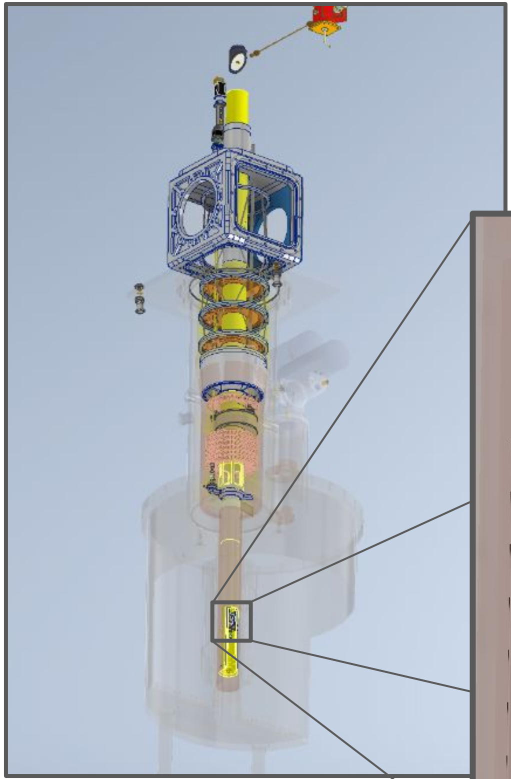
3 cc PCTFE Sample Cell



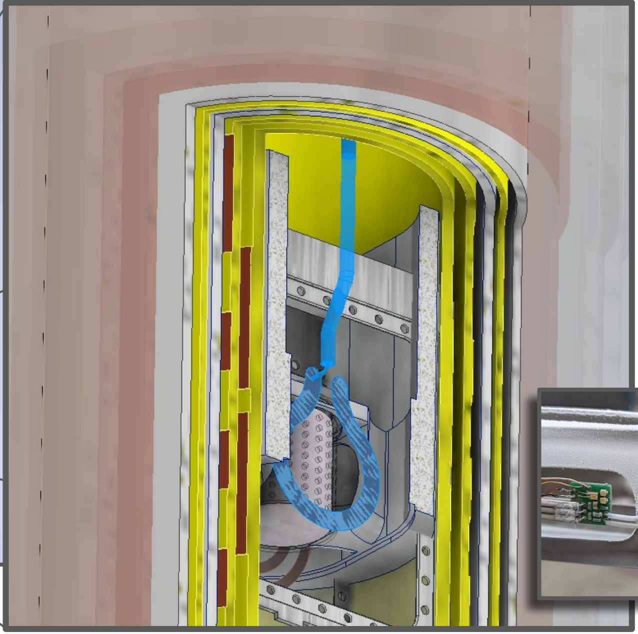
1.5 cc PCTFE Sample Cell

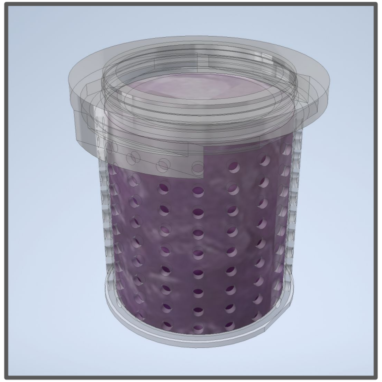


DNP Targets - Insert w/ NMR Coils, Super Conducting Helmholtz Coils



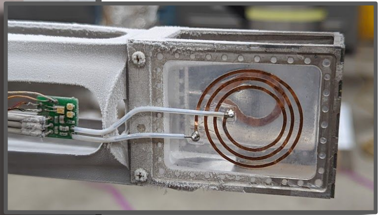
FEP Laminated Cu NMR Coils
on MgAl Frame



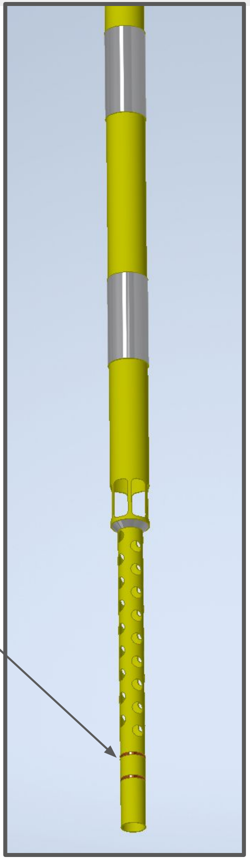


3 cc PCTFE Sample Cell

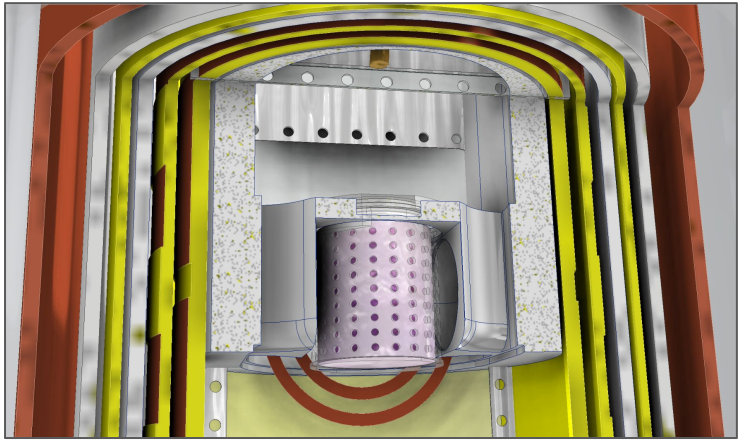
RGC Deuteron Coil

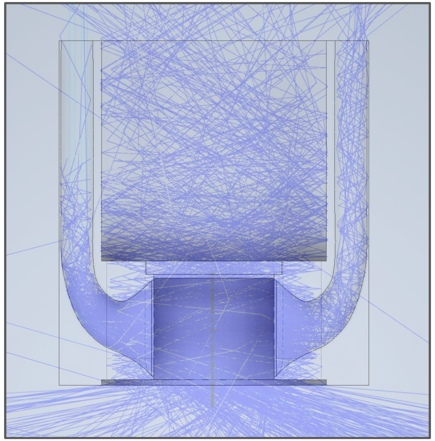


Helmholtz Coils

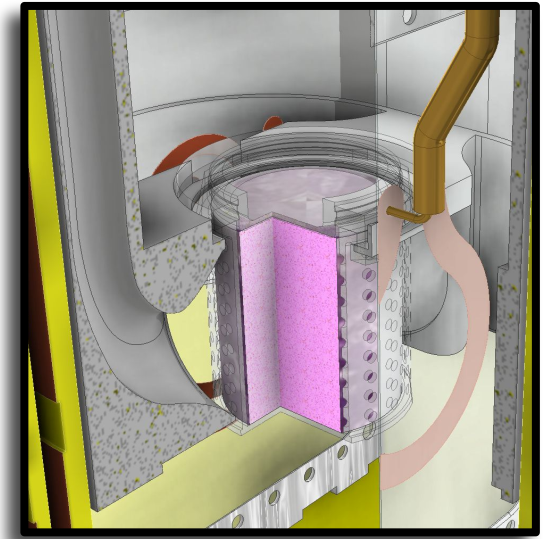


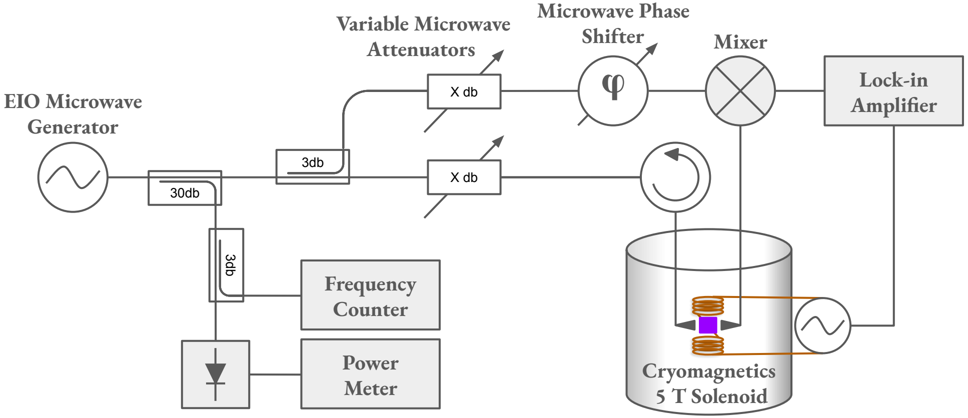
DNP Targets - Microwave Horn and ESR Spectroscopy





Direct Metal Laser Sintering (DMLS)
AlSi10Mg





Ray tracing software was used to estimate microwave propagation through the oversized waveguides because of the multimode quasi-optical transmission.

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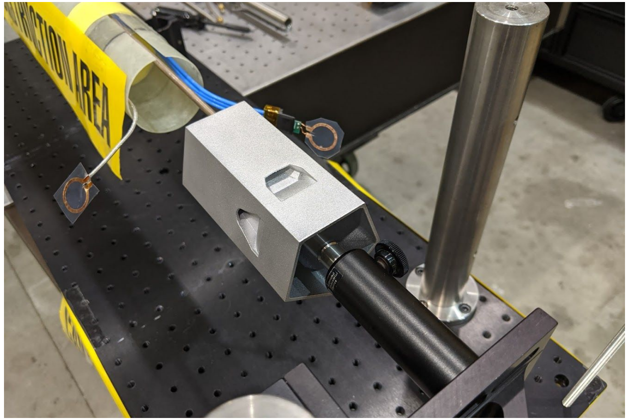
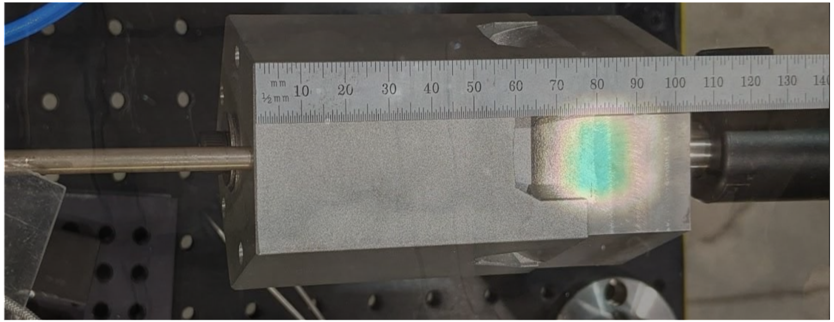
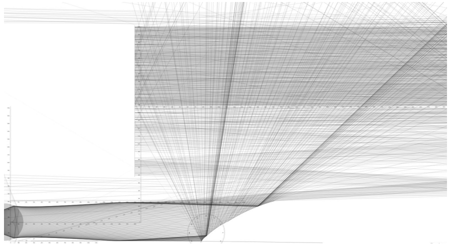
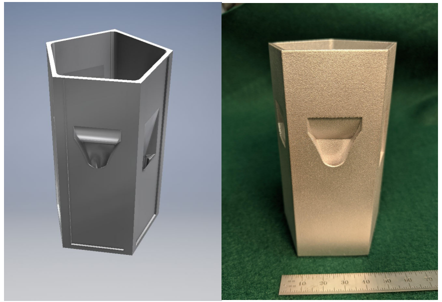


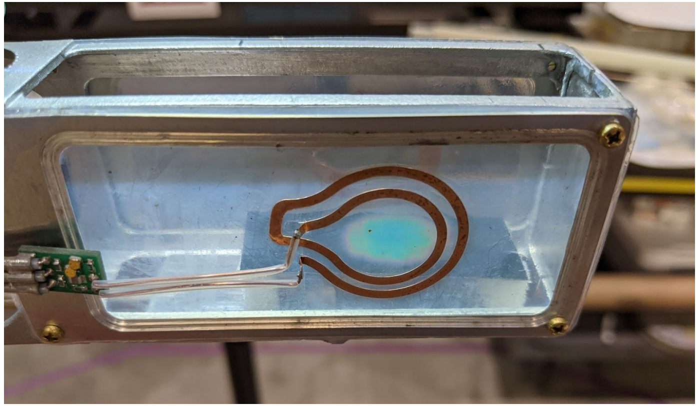
Development of 3D Printed Microwave Horn for RGC using Thermochromic Film



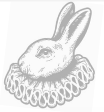


Direct Metal Laser Sintering (DMLS) AlSi10Mg





Literature Findings on Irradiated Frozen Ammonia



Chemical Physics Letters
The Astrophysical Journal
J. Chem. Phys. etc. ...

- IR spectra identifies the chemical species generated post irradiation eg. diazene, hydrazine, hydrazoic acid and aminos radicals. This has been investigated in the chemistry of **low-mass young stellar objects (YSOs)** and **dark cloud chemistry** of nitrogen (3 - 10 K)
- Sublimation, Mobility, and Abstraction of Irradiated NH_3.
- Phase Transition at 56K NH_3, amorphous to Crystalline. Sublimation occurs at 80K in vacuum.

- Investigate the ESR spectral structure of the paramagnetic radicals produced from radiation dose NH_3 and ND_3
- Monitor the absorption spectra of processed(ing) NH_3

- Possibility of real time Polarized Target Monitoring and Conditioning: “Selective Annealing”

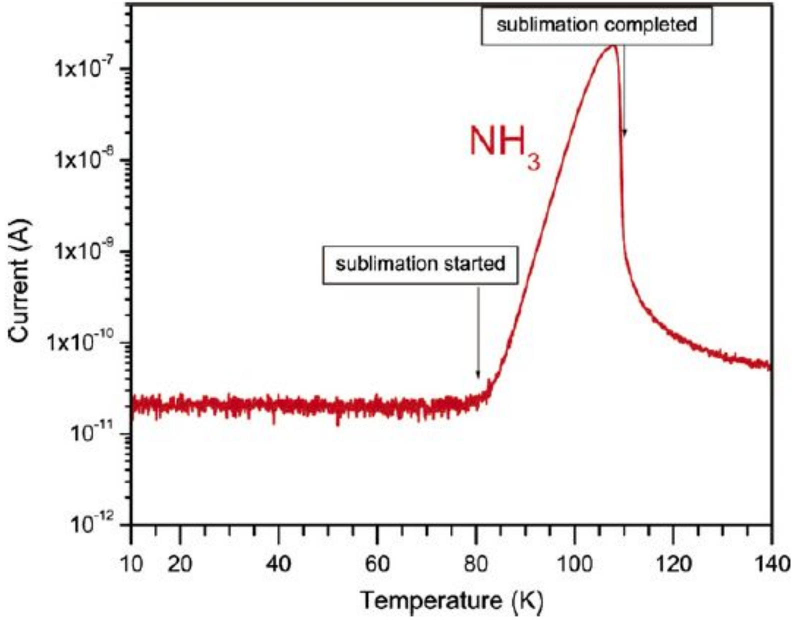


Fig. 1. The ion current of ammonia ($m/z = 17$) monitored via the quadrupole mass spectrometer in the gas phase versus the temperature of the solid ammonia sample.

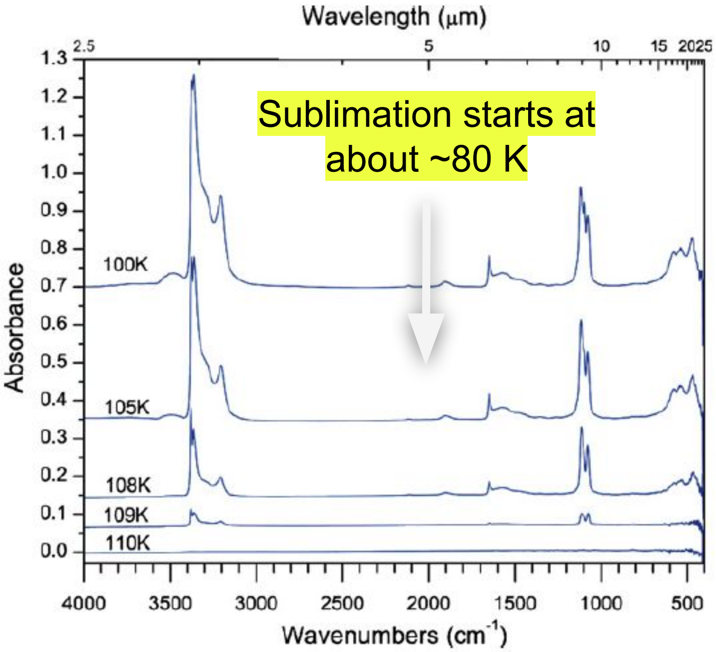
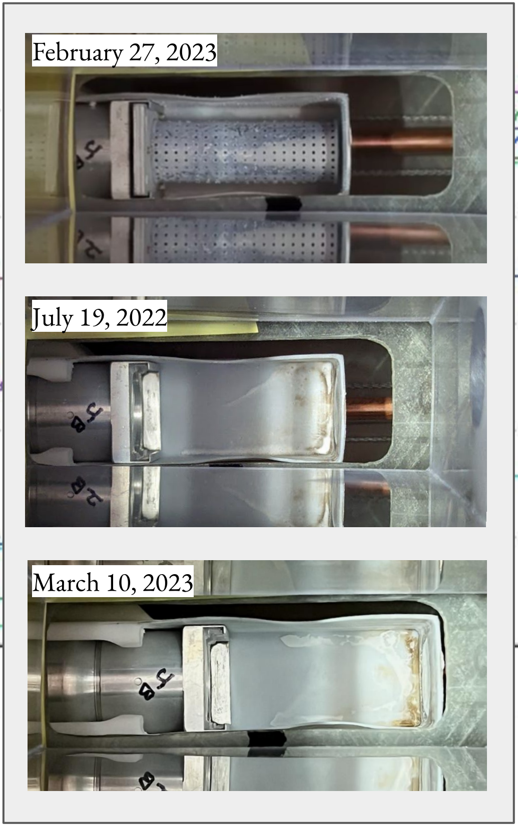


Fig. 2. Infrared spectra of ammonia at various temperatures in the sublimation range of the sample.

W. Zheng, R.I. Kaiser / Chemical Physics Letters 440 (2007) 229–234

DNP Targets - NH_3 Packing Fraction, Material Settling, Sublimation, H_2 Abstraction



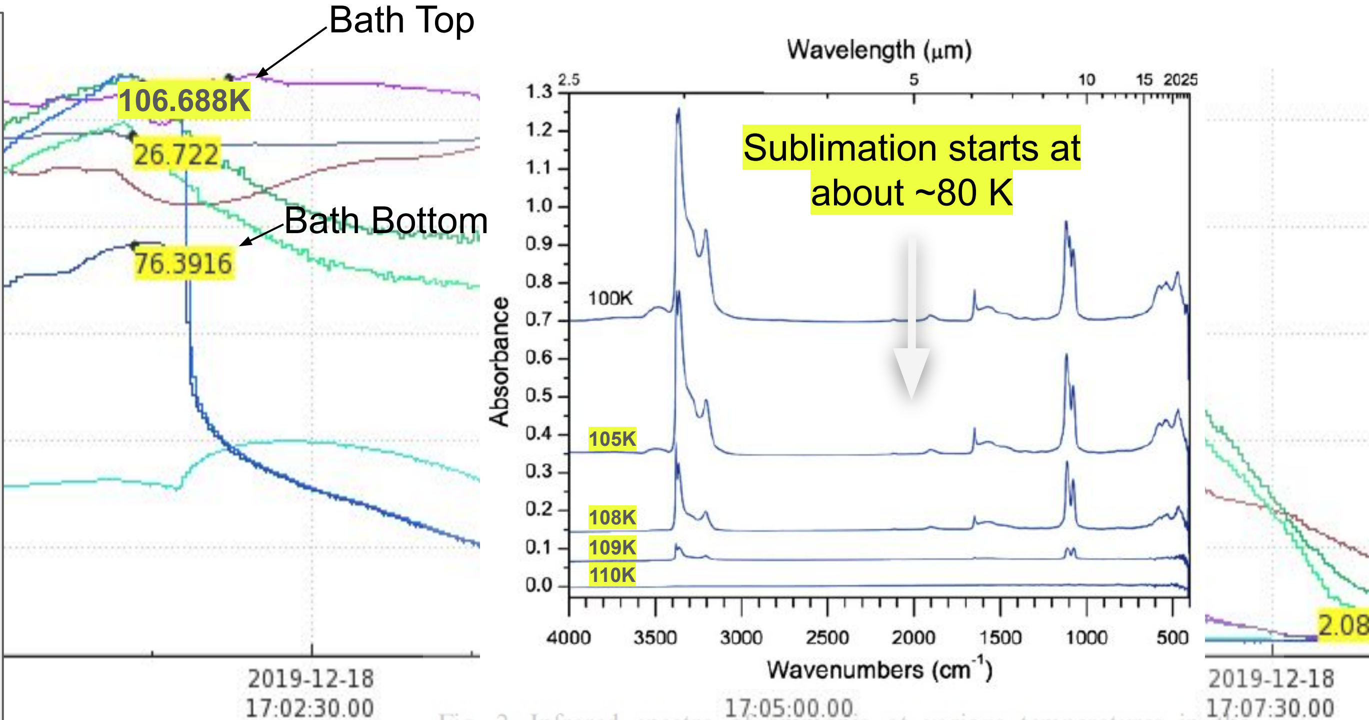


Fig. 2. Infrared spectra of ammonia at various temperatures in the sublimation range of the sample.

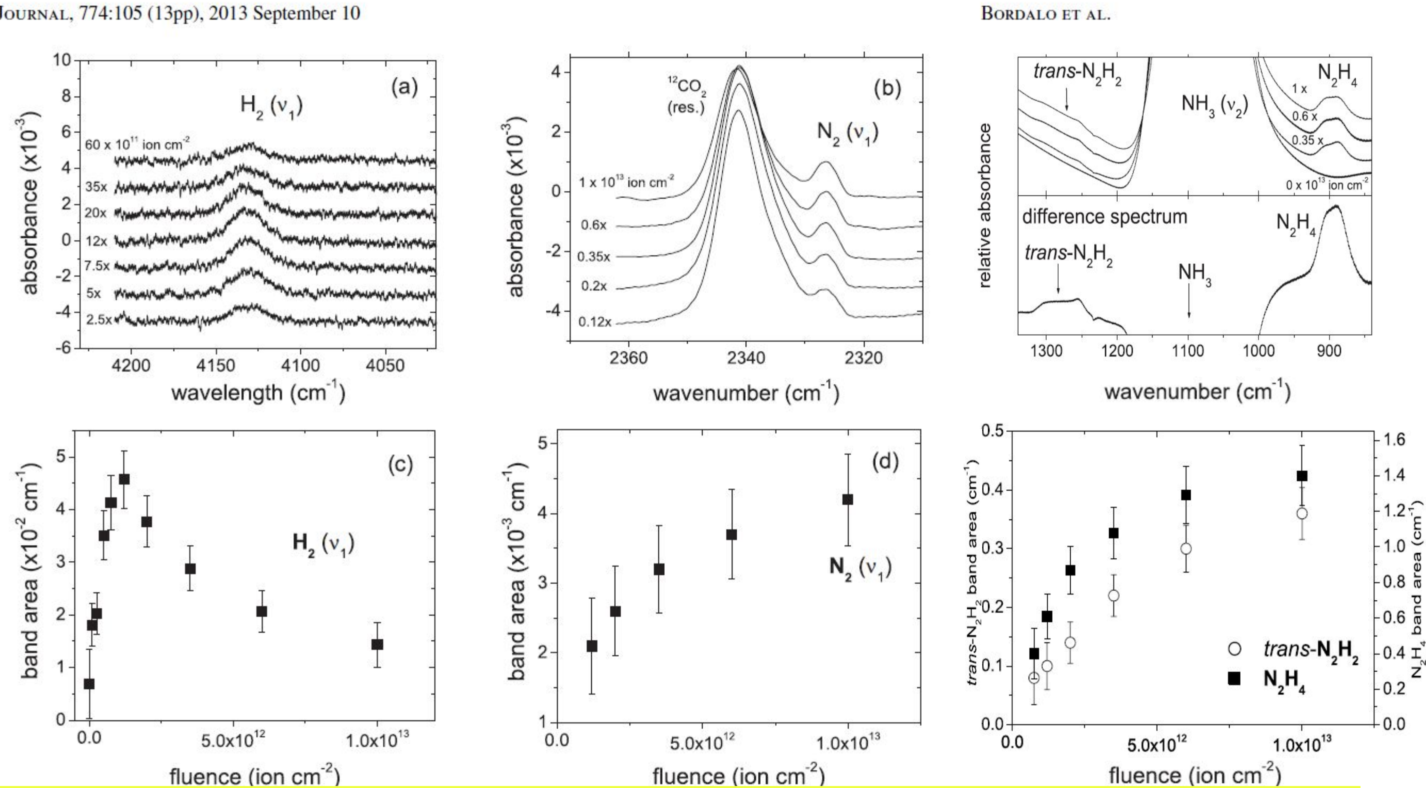
W. Zheng, R.I. Kaiser / Chemical Physics Letters 440 (2007) 229–234

DNP Targets - NH_3 Infrared Spectral Analysis - H_2 Abstraction

Irradiation Effect on Hydrogen Abstraction from Ammonia Ice

NH_3 on CeI at 14 K
(thin sample is assumed amorphous)

Heavy Ion irradiation 0.53-0.6 GeV
δ-ray are predominantly responsible for excitation and ionizations.



During Irradiation, N_2, N_2H_4, etc. remains while H_2 Abstraction occurs in the solid at 14 K

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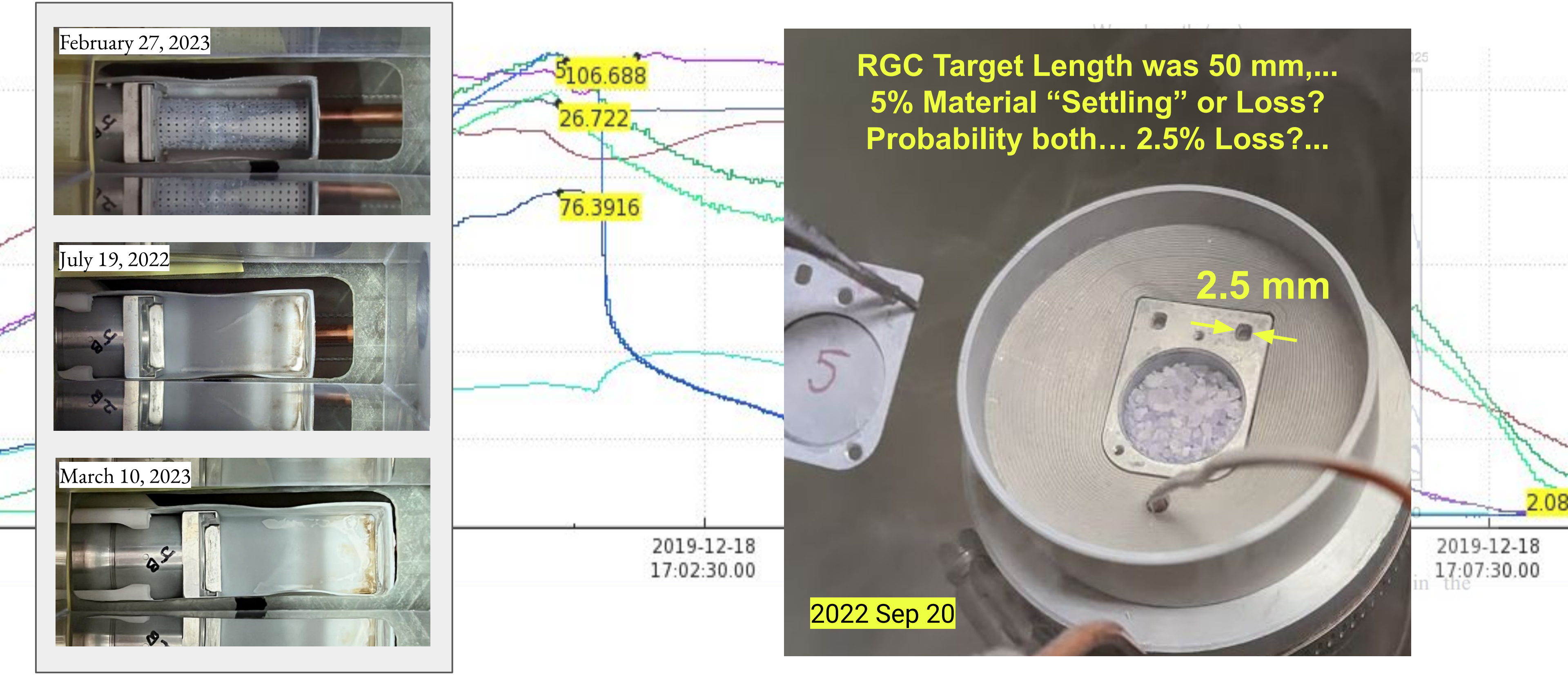
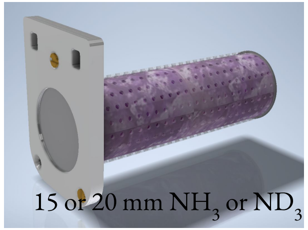
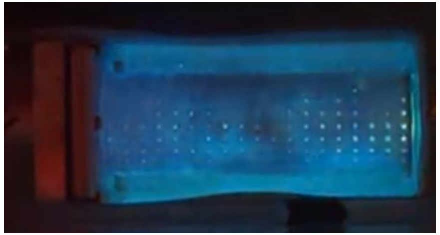
DNP Targets - NH_3 Packing Fraction, Material Settling, Sublimation, H_2 Abstraction

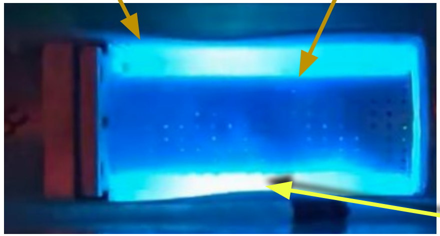


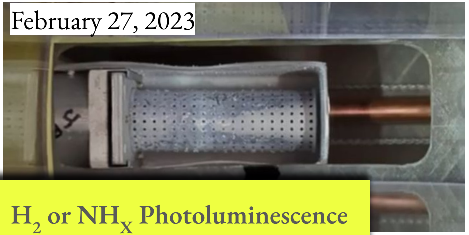
W. Zheng, R.I. Kaiser / Chemical Physics Letters 440 (2007) 229–234

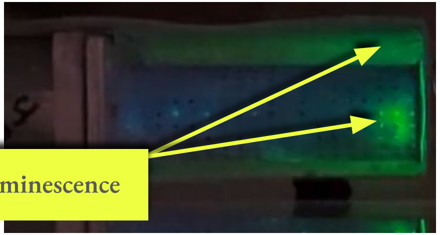
DNP Targets - Longitudinally Polarized Target for CLAS12

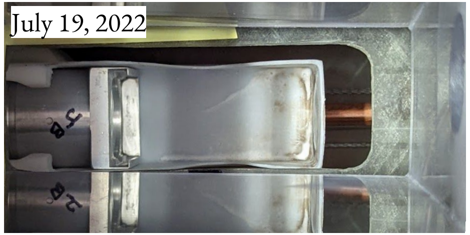


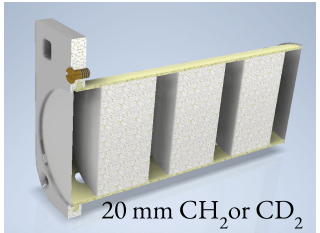


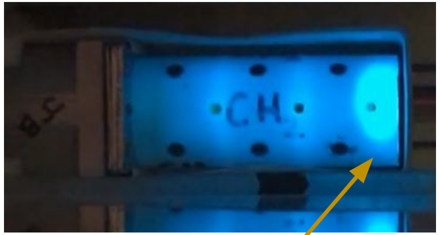


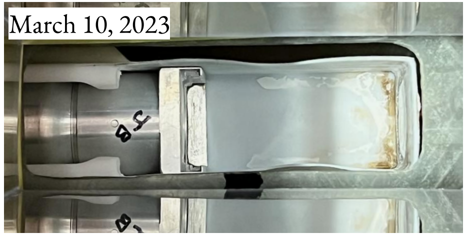












An infrared spectroscopy study of the phase transition in solid ammonia

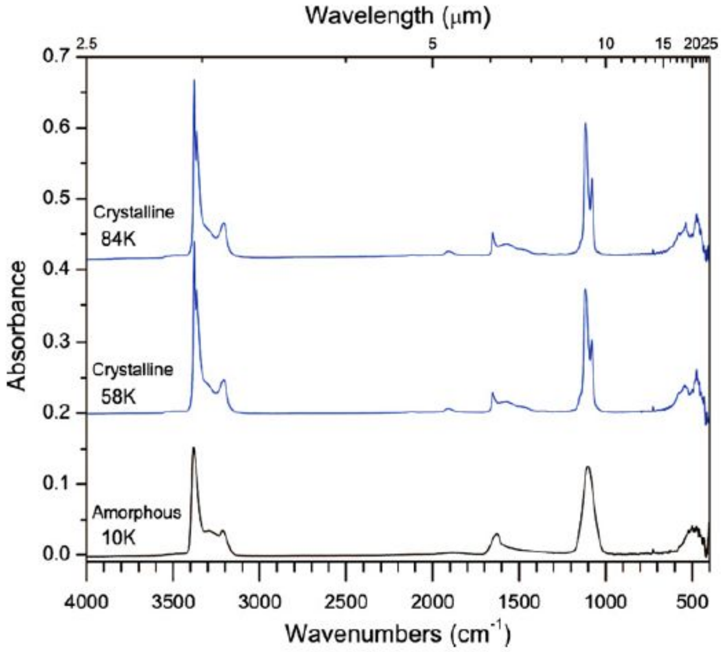


Fig. 4. Mid-infrared spectra of a 40 nm thick solid ammonia film at 10 K (amorphous), 58 K (cubic crystalline), and 84 K (cubic crystalline).

Phase Transition
amorphous to cubic crystalline
~57 K

W. Zheng, R.I. Kaiser / Chemical Physics Letters 440 (2007) 229–234

Mobility in the Lattice

Ammonia ice condensed at 10 K form anhydrous ammonia and heated from 10 K for the first time.

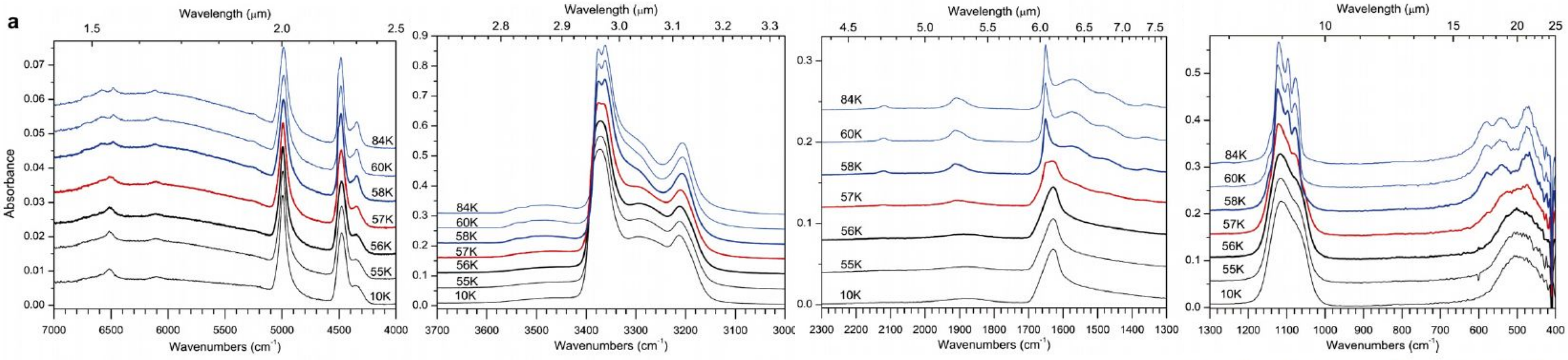


Fig. 3. Infrared spectra of ammonia at different temperatures during the warm up phase of amorphous ammonia. The amorphous-cubic phase transition happens at about **57 K**. The spectra are offset for clarity.

W. Zheng, R.I. Kaiser / Chemical Physics Letters 440 (2007) 229–234

Table 4
The Infrared Absorption Features of Solid Ammonia and Species Formed after Irradiation

(μm)	Absorption (cm^{-1})	References	Species	Feature Assignments
			NH_3 amorphous sample at 14 K	
2.002	4994	1	NH_3	$\nu_3 + \nu_4$
2.233	4478	1	NH_3	$\nu_3 + \nu_2$
2.293	4361	1	NH_3	$\nu_1 + \nu_2$
2.875	3478	1	NH_3	$\nu_1 + \nu_L$
2.891	3459	1,2	NH_3	ν_3 deg. N–H stretch.
3.114	3211	1,2	NH_3	ν_1 sym. N–H stretch.
5.319	1880	1	NH_3	$\nu_4 + \nu_L$
6.154	1625	1,2	NH_3	ν_4 deg. N–H deform.
9.311	1074	1,2	NH_3	ν_2 sym. N–H deform./“umbrella”
			Radiolytic products	
2.421	4131	3	H_2	ν_1 H–H stretch.
3.289	~3040	4,11	*cis*-N_2H_2	ν_5 N–H stretch.
3.460	2890	5	NH_4^+	$2\nu_4$
3.597	~2780	4,11	*iso*-N_2H_2	ν_5 N–H stretch.
4.299	2326	3,4	N_2	ν_1 N–N stretch.
4.798	2084–2060	4,6	$NH_4^+N_3^-$	N–N–N asym. stretch.
4.854	2022	6,7	N_3^-	N–N–N asym. stretch.
4.946	~1500	5	NH_4^+	ν_4 N–H bend.
6.667		8,9,12	NH_2	ν_2 N–H bend.
7.813	~1280	10,11	*trans*-N_2H_2	ν_5,ν_6 N–H bend.
11.173	~895	4	N_2H_4	ν_6 NH_2 rock

References. (1) Zheng & Kaiser 2007; (2) Shimanouchi 1972; (3) Loeffler et al. 2010; (4) Zheng et al. 2008; (5) Schutte & Khanna 2003; (6) Carlo et al. 2001; (7) Tian et al. 1988; (8) Gerakines et al. 1996; (9) Suzer & Andrews 1988; (10) Rosengren & Pimentel 1965; (11) Biczysko et al. 2006; (12) Milligan & Jacox 1965.

Selective Annealing Irradiated Ammonia

Over increasing temperature ranges, use IR Bandpass Filters to mask **selective** absorption peaks for different vibrational modes of amidogen radicals while monitoring polarization and ESR Spectra.

Begin w/ over irradiated material…

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DNP Targets - NH_3 Infrared Absorption Peaks & Annealing

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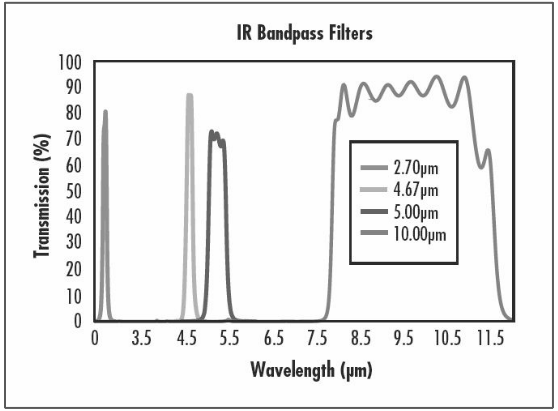
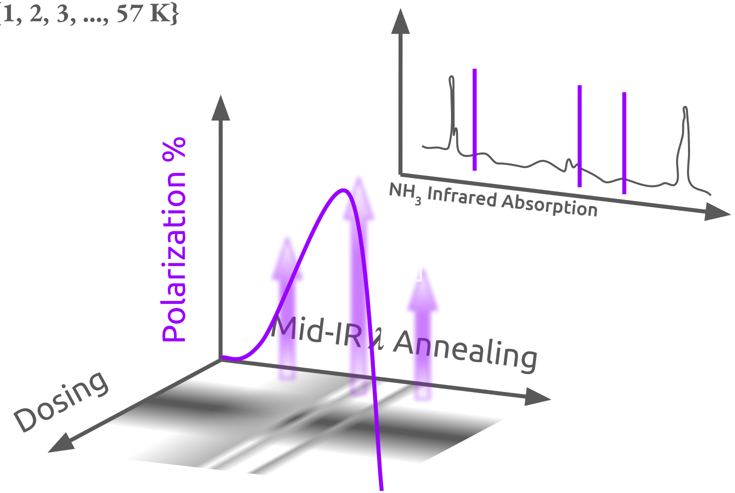


V. Bordalo, et al/ The Astrophysical Journal, 774:105 (13pp), 2013 September 10

$T_n = \{1, 2, 3, ..., 57\ K\}$



Selective Annealing Irradiated Ammonia

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DNP Targets - NH_3 Infrared Absorption Peaks & Annealing

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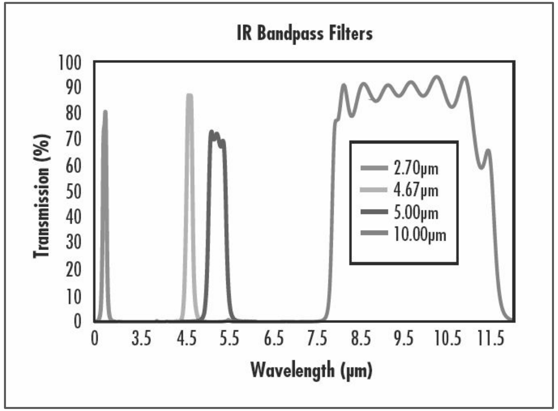
Selective Annealing Irradiated Ammonia

OR, over increasing temperature ranges, use IR light source circa **6.67μm** to mobilize amino radicals (•NH_2) and ammonium (N_2H_4) radicals to reduce the paramagnetic centers in over irradiated material while monitoring polarization and recording ESR Spectra.



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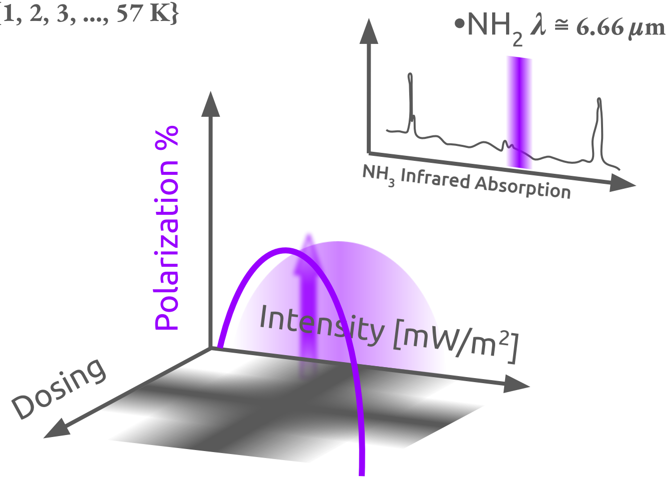
$T_n = \{1, 2, 3, ..., 57\ K\}$



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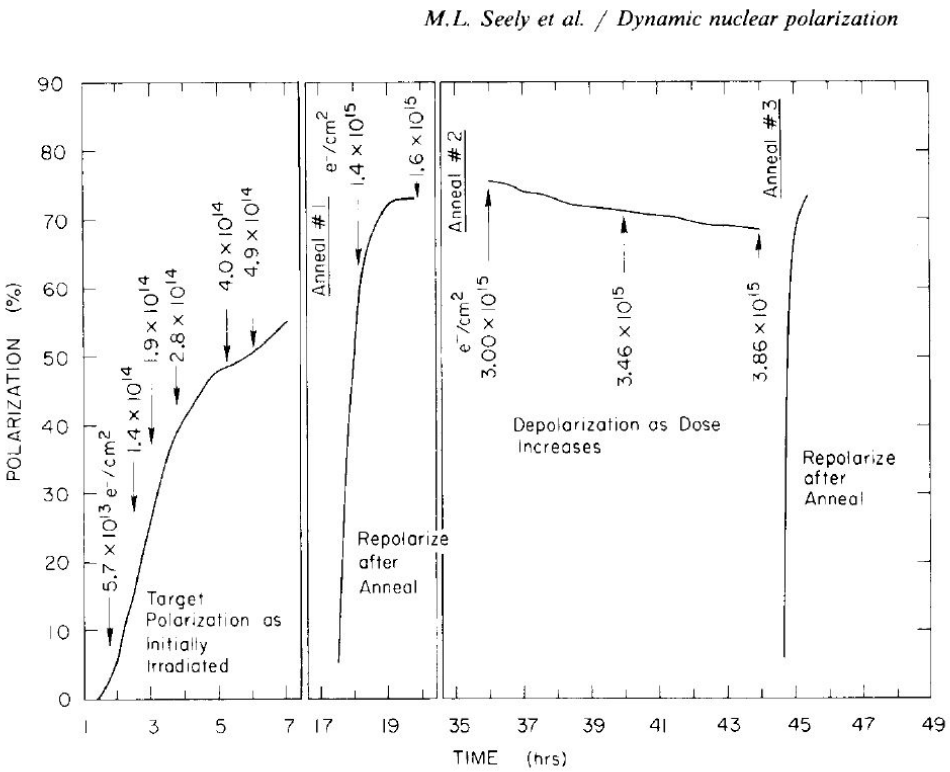
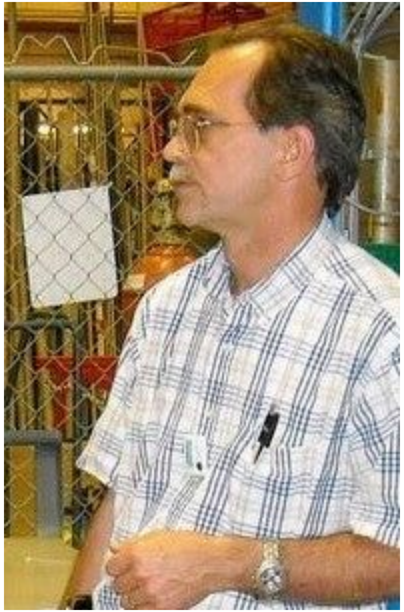
Thank you,
End

DNP Targets - Effects of Material Dosing

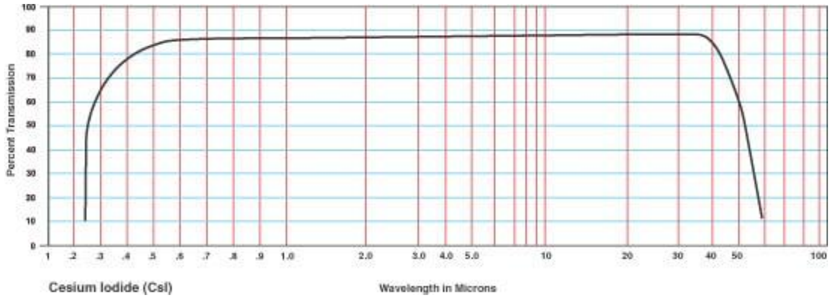


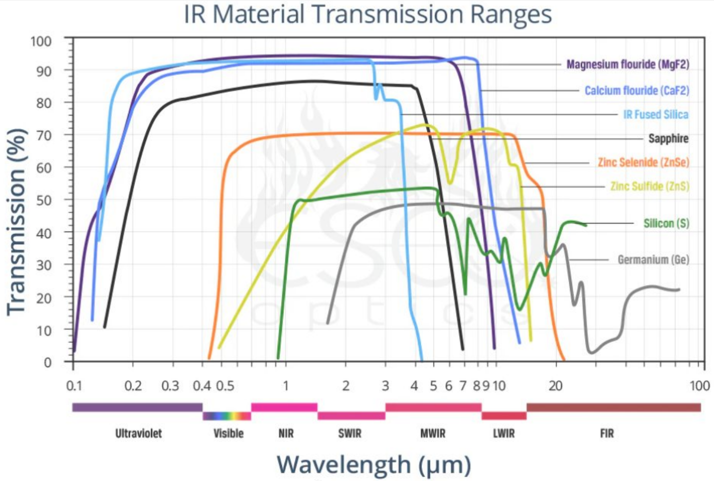


Mikell L. Seely
(1957 - 2023)

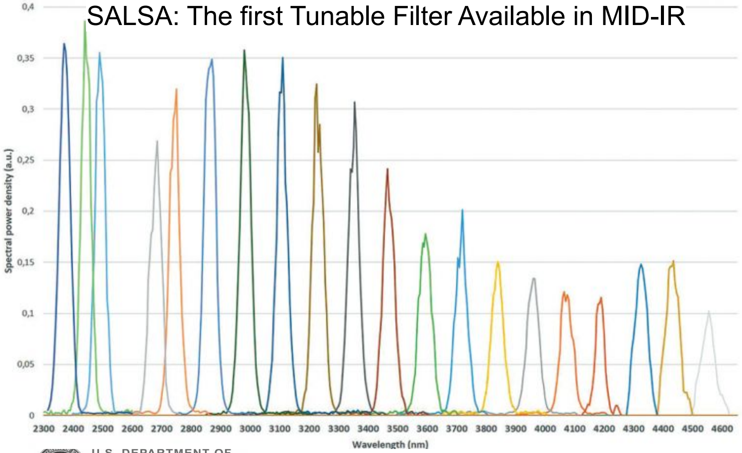
DNP Targets - NH_3 Infrared Absorption Peaks

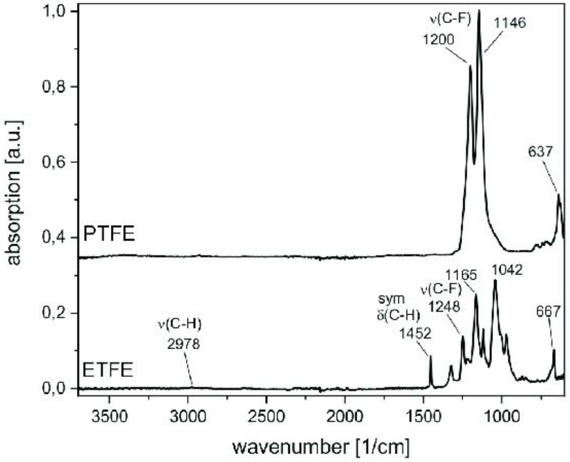
DNP Targets - NH_3 Infrared Spectral Analysis



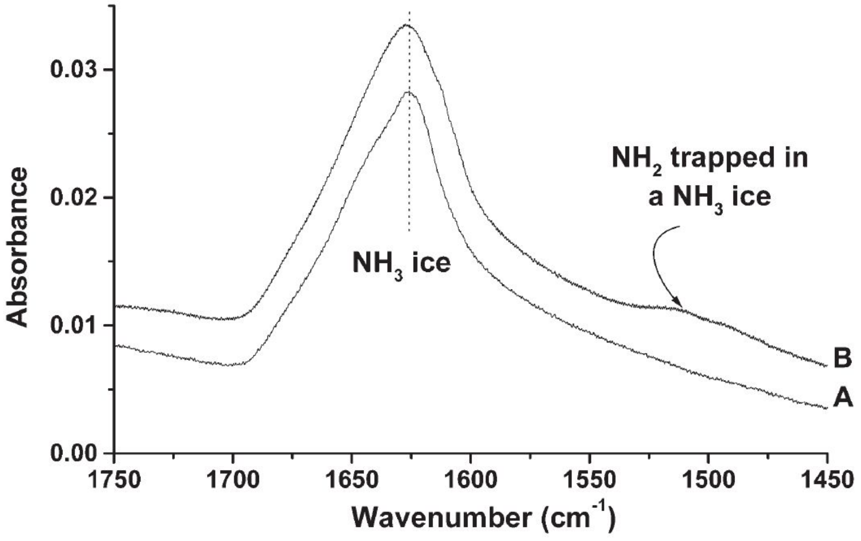


SALSA: The first Tunable Filter Available in MID-IR

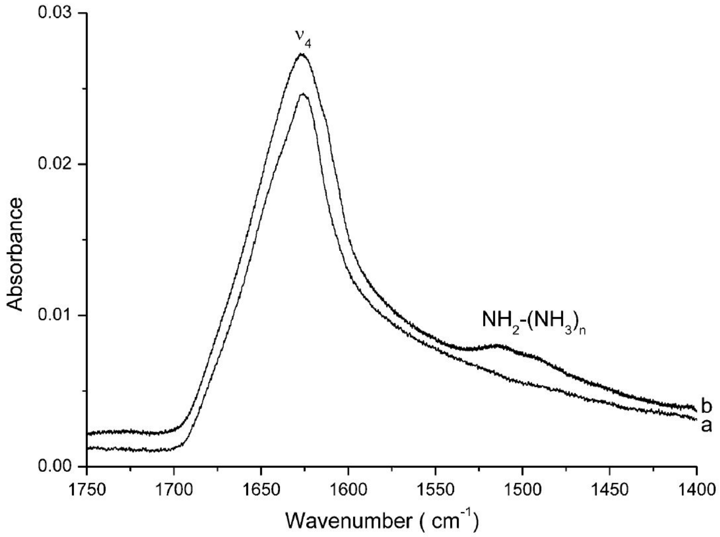




Deuterium Lamp VUV Photolysis of NH_3 (30 min)



E. L. Zins* and L. Krim/RSC Advances, 2013, 3, 10285



Sendres Nourry and Lahouari Krim*/Phys.Chem.Chem.Phys., 2016, 18, 18493

DNP Targets - Chemical Vapor Deposition at 3 K, Followed by a 10 K Anneal $NH_3 + N + N_2$

Co-condensing NH_3 molecules and N atoms at 3 K followed by 10 K Annealing

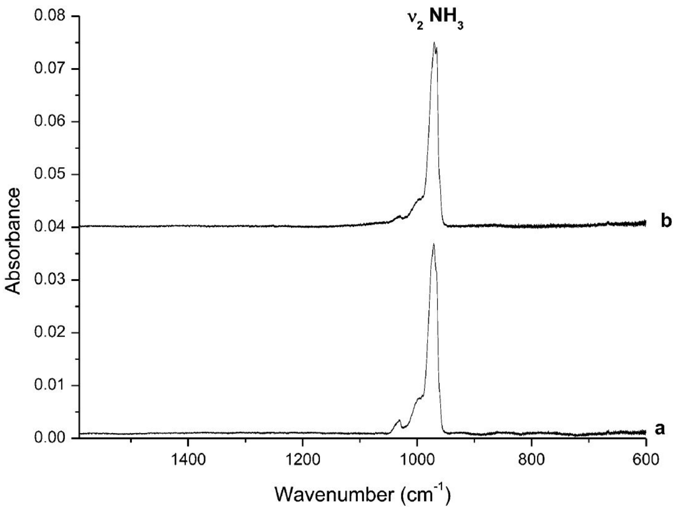


Fig. 3 NH_3 and NH_2 spectral regions. (a) $NH_3 + N + N_2$ reaction and co-injection of the NH_3 and N/N_2 mixture at 3 K. (b) $NH_3 + N_2$ reaction and co-injection of NH_3 and N_2 gas at 3 K.

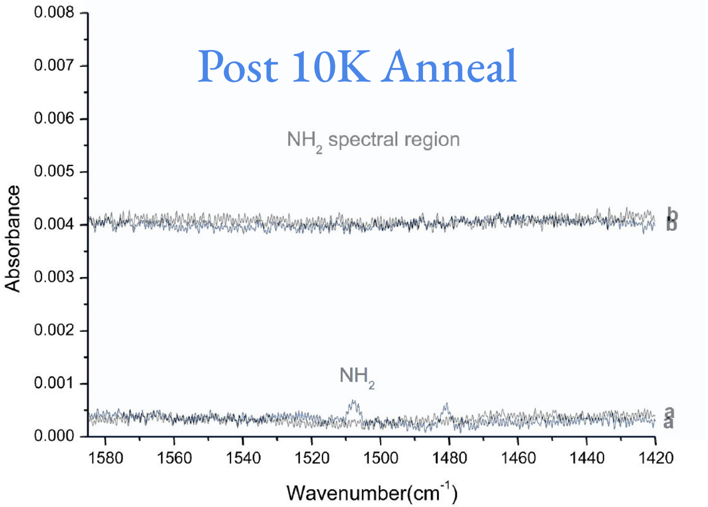


Fig. 5 NH_2 spectral region. (a) $NH_3 + N + N_2$ reaction, co-injection of the NH_3 and N/N_2 mixture at 3 K and heating of the sample at 10 K. (b) $NH_3 + N_2$ reaction and co-injection of NH_3 and N_2 gas at 3 K, and heating of the sample at 10 K. All the IR spectra are recorded at 3 K.

Sendres Nourry and Lahouari Krim*/Phys.Chem.Chem.Phys., 2016, 18, 18493

Co-condensing NH_3 molecules and N atoms at 3 K followed by 10 K Annealing

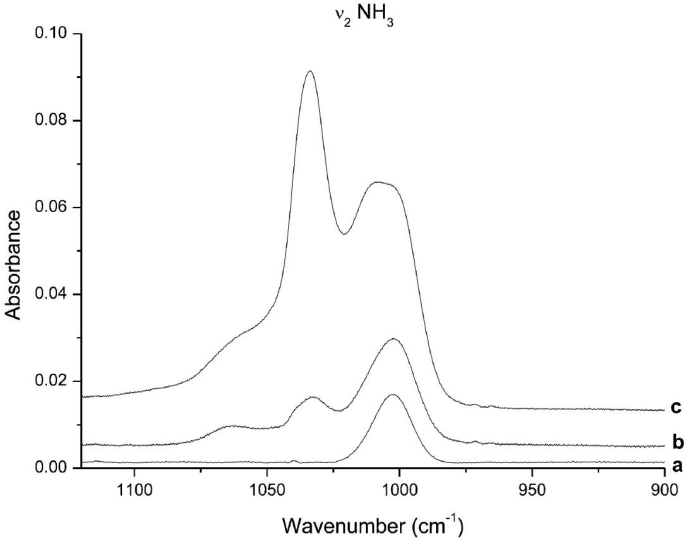


Fig. 6 NH_3 spectral region. $NH_3 + N + N_2$ reaction, co-injection of the NH_3 and N/N_2 mixture at 3 K and heating of the sample at 10 K. (a) $[NH_3] = 0.2 \times 10^{17}$ molecules per cm^2, (b) $[NH_3] = 0.7 \times 10^{17}$ molecules per cm^2, and (c) $[NH_3] = 2.5 \times 10^{17}$ molecules per cm^2. All the IR spectra are registered at 3 K.

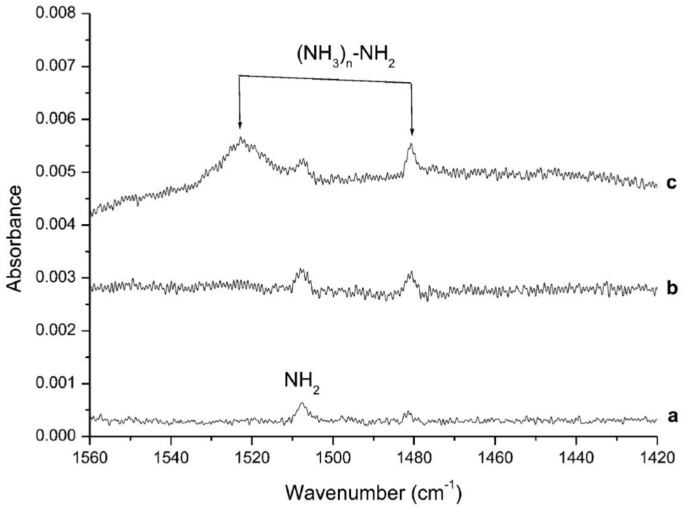
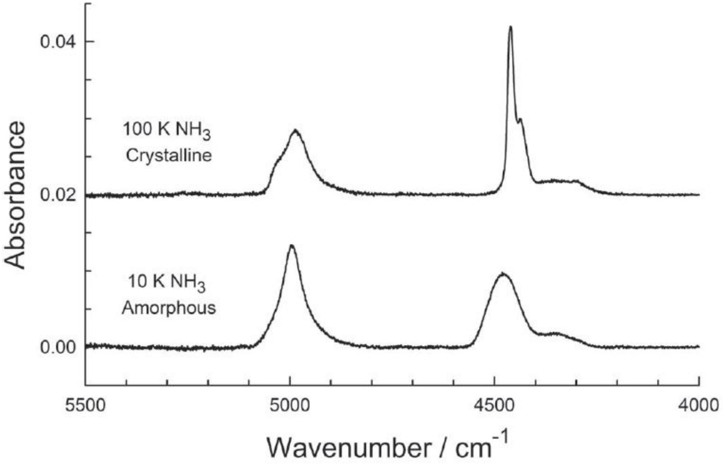


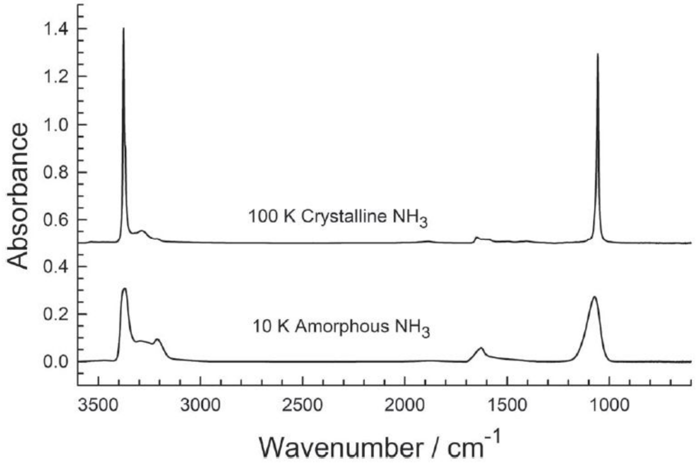
Fig. 7 NH_2 spectral region. $NH_3 + N + N_2$ reaction, co-injection of the NH_3 and N/N_2 mixture at 3 K and heating of the sample at 10 K. (a) $[NH_3] = 0.2 \times 10^{17}$ molecules per cm^2, (b) $[NH_3] = 0.7 \times 10^{17}$ molecules per cm^2, and (c) $[NH_3] = 2.5 \times 10^{17}$ molecules per cm^2. All the IR spectra are registered at 3 K.

Sendres Nourry and Lahouari Krim*/Phys.Chem.Chem.Phys., 2016, 18, 18493

An infrared spectroscopy study of the phase transition in solid ammonia

Amorphous Grown at 18 K Cubic Crystalline Grown at 100 K





Hudson, Gerakines, & Yarnall / The Astrophysical Journal, 925:156 (8pp), 2022 February 1

VUV and FTIR spectroscopy study of the temperature dependent phase transition in solid ammonia

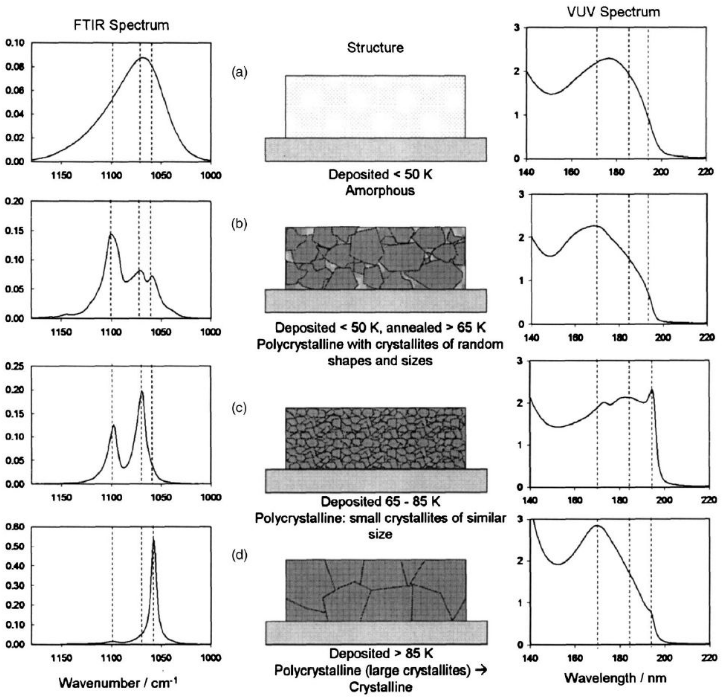


FIG. 9. A summary of the morphology of solid ammonia films as interpreted from the FTIR (left) and the VUV (right) spectra.

Structure and morphology depends on Deposition and Cycling Temperature

Anita Dawes, Robin J. Mukerji, Michael P. Davis, et al. / J. Chem. Phys. 126, 244711 (2007)

DNP Targets - Development of the 3D Printed RGC Microwave Horn