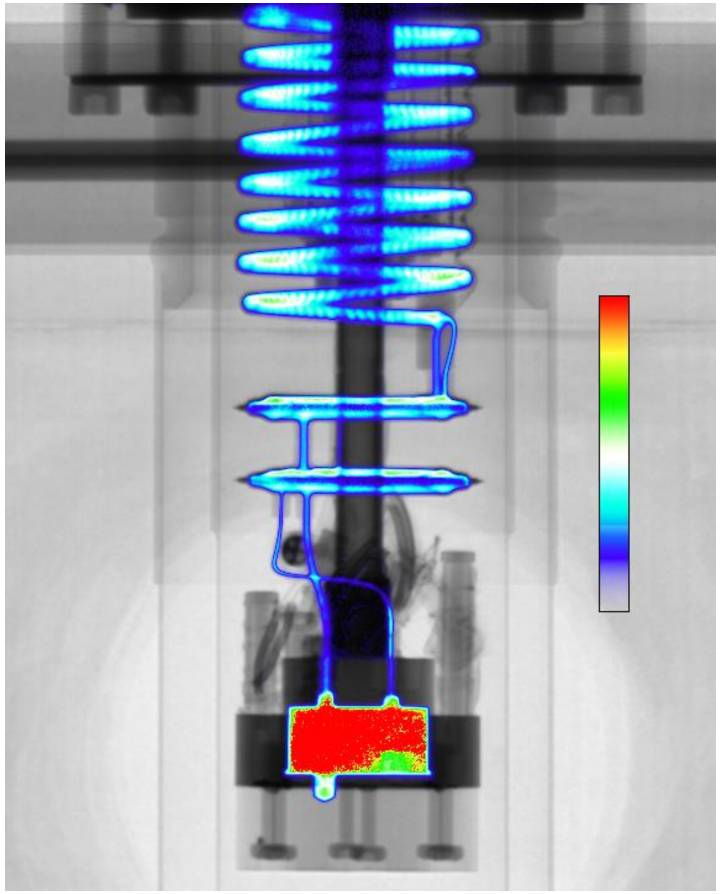
Neutron Imaging of an Operational Dilution Refrigerator

Chris Lawson, Alex Jones, Winfried Kockelmann, Sasha Horney, Oleg Kirichek

ISIS Neutron & Muon Source, Oxford, UK

ICEC29 – Geneva

23rd July 2024



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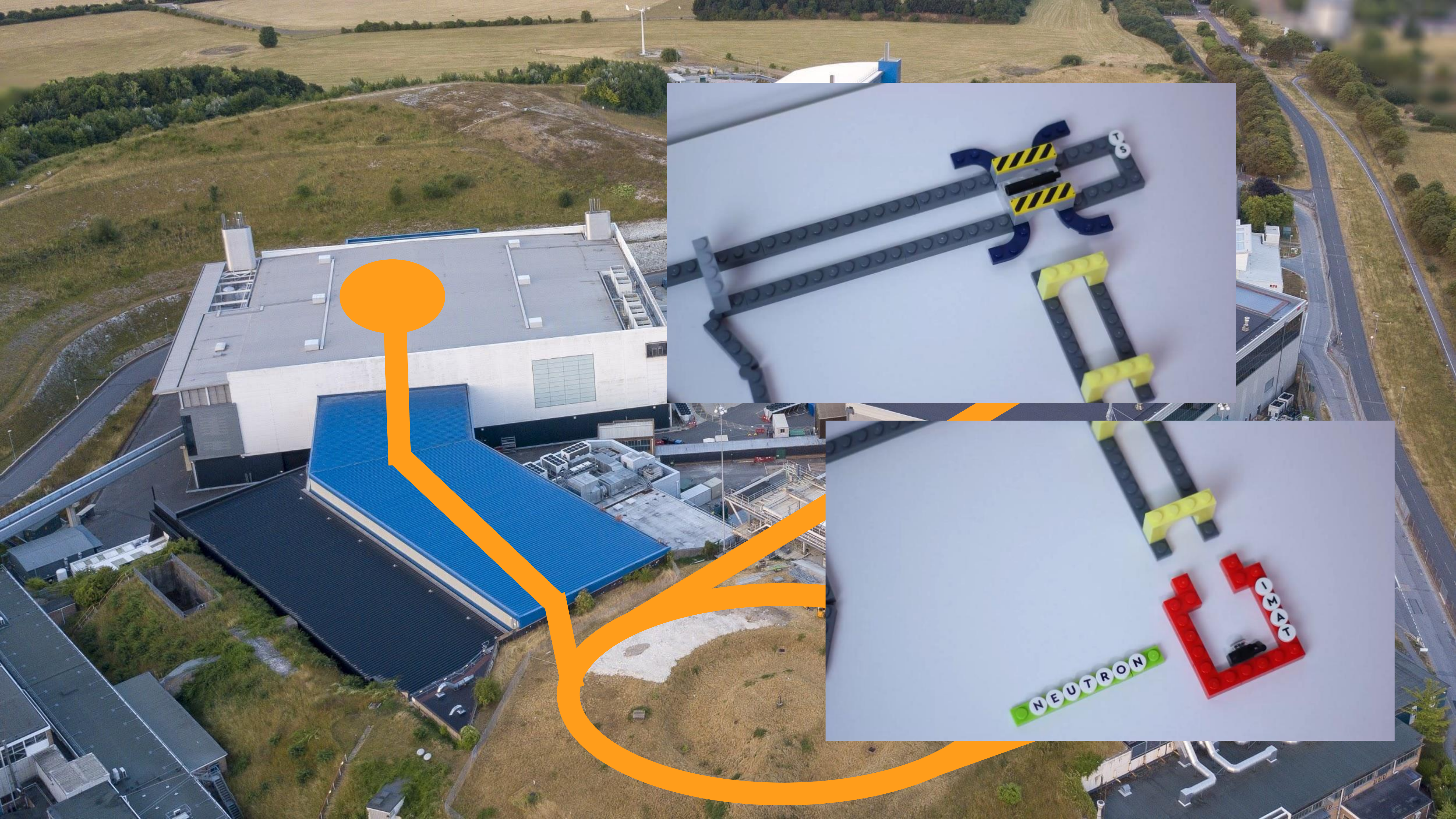
Outline

1 **ISIS Neutron & Muon Source**

2 **IMAT Instrument and Dilution Refrigerator modification**

3 **Discussion of Results**

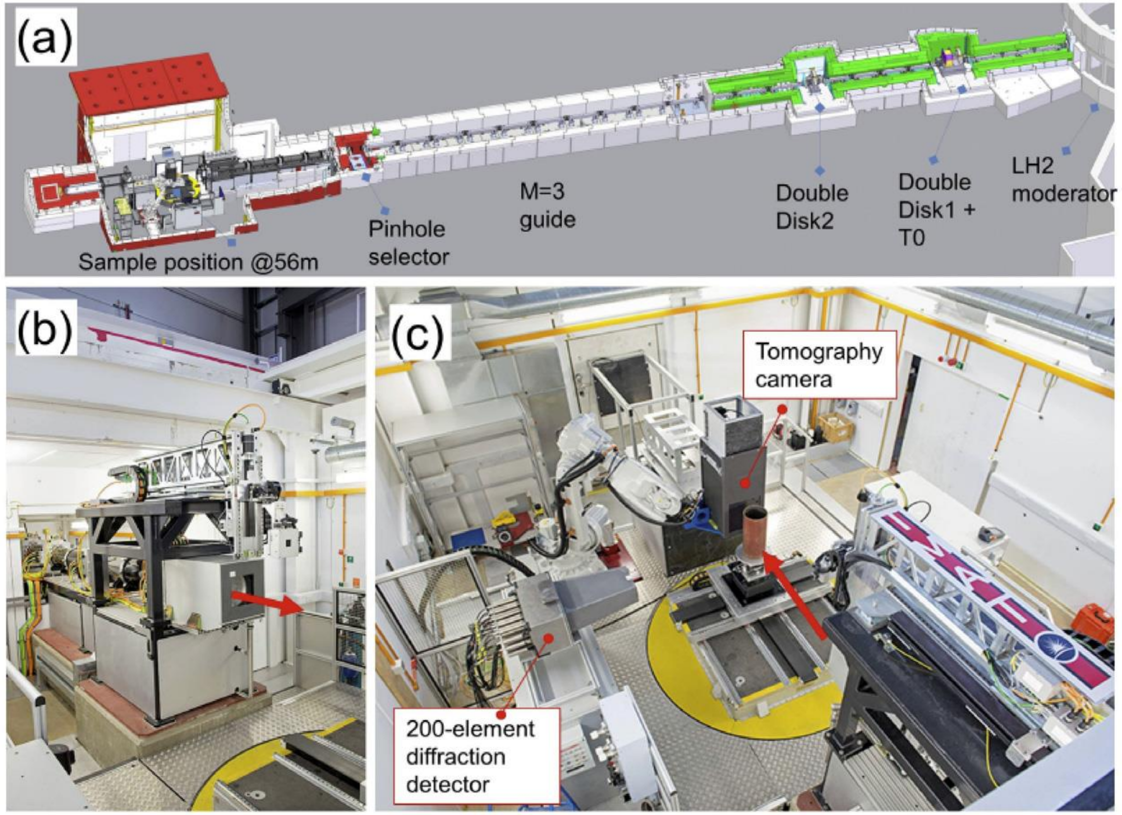

SYNC
T S
ELECTRON

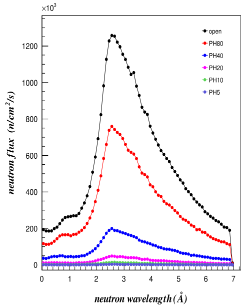

TS
IMAT
NEUTRON

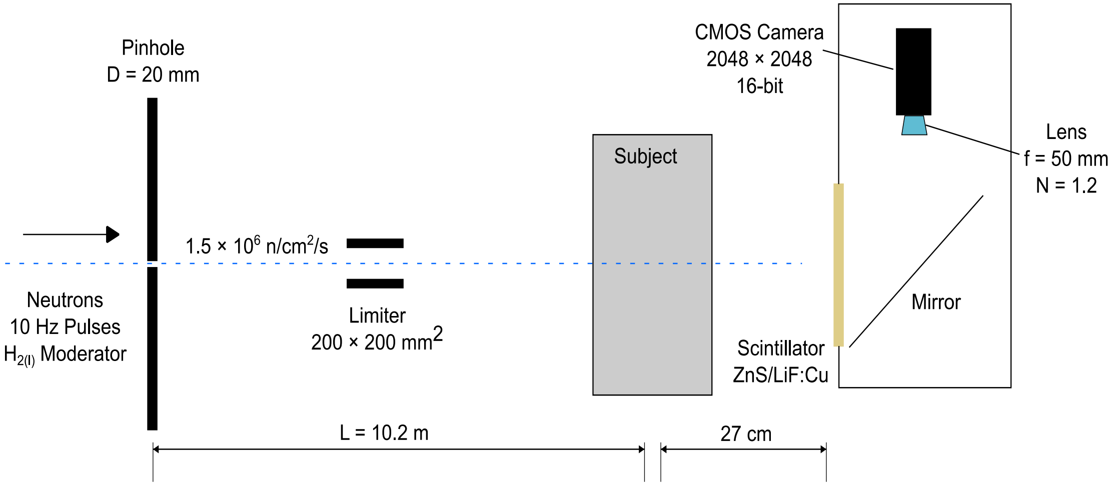
The IMAT Instrument

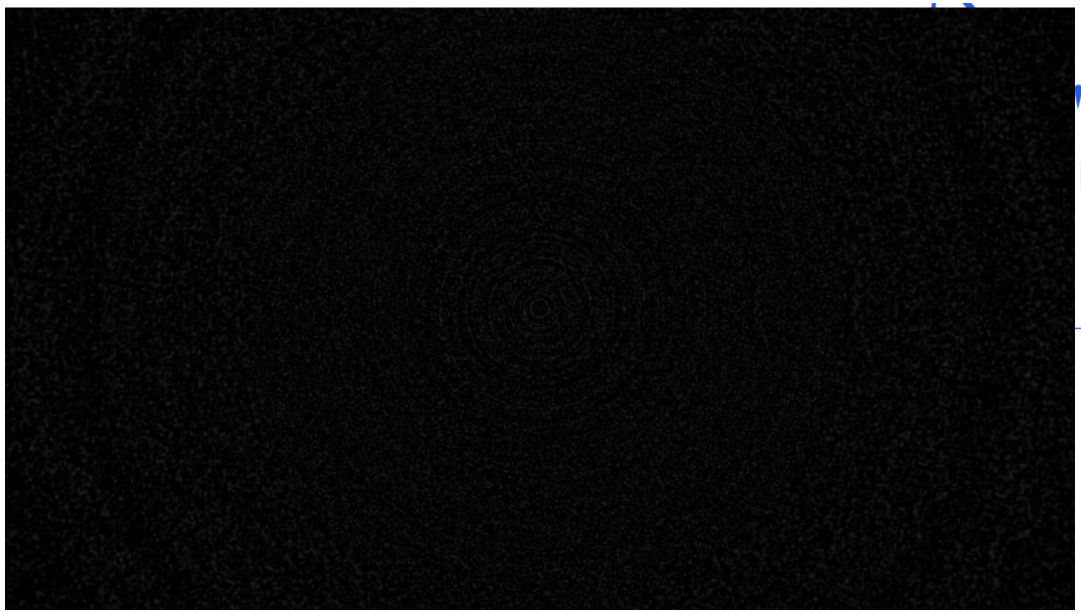
- IMAT (Imaging and Materials Science & Engineering) is a neutron imaging and diffraction instrument for a broad range of materials sciences.

- IMAT currently provides neutron radiography, neutron tomography, and energy-resolved neutron imaging.

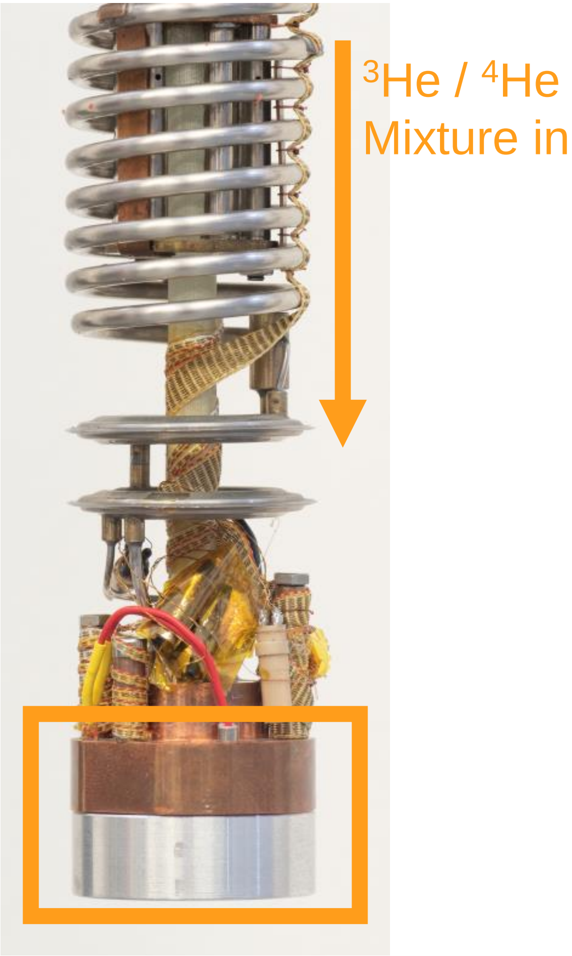


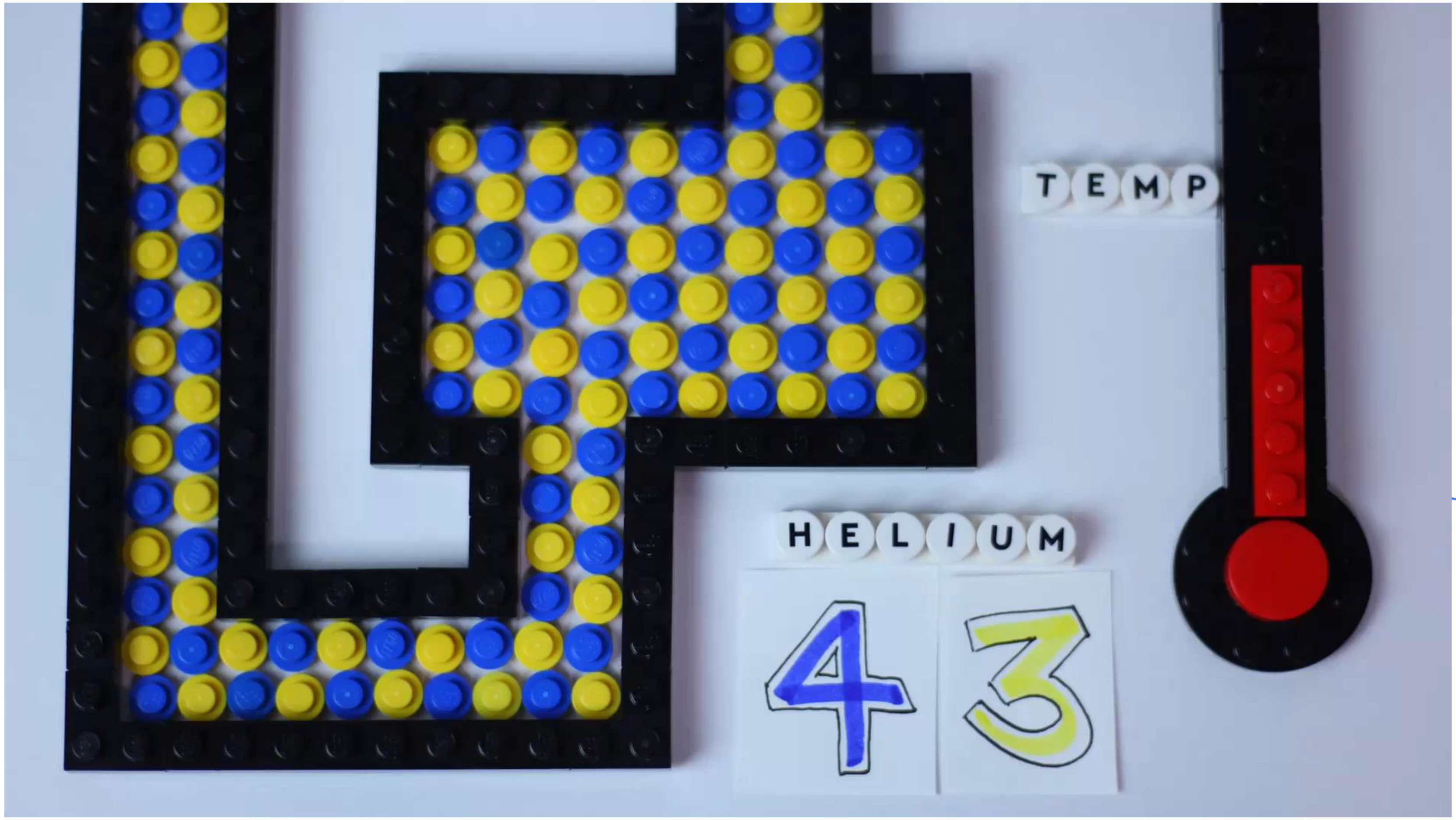






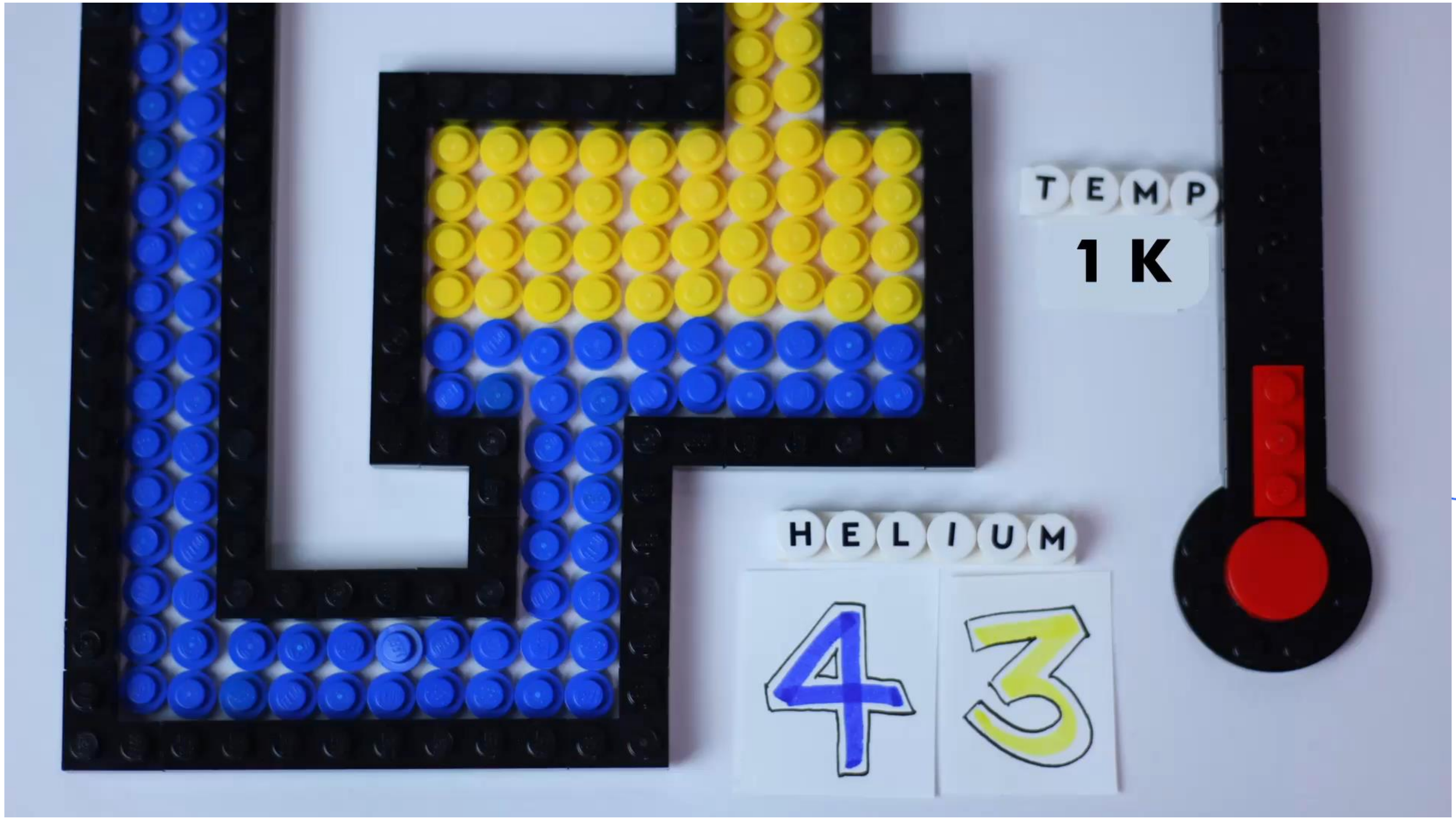
Dilution Refrigeration





Dilution Refrigeration

Circulate
^{3}He / ^{4}He
Mixture



Application of Neutron Imaging Techniques to Dilution Refrigeration

For a beam of neutrons passing through some substance, the reduction in beam intensity I from absorption by nuclei is given by

$$\frac{I}{I_0} = \exp(-\Sigma t),$$

For material thickness t and macroscopic neutron absorption cross section

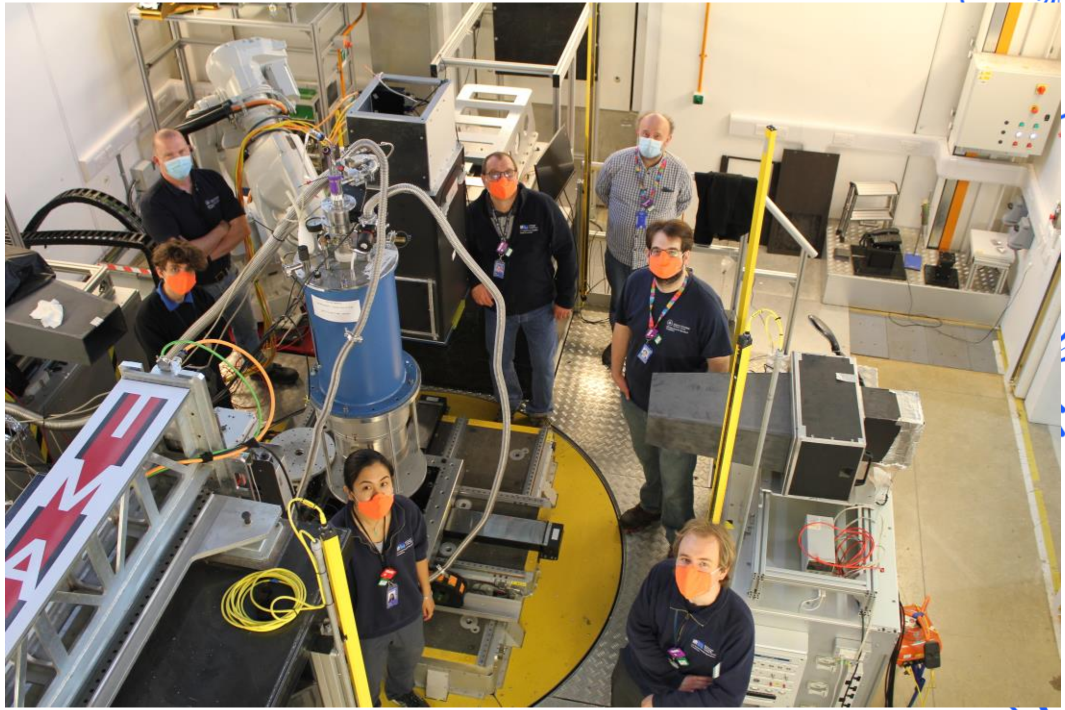
$$\Sigma = \sum_i N_i \sigma_i,$$

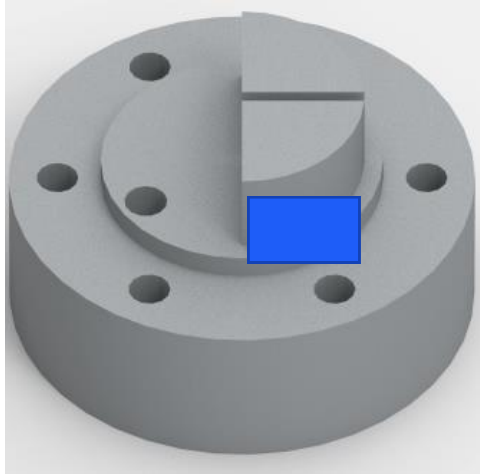
For N_i the number density of atom i with microscopic absorption cross section σ_i.

For 1.798 Å neutrons incident on helium atoms

$$^4He\ \sigma_4 = 0.0075\ \mathrm{b},$$
$$^3He\ \sigma_3 = 5333\ \mathrm{b}.$$

So, neutron absorption is a proxy for ^{3}He number density (^{4}He is essentially transparent).



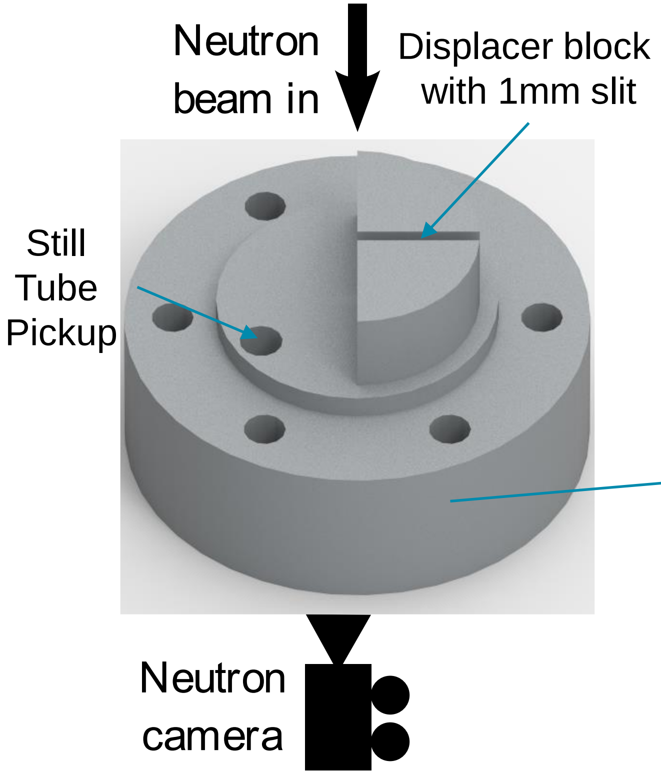


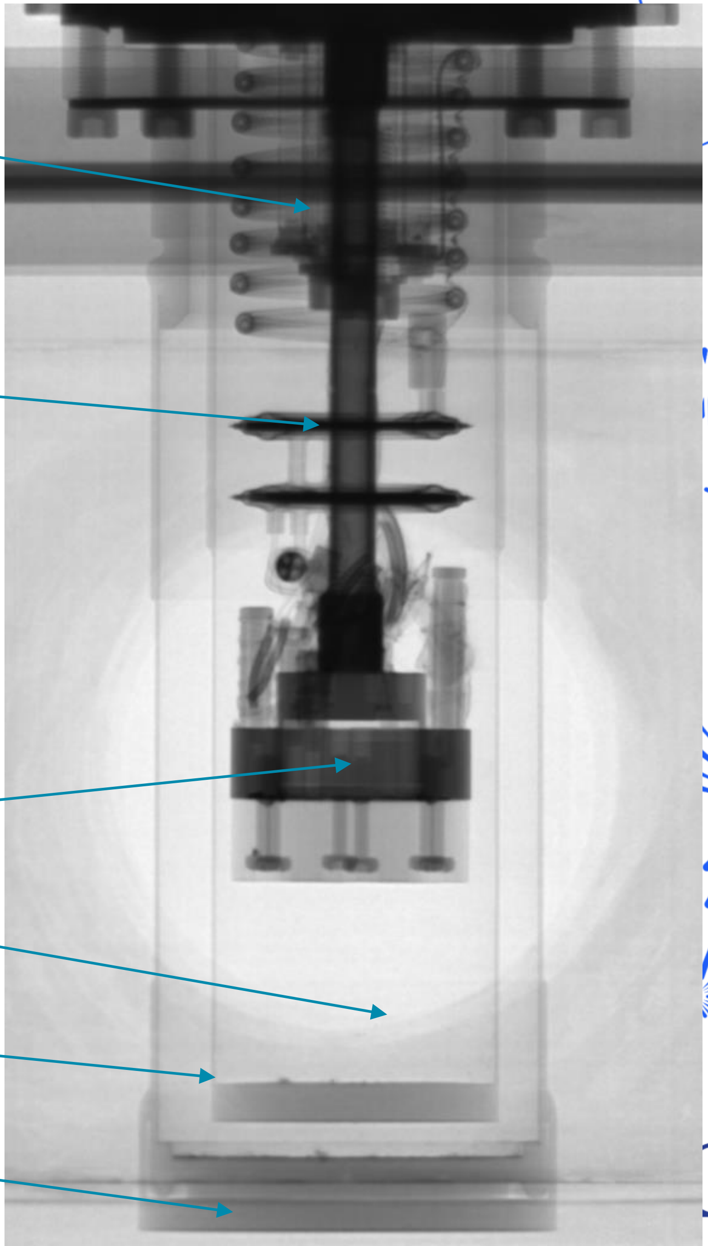
Even 6.6% ^{3}He is too absorbing, we need to reduce path length through the mixture.

Neutron transmission through 1 mm of:
Pure ^{3}He = 10^{-6}%
6.6% ^{3}He/^{4}He mixture = 12.5%

Application of Neutron Imaging Techniques to Dilution Refrigeration

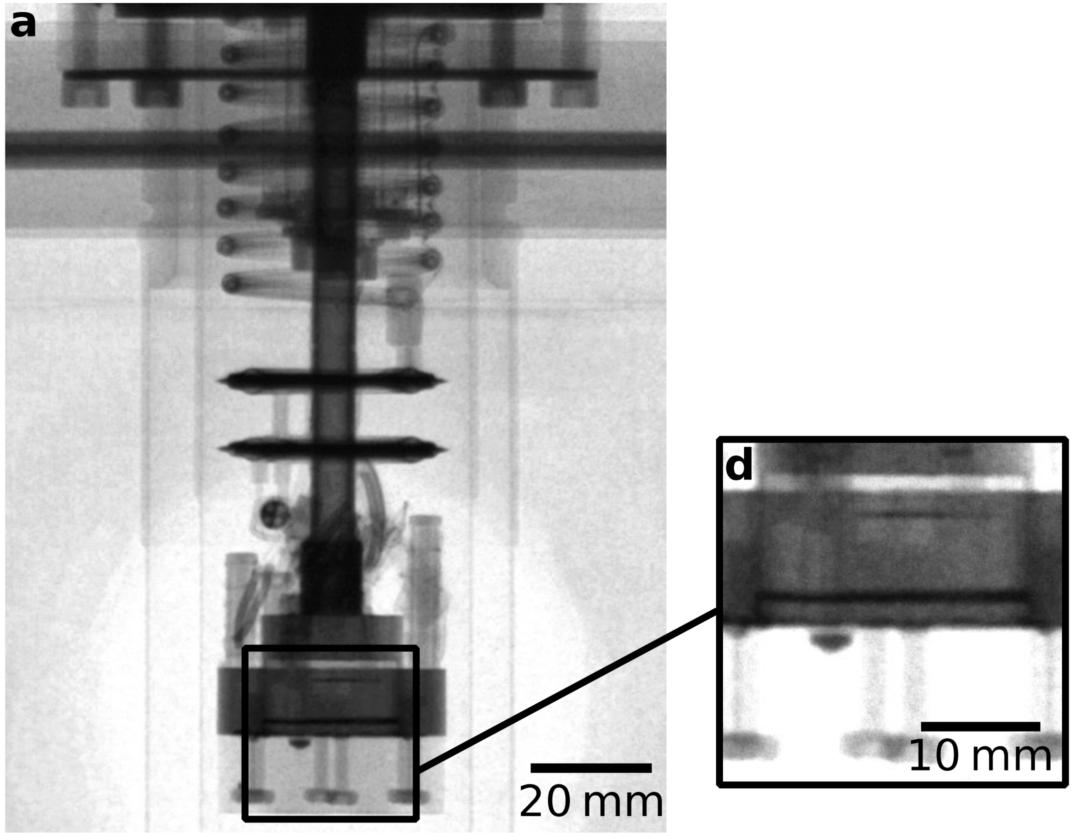
- Source operating at 10Hz
- Liquid H2 Moderator T <20K
- Peak λ=2.6Å
- 4mp sCMOS camera
- ZnS/LiF:Cu scintillator
- 0.53mm spatial resolution
- Dark & light frame corrections + beam intensity normalisation





10s exposure x50

Results – Mixture Condensation – 10K to 1K

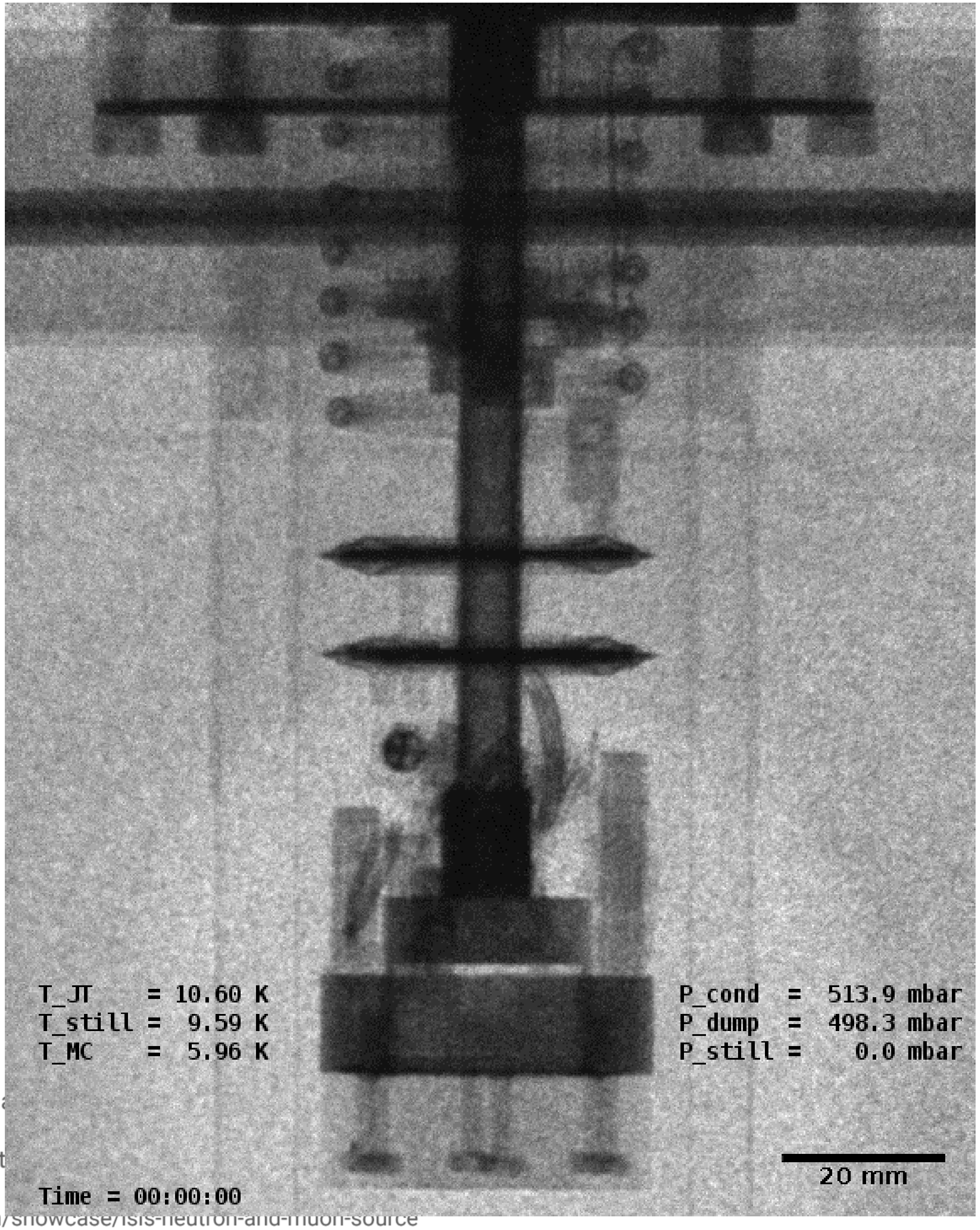


- 10s exposures
- Insert images contrast adjusted

*Figure 2. Neutron images of the DR during mixture condensation. Panels **a-c** show the DR progressing through the condensation process. Note the small layers of mixture in the still tube pickup, at the MC base, and on top of the displacer in the inset **d**, which is a zoomed-in and contrast-adjusted region of **a**, captured shortly after the initiation of condensation. Panel **e** is a zoomed-in and contract-adjusted region of **b** showing mixture flowing through the inner tube of the continuous heat exchanger. A video of the full condensation process is in supplementary material S1.*

Results –
Mixture Condensation Video

- Fridge mixture condensation from 10K
- 1s exposure, ~1000 frames, frames stacked, compressed & factor 2 binning



```
T_JT    = 10.60 K
T_still =  9.59 K
T_MC    =  5.96 K
```

```
P_cond  =  513.9 mbar
P_dump  =  498.3 mbar
P_still =    0.0 mbar
```

```
Time = 00:00:00
```

Results – Operating Dilution Fridge – Temperature Ramp

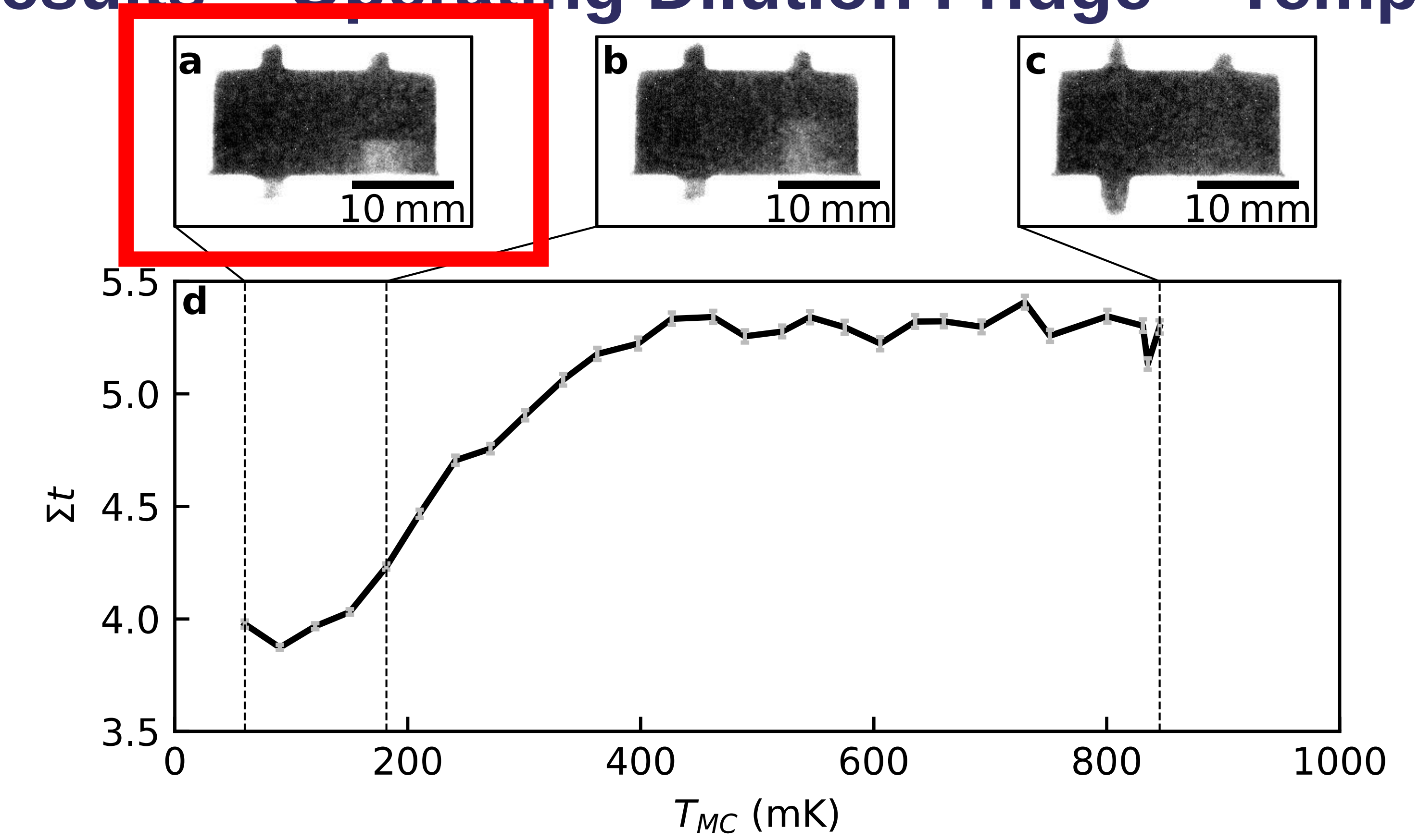
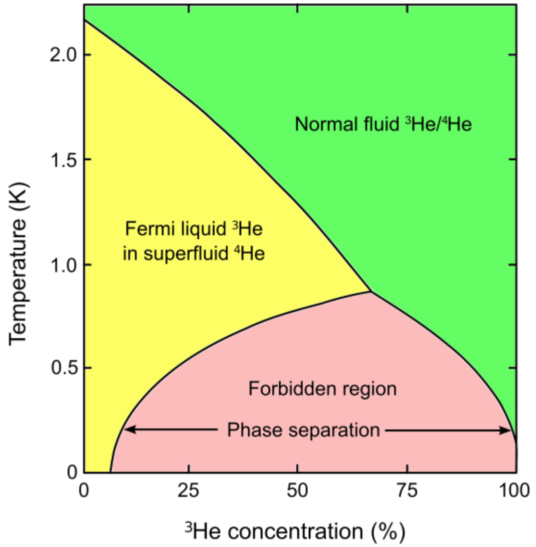


Figure 3. Changing ^{3}He concentration with temperature in the mixing chamber of the DR. Panels ***a-c*** *show the mixing chamber of the DR at the three indicated temperatures in panel* ***d****, which is a plot of the quantity Σt against mixing chamber temperature. Note how Σt increases with temperature, indicating further attenuation of the neutron beam and hence greater ^{3}He concentration in the dilute phase, and the phase boundary moves upwards, as expected because of the resulting redistribution of the mixture. The standard deviation of each data point in panel* ***d*** *is shown by the vertical error bars. Panel* ***e*** *shows the quantity Σt throughout the entire dilution refrigerator circuit for a mixing chamber temperature of 60 mK and indicates the measurement position for the graph in* ***d*** *with the black square at the lower-right of the mixing chamber.*

- 15x 30sec exposures at each T
- Calibrate zero transmission as ^{3}He rich section of MC
- Subtract signal through empty fridge
- Σt is a measure of neutron attenuation

$$I = I_0 e^{-\Sigma_m t_m}$$

$$\Sigma_m = \frac{\rho_m \sigma_3}{m_3} c_3,$$



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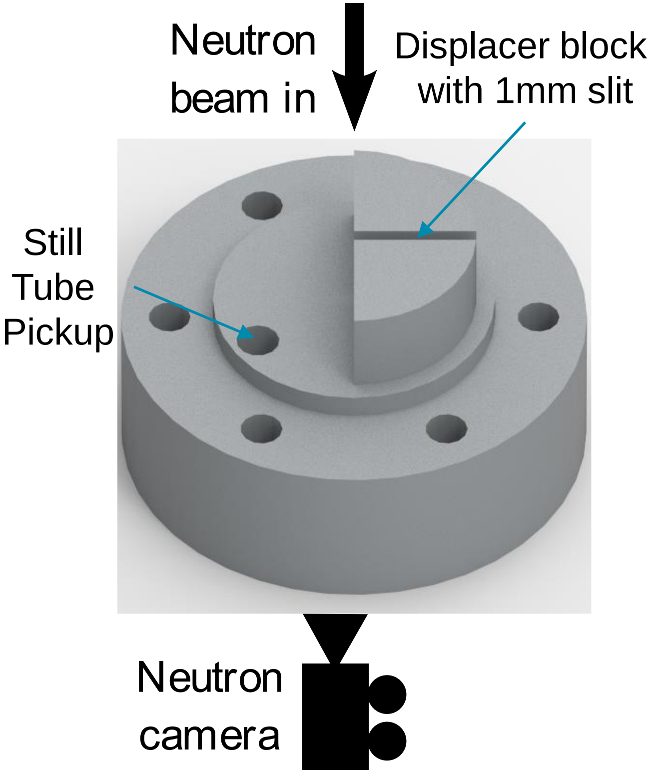
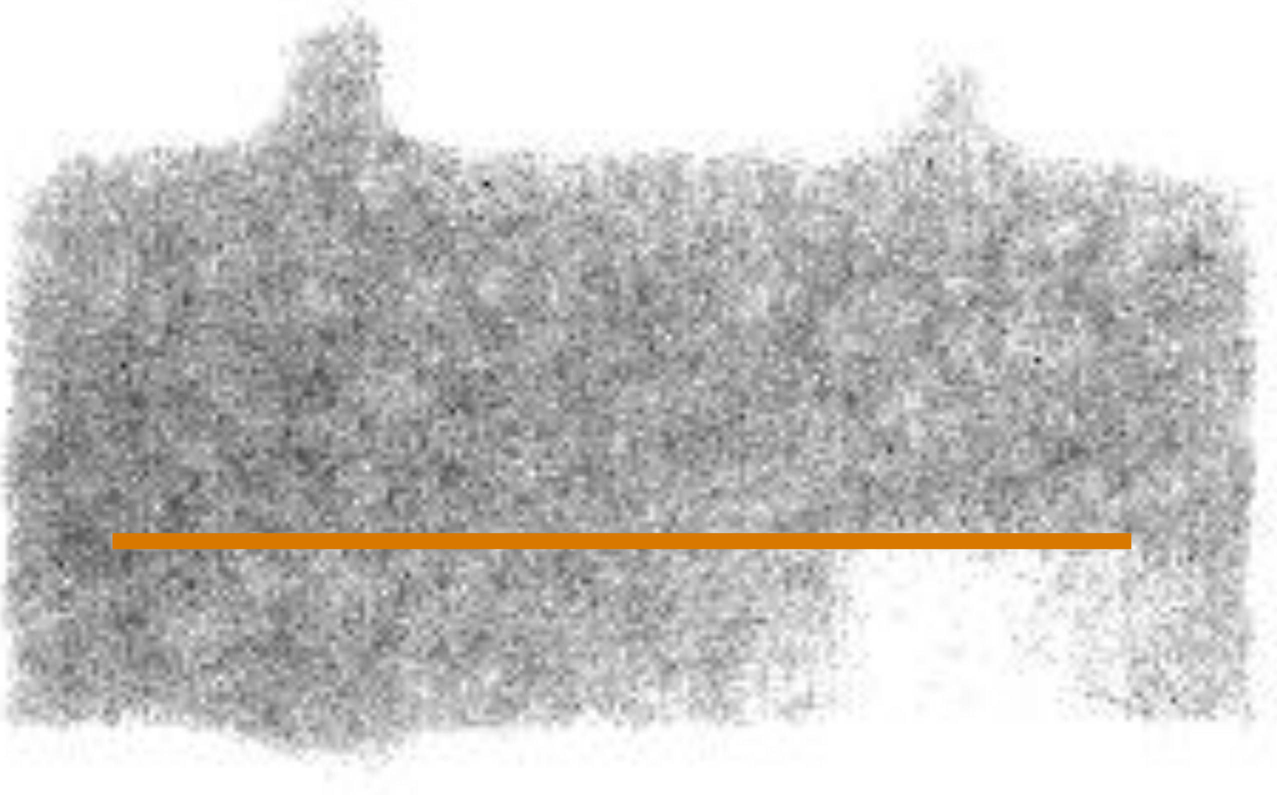
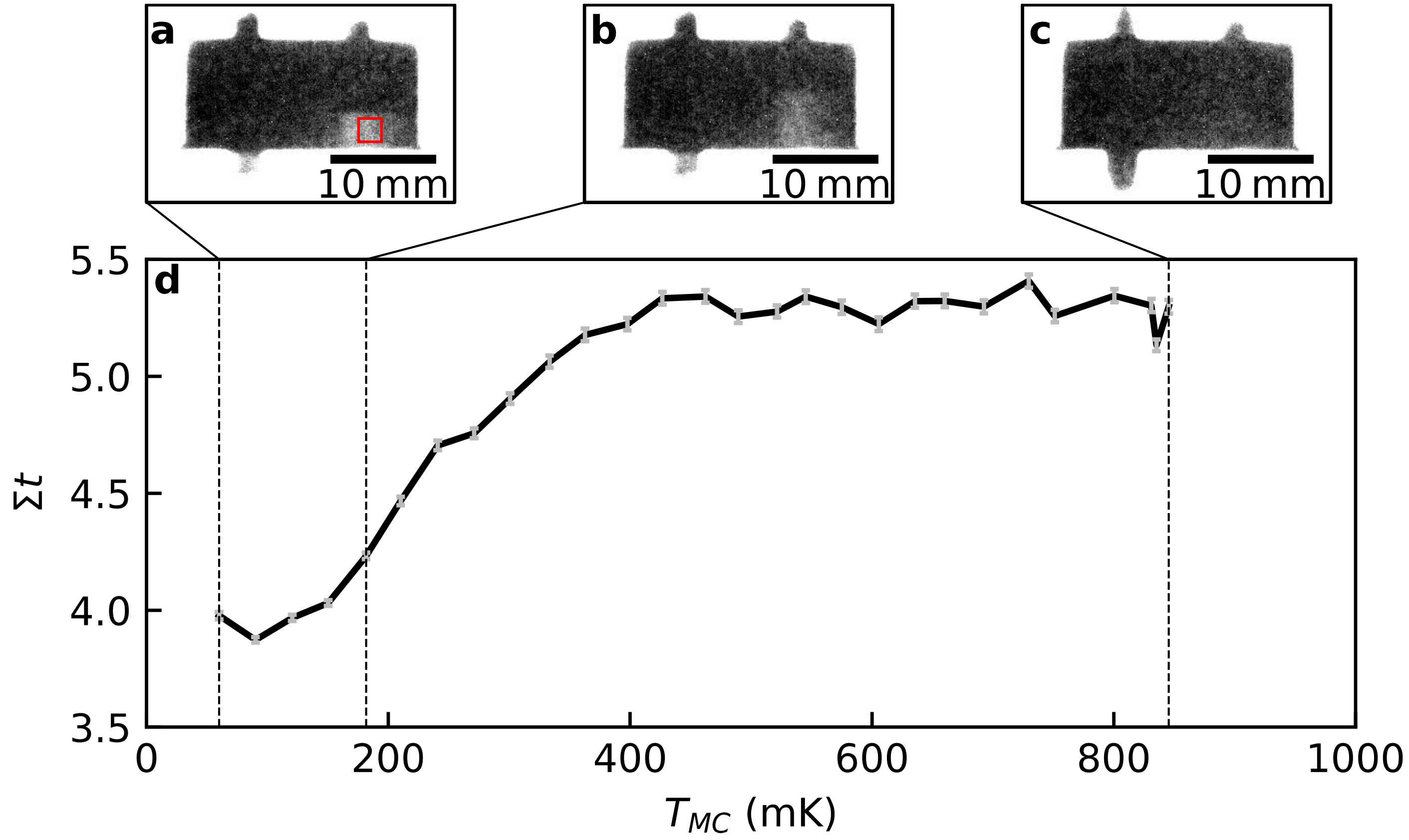
Results – Operating Dilution Fridge – Phase Boundary





Results – Operating Dilution Fridge – Temperature Ramp



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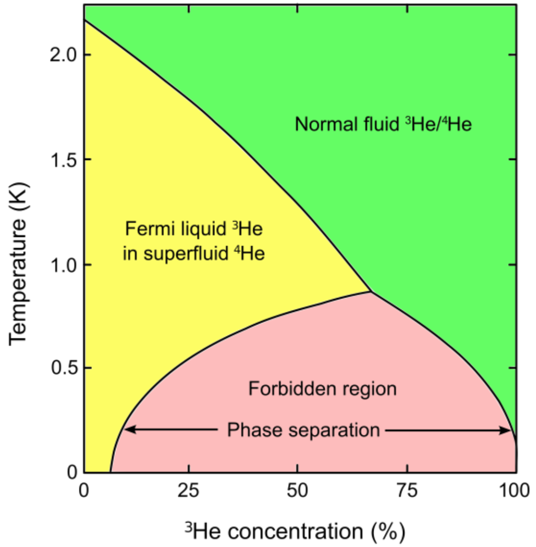
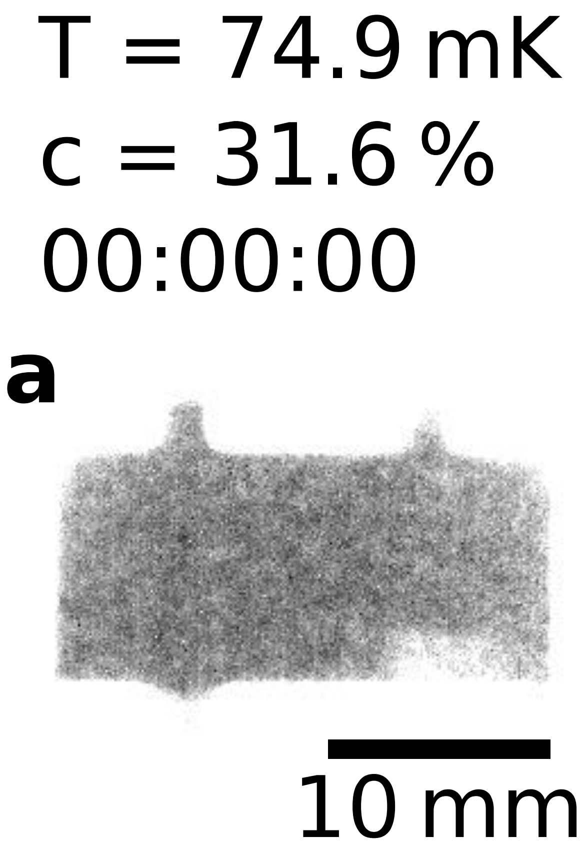
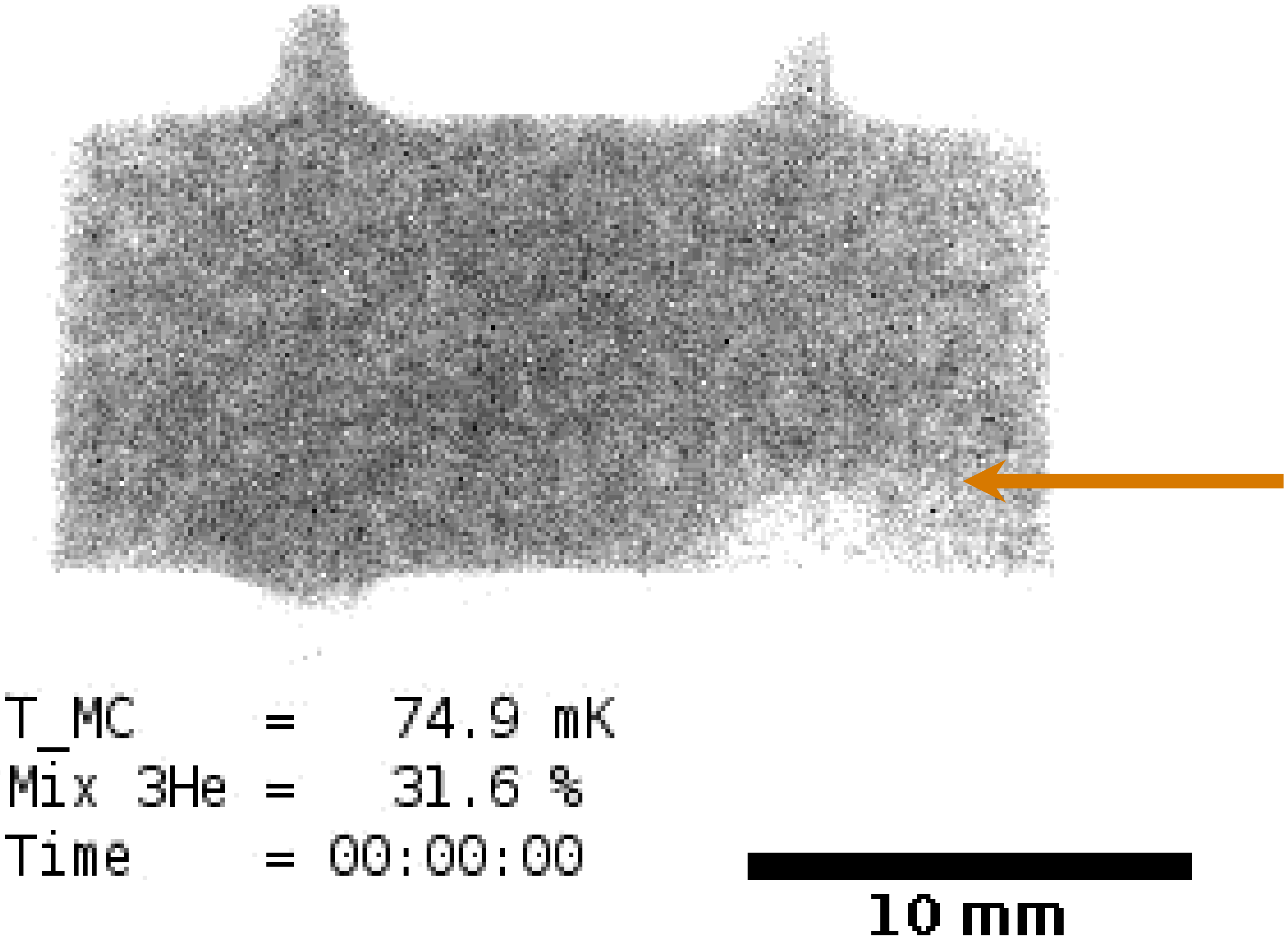


Results – Single Shot of Dilution Refrigerator



- 10s exposures
- 10 exposure moving average

Results – Single Shot of Dilution Refrigerator



Results: Incorrect Mixture

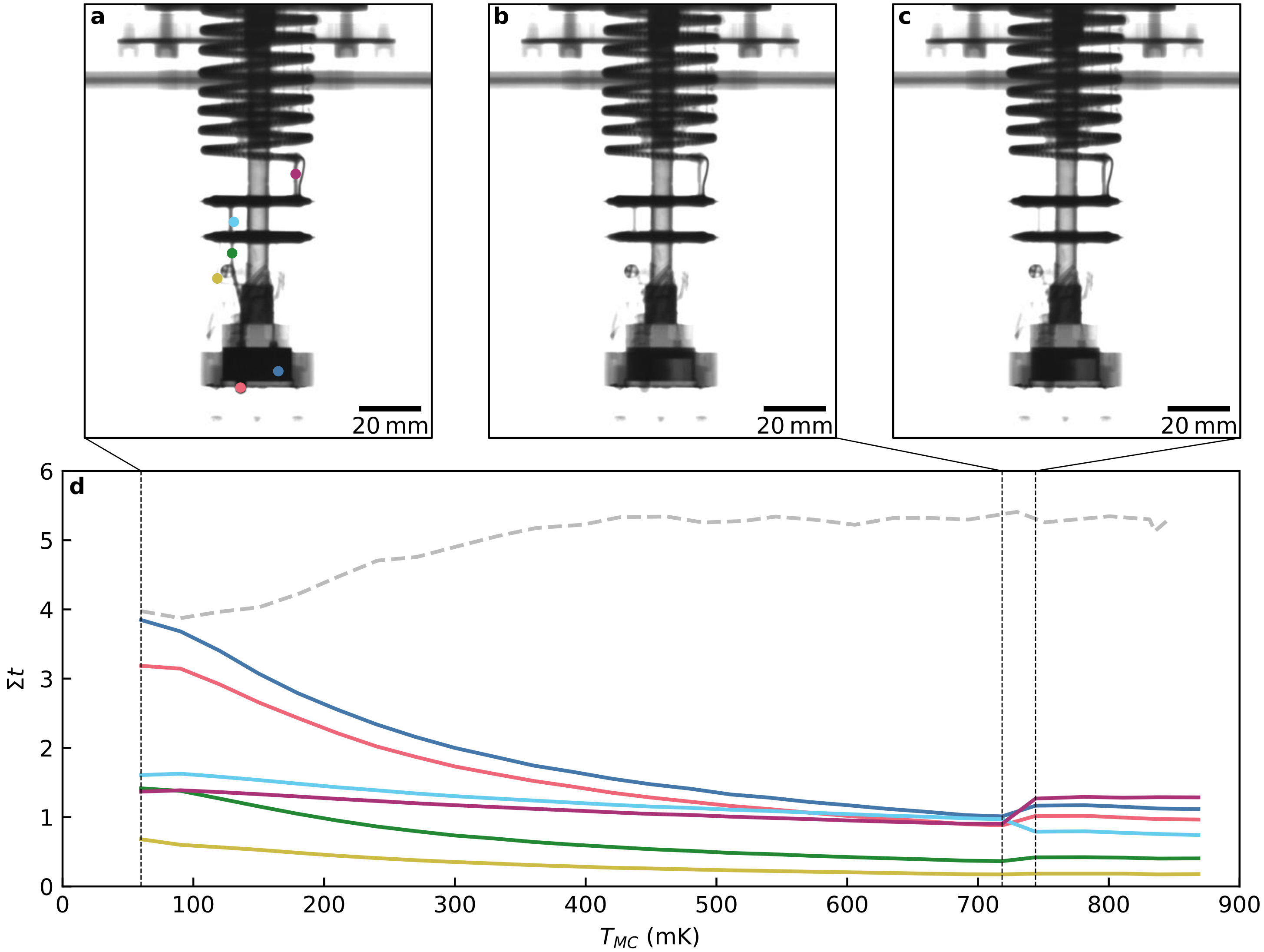
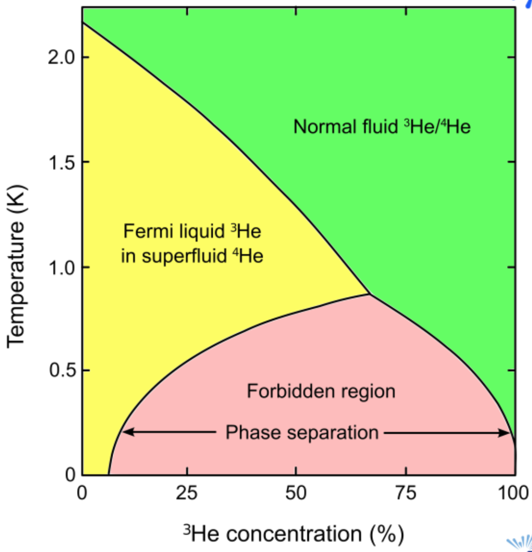


*Figure 5. Operation of the dilution refrigerator with insufficient ^{3}He (28%). Panels **a-c** show neutron images of the operating DR, contrast adjusted in order to more clearly show the mixture. Panel **d** shows the quantity Σt for neutrons penetrating several parts of the DR, as indicated by the coloured circles in panel **a**. Note that the neutron absorption in the mixing chamber initially reduces with increasing temperature, the opposite of what would normally be expected (see the dashed grey line, reproduced from the Fig. 3 data), and then changes suddenly around 750 mK.*

$$I = I_0 e^{-\Sigma_m t_m}$$

$$\Sigma_m = \frac{\rho_m \sigma_3}{m_3} c_3,$$



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Conclusions

- Using neutron radiography we are able to clearly see internal processes occurring at millikelvin temperatures inside an operational dilution refrigerator.
- Changes in ^{3}He concentration can be measured via analysis of neutron transmission. Quantitative analysis should be possible in the future.
- This will prove useful for designers of unusual dilution refrigerators, technicians, and educators alike.

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OPEN **Neutron imaging of an operational dilution refrigerator**

C. R. Lawson, A. T. Jones, W. Kockelmann, S. J. Horney & O. Kirichek

The invention of the ^{3}He/^{4}He dilution refrigerator opened a new chapter in experimental ultra-low temperature physics. Dilution refrigerators became essential for providing ultra-low temperature environments for nuclear demagnetisation experiments, superconducting-qubit quantum processors and highly sensitive bolometers used in fundamental physics experiments. Development of dilution refrigeration technology requires thorough understanding of the quantum mechanical processes that take place in liquid helium at ultra-low temperatures. For decades the quantum fluids research community provided valuable information to engineers and designers involved in the development of advanced dilution refrigerators. However, the lack of methods that allow the measurement of physical parameters of liquid helium during the operation of a dilution refrigerator was hindering development of the technology. Here we show direct imaging of an operational dilution refrigerator using neutron radiography. This allows direct observation of the dilution process in ^{3}He/^{4}He mixtures and opens an opportunity for direct measurement of the ^{3}He concentration. We observe the refrigerator behaviour in different regimes, such as continuous circulation and single shot, and show that our method allows investigation of various failure modes. Our results demonstrate that neutron imaging applied to the study of dilution refrigeration processes can provide essential information for developers of ultra-low temperature systems. We expect that neutron imaging will become instrumental in the research and development of advanced dilution refrigerators.

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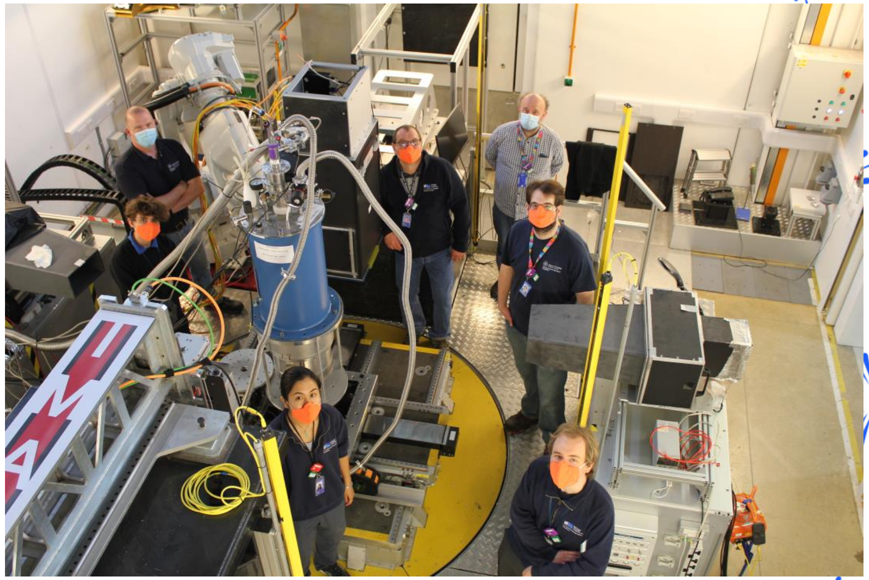
Sci Rep **12,** 1130 (2022).

Thanks

Oleg Kirichek
Winfried Kockelmann
Alex Jones
Sasha Horney
Paul Frodsham



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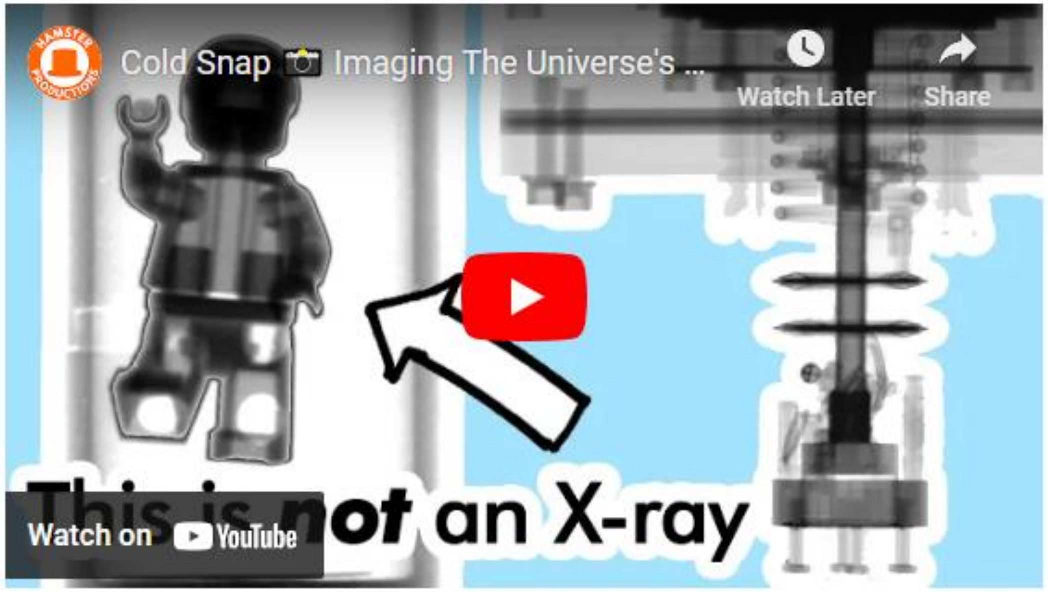

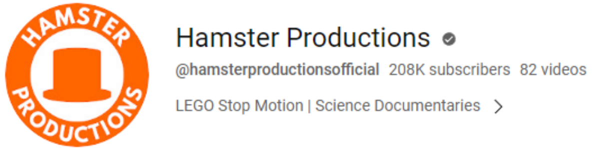

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