

# **LAr Purification Studies and a Novel Recirculation System**

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# Talk Outline

**Why argon purity matters**

**Effects of various contaminations on gas argon scintillation light**

**Purification chemicals - How do they work?**

**Summary of a set of experiments performed to determine the efficiency of the purification chemicals**

**The 40L LAr Liverpool Cell**

- Construction**
- A novel recirculation pump and LAr purification**
- Effects of O<sub>2</sub> contamination on LAr scintillation light**

# Why Purity Matters

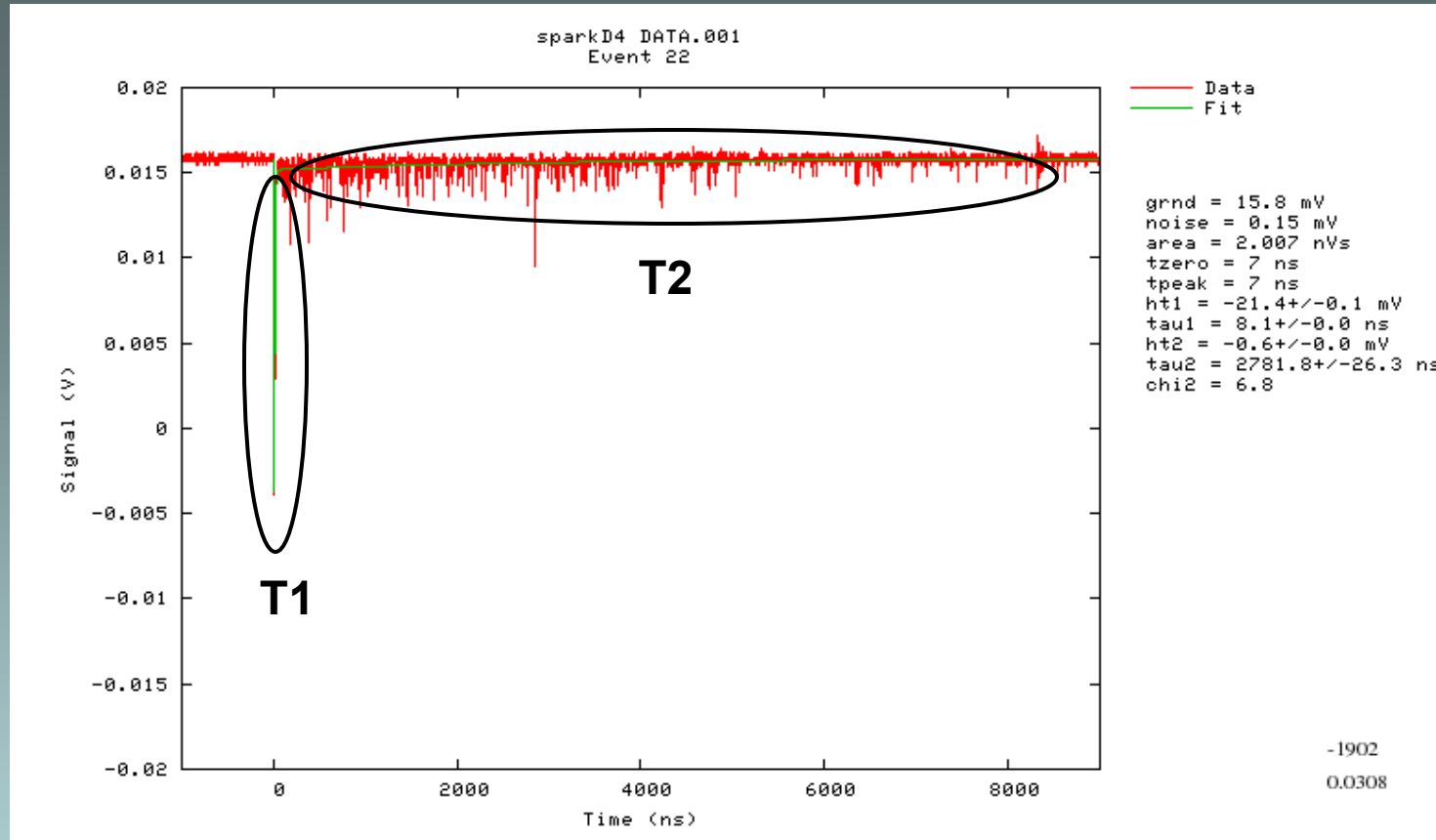
The ionised electrons produced in a liquid argon detector such as the ArDM must be able to drift over a distance longer than a metre, without substantial capture by electronegative impurities ( i.e  $O_2, H_2O, CO_2, N_2$ )

Typically better than 1 ppb purity is required

Purest argon gas in the market (N6 cylinder) contains 1 ppm impurities

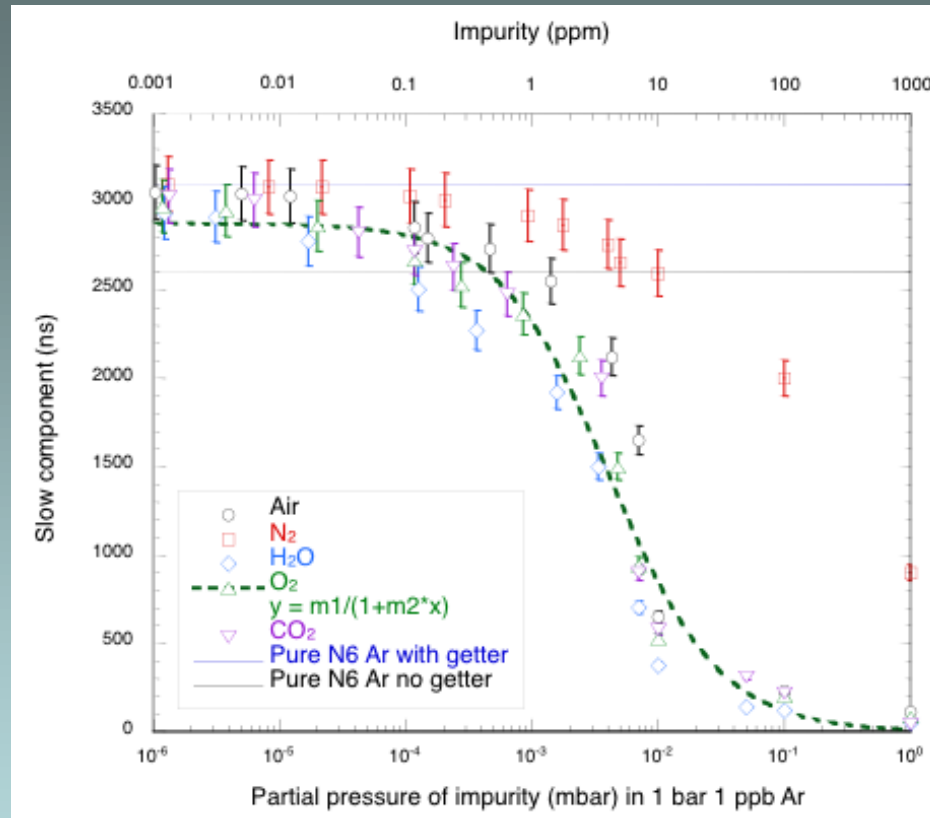
Therefore purification cartridges and recirculation systems have to be employed

# Argon Gas Scintillation

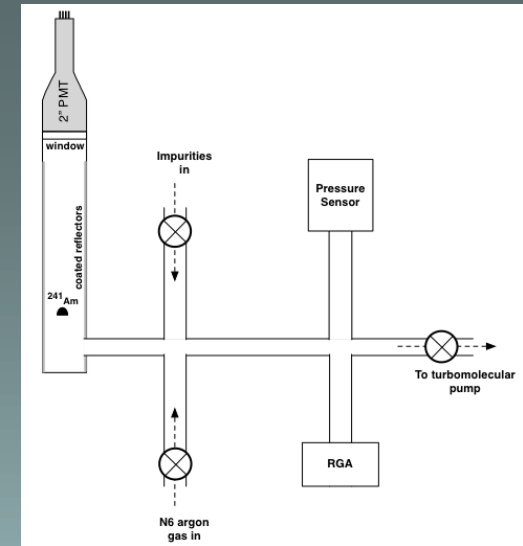


Scintillation light from argon has two distinct decay times - a slow component, T2 (triplet), and a fast component, T1 (singlet). The slow component, T2, can be used as a measure of Argon purity. The purest gas argon has a T2 of about 3200 ns (J. W. Keto *et al*, PRL, 1974).

# Effect of various impurities on GAr scintillation light



K. Mavrokoridis, PhD Thesis



--H<sub>2</sub>O was found to be very marginally the worst impurity followed by CO<sub>2</sub> and O<sub>2</sub>.

--N<sub>2</sub> was found to be the most benign impurity within argon gas.

--Birks law type function fit  
 $y = m1/(1+m2*x)$ ;  $m1 = 2878 \text{ ns}$ ,  $m2 = 0.24$

**Correlation of the slow component decay time to partial pressure of impurities within 1 bar ppb argon gas**

# The Argon Purification Chemicals (a)

## Molecular Sieves



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graph TD; A[Molecular Sieves] --> B[Size/Steric Exclusion]; A --> C[Thermodynamic Selectivity]
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### Size/Steric Exclusion

Molecules small enough to pass through the pores are adsorbed while larger molecules are not

Molecular sieves can be graded to capture molecules with sizes between 3 Å and 10 Å

### Thermodynamic Selectivity

A measure of preferential absorption of certain molecules over others when all molecules can enter the pores.

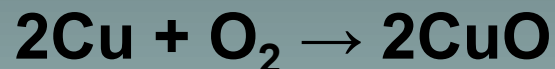
Is achievable because the adsorption of a particular gas is favourable over another on the accessible cationic sites within the crystal.

**Molecular sieves can be used to purify argon based on the size/steric exclusion and thermodynamic selectivity properties**

# The Argon Purification Chemicals (b)

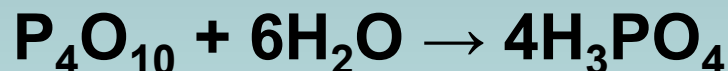
## Copper

Copper reacts strongly with oxygen forming copper oxide.



## Phosphorous pentoxide

$\text{P}_4\text{O}_{10}$  reacts exothermically very strongly with water producing orthophosphoric acid.

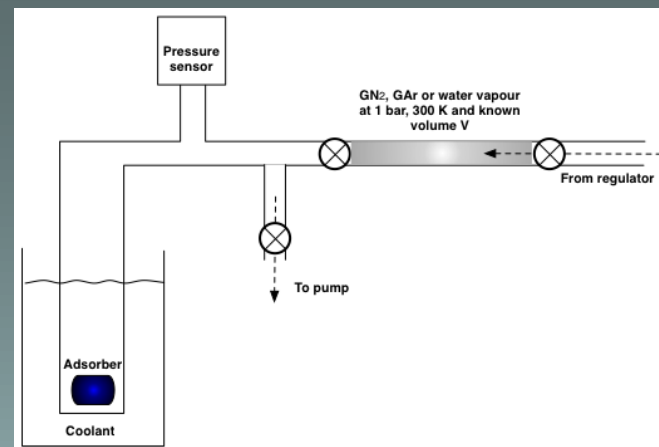


Hazardous upon skin contact and inhalation

# Adsorption of H<sub>2</sub>O, N<sub>2</sub>, Ar

Measurements of the enthalpy of adsorption, total capacity, and effective surface area of interaction, for argon gas, nitrogen gas and water vapour on a range of molecular sieves and anhydrous complexes were performed.

The BET (Brunauer, Emmett, Teller) isotherm describes adsorption and provides a route to measuring both the average enthalpy of adsorption and the effective surface area of the adsorber (e.g. activated charcoal)

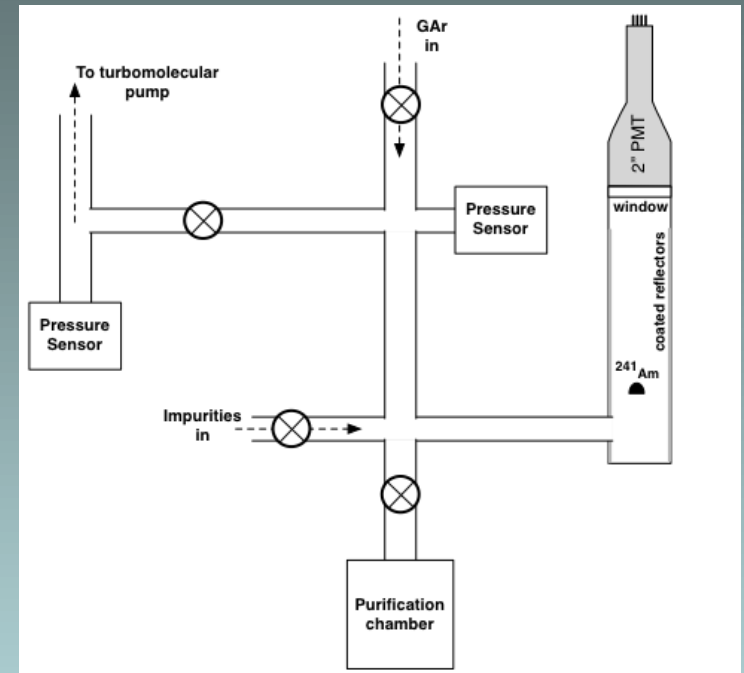
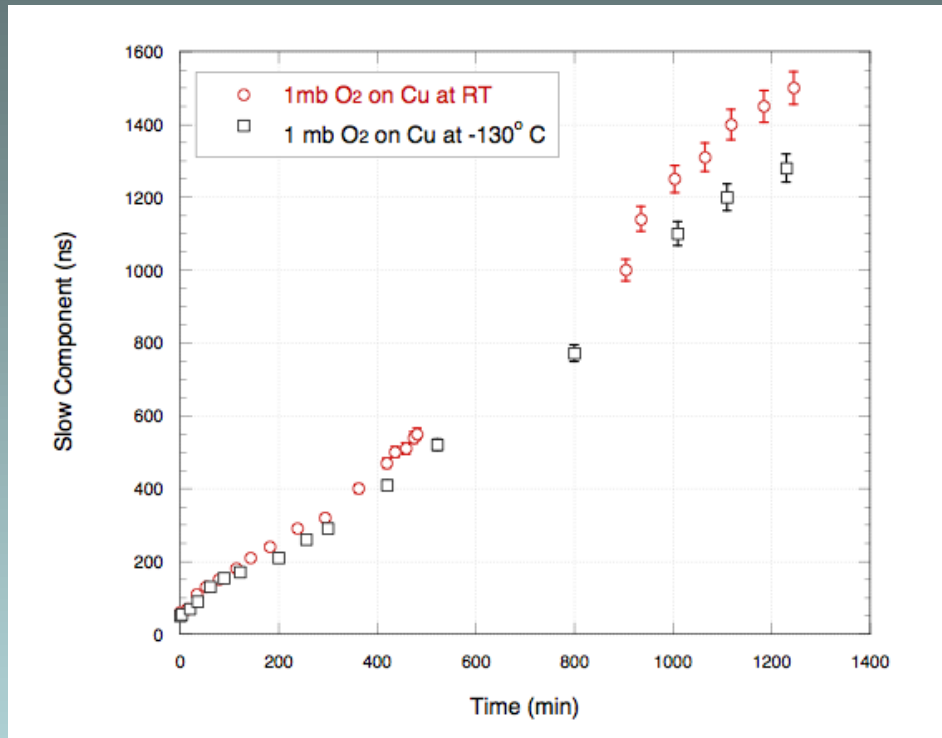


**Adsorption results for the BET isothermal experiment**

Combination	Enthalpy of adsorption (kJ/mol)	Surface area (m <sup>2</sup> )	Maximum capacity (%wt)
N <sub>2</sub> and activated carbon	9.1	65836	37
Ar and activated carbon	11.1	62451	42
N <sub>2</sub> and 3A Mol. sieve	10.9	26264	23
Ar and 3A Mol. sieve	11.4	29477	27
N <sub>2</sub> and activated alumina	10.2	17330	13
Ar and activated alumina	11.1	16309	16
H <sub>2</sub> O and calcium sulphate	48.4	4520	6
H <sub>2</sub> O and 3A Mol. sieve	46.3	22785	14
H <sub>2</sub> O and cobalt chloride	45.0	3275	3
H <sub>2</sub> O and magnesium sulphate	45.1	3799	4

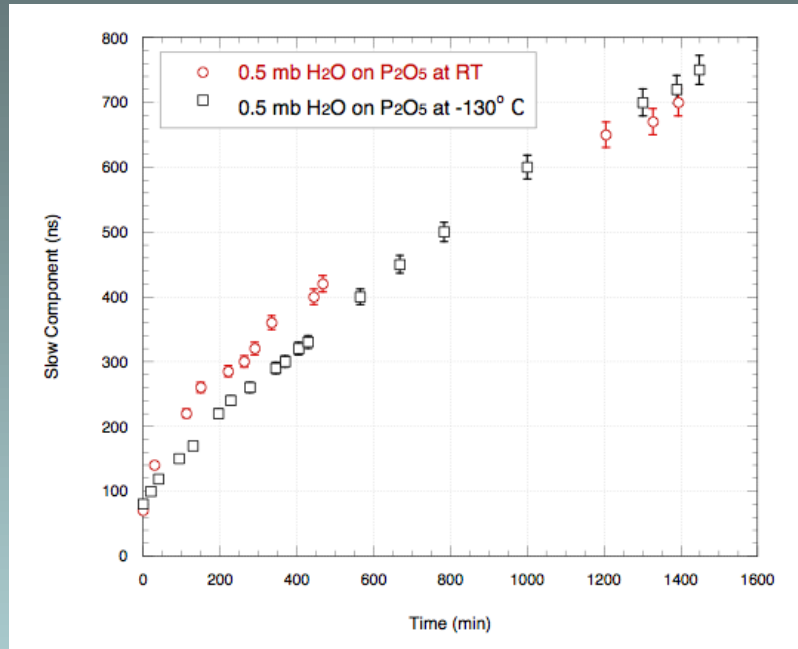


# Efficiency of Cu and $P_4O_{10}$ at removing $O_2$ and $H_2O$

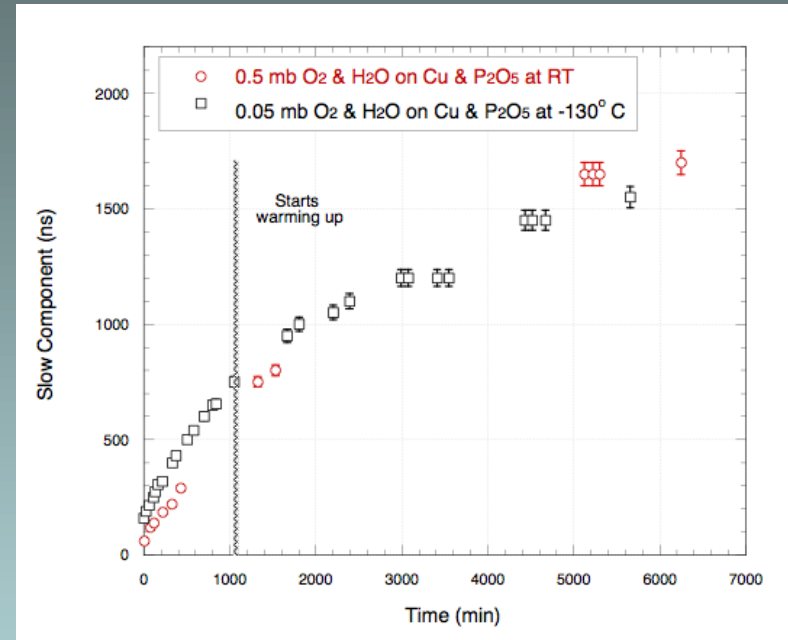


Efficiency of copper at room temperature and -130 °C at removing 1 mbar  $O_2$  partial pressure impurity in 1 bar N6 argon gas

# Efficiency of Cu and $P_4O_{10}$ at removing $O_2$ and $H_2O$



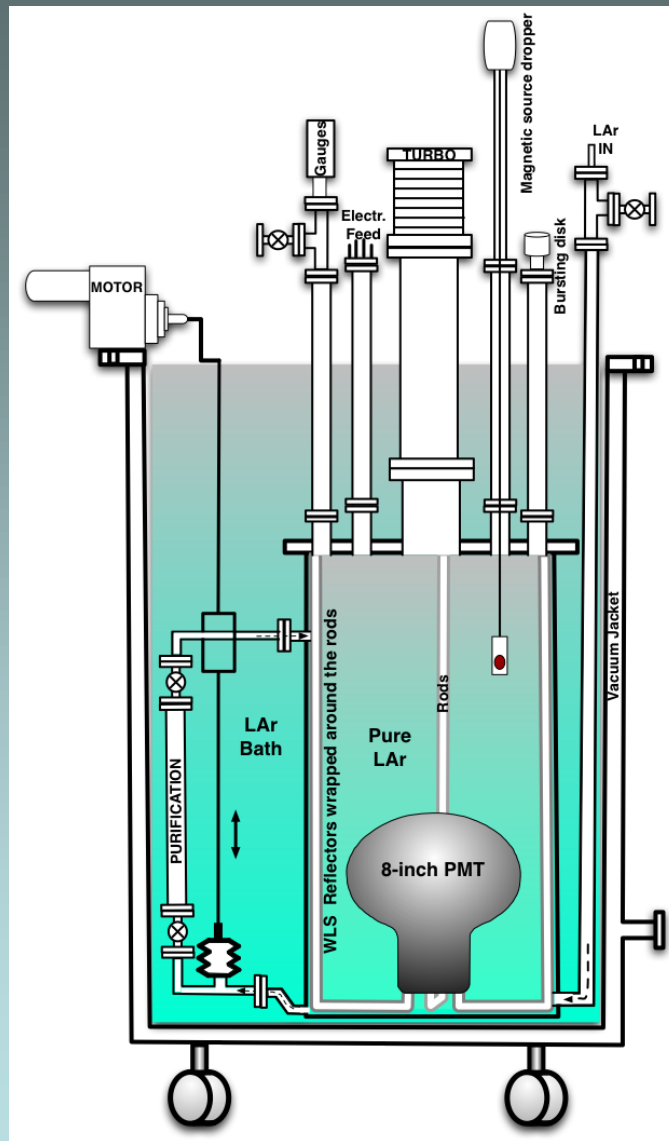
$P_4O_{10}$  at RT and  $-130^\circ C$  removing 0.5 mbar  $H_2O$  partial pressure impurity in 1bar N6 argon gas



Cu and  $P_4O_{10}$  mixture at RT and  $-130^\circ C$  removing 0.5 mbar and 0.05 mbar respectively  $O_2/H_2O$  partial pressure impurity in 1bar N6 argon gas

Within 30 hours the slow component decay time increased from 70 to 1650ns corresponding to  $\sim 3$  ppm  $O_2$  equivalent impurity

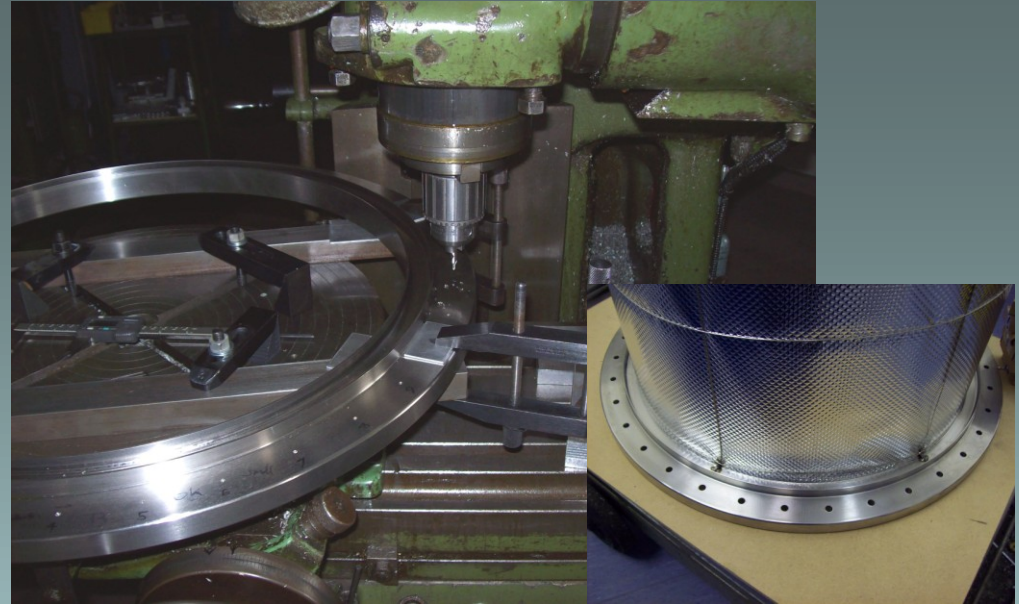
# The Liverpool LAr Cell -Schematic



**250 L LAr bath**  
**40 L LAr target**

- Bellow pump powered by an external geared motor
- Heat load loses  $\sim 115$  W
- Boiling rate  $\sim 12$  l/min
- Loosing about 20 L in 24h-  
assuming 10cm thick  
polyethylene lid

# Construction of the Liverpool LAr Cell



**Left: Rolling the stainless steel sheet**

**Right: Machining the top flange**

**Construction by Kevin McCormick**



# The Liverpool LAr Cell -Construction



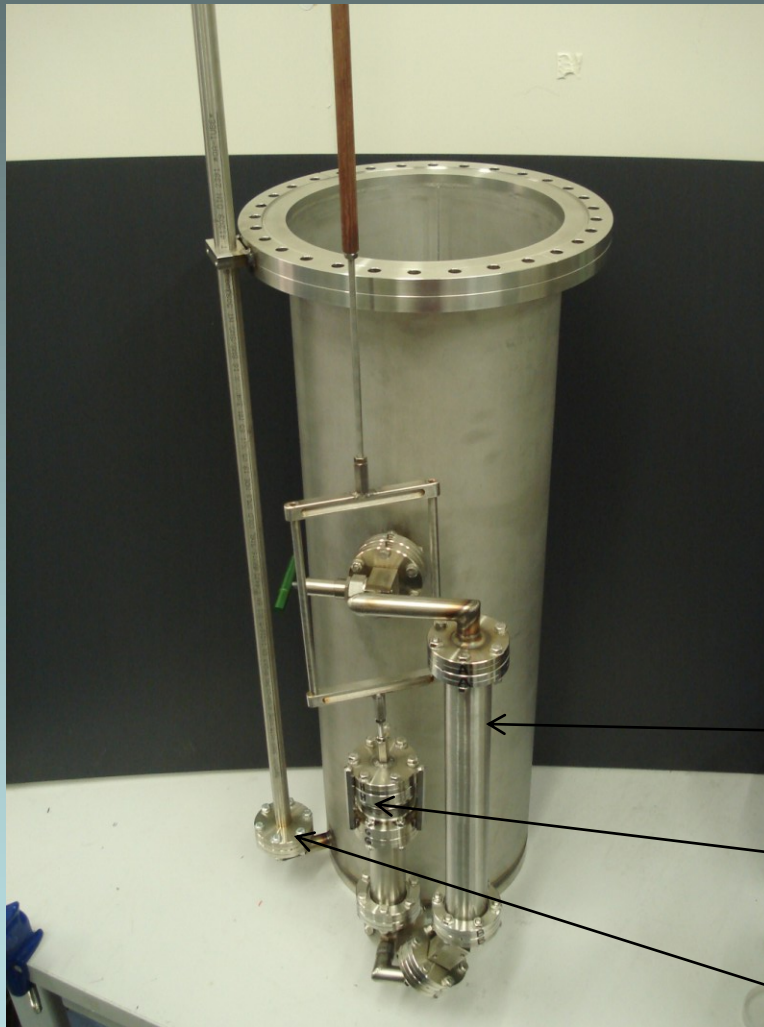
**Wrapped with Mylar reflector to minimize radiation losses**



**All the welding performed using argon gas to avoid oxidation and achieve UHV**



# The 40 L Target Vessel and Recirculation System



Development of a novel one way recirculation system using metal balls and a bellow

Scalable design

Purification Cartridge

Metal bellow

LAr feedthrough



# The Liverpool LAr Cell– The Purification Cartridge

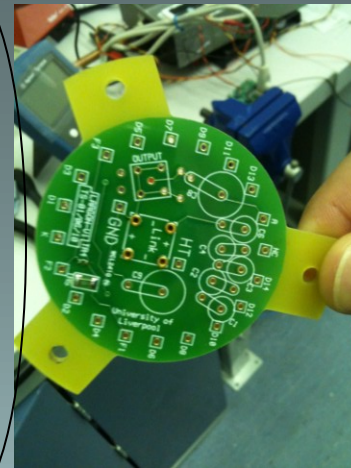
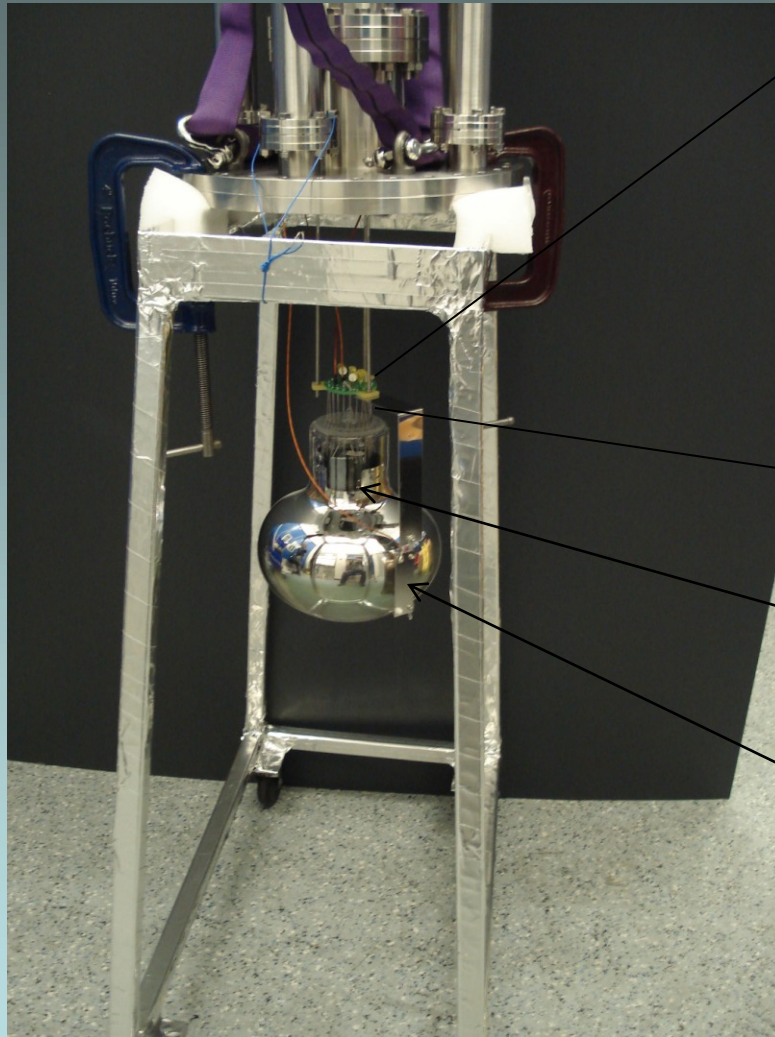


● 3A,4A,13X Molecular Sieves for removing,  $\text{N}_2, \text{H}_2\text{O}, \text{CO}_2$

● Copper for removing  $\text{O}_2$  ( $2\text{Cu} + \text{O}_2 \rightarrow 2\text{CuO}$ )

**Filling the purification cartridge within an argon bag in order to avoid reaction with air molecules**

# The Liverpool LAr Cell – Light Readout



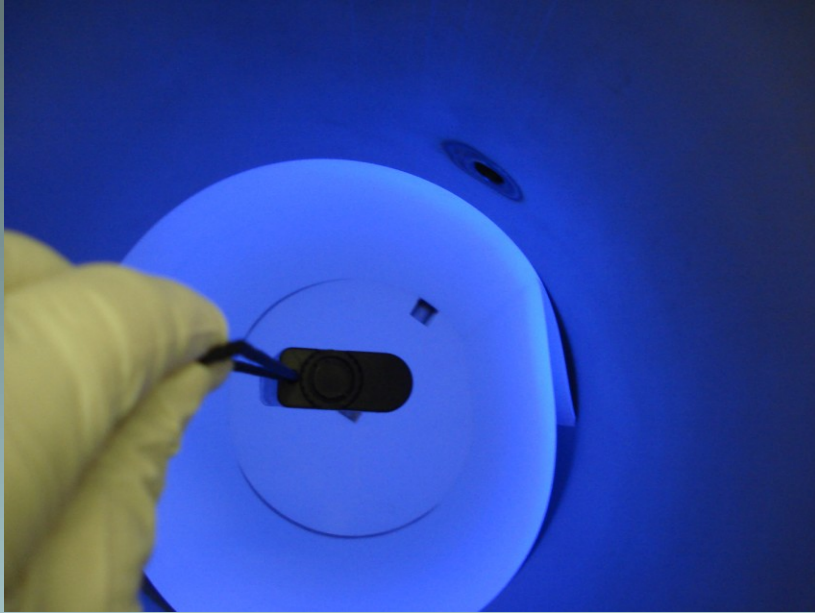
**Development of  
cryogenic voltage  
divider**

**Cryogenic 8-inch  
Hamamatsu R5912-02MOD  
PMT (~2500 GBP)**

**Capacitor LAr level meter**



# The Liverpool LAr Cell – The WLS Coated Reflectors



TPB coated 3M foil under a UV lamp

Shifts 128 nm  $\rightarrow$  430 nm

TPB coated PMT under a UV lamp



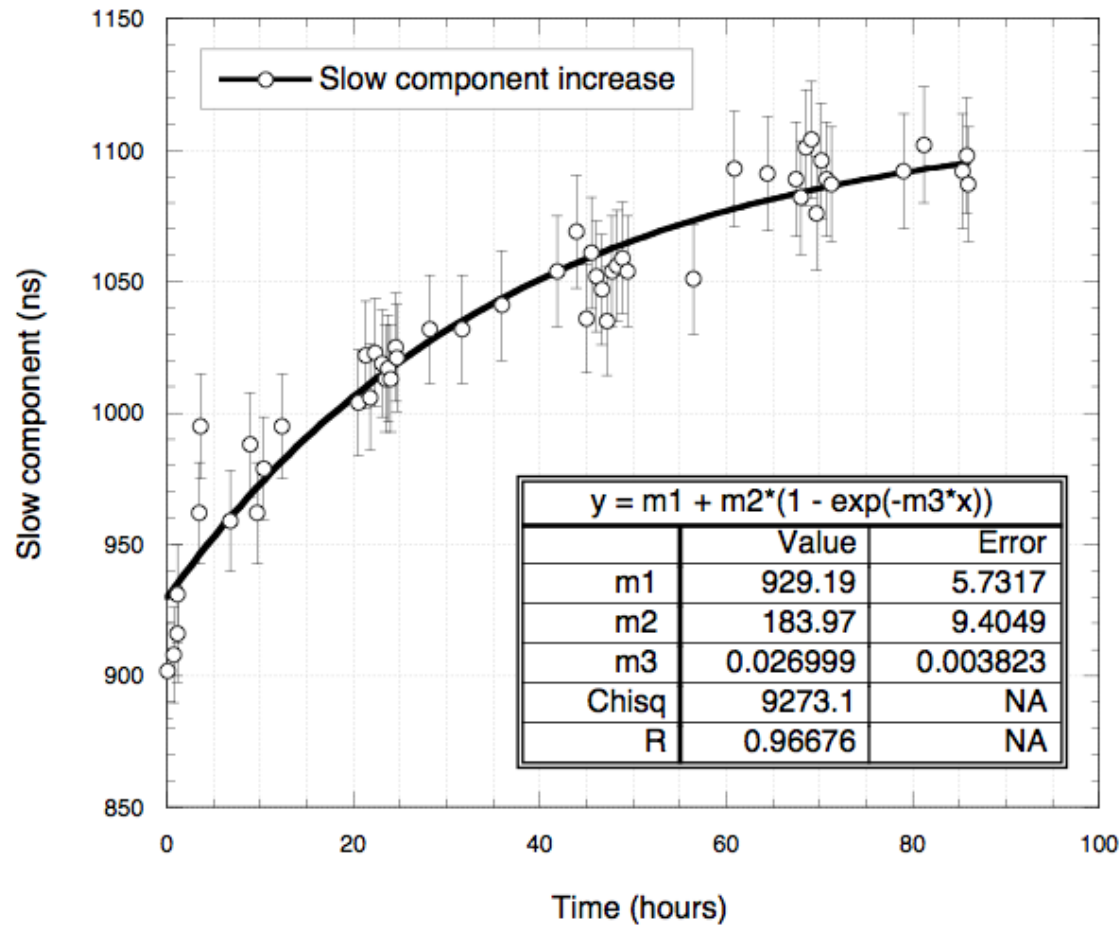
# The Liverpool LAr Cell- Assembled



**Closing the detector**



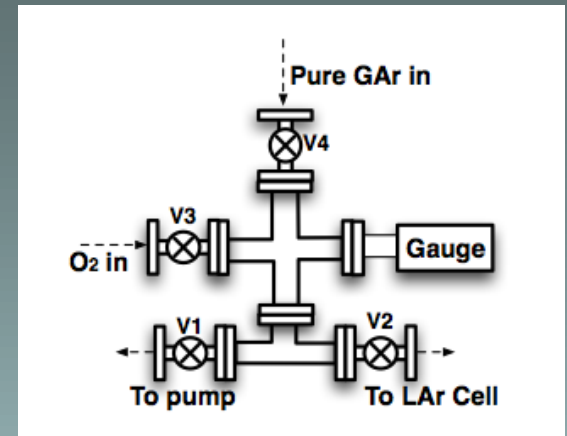
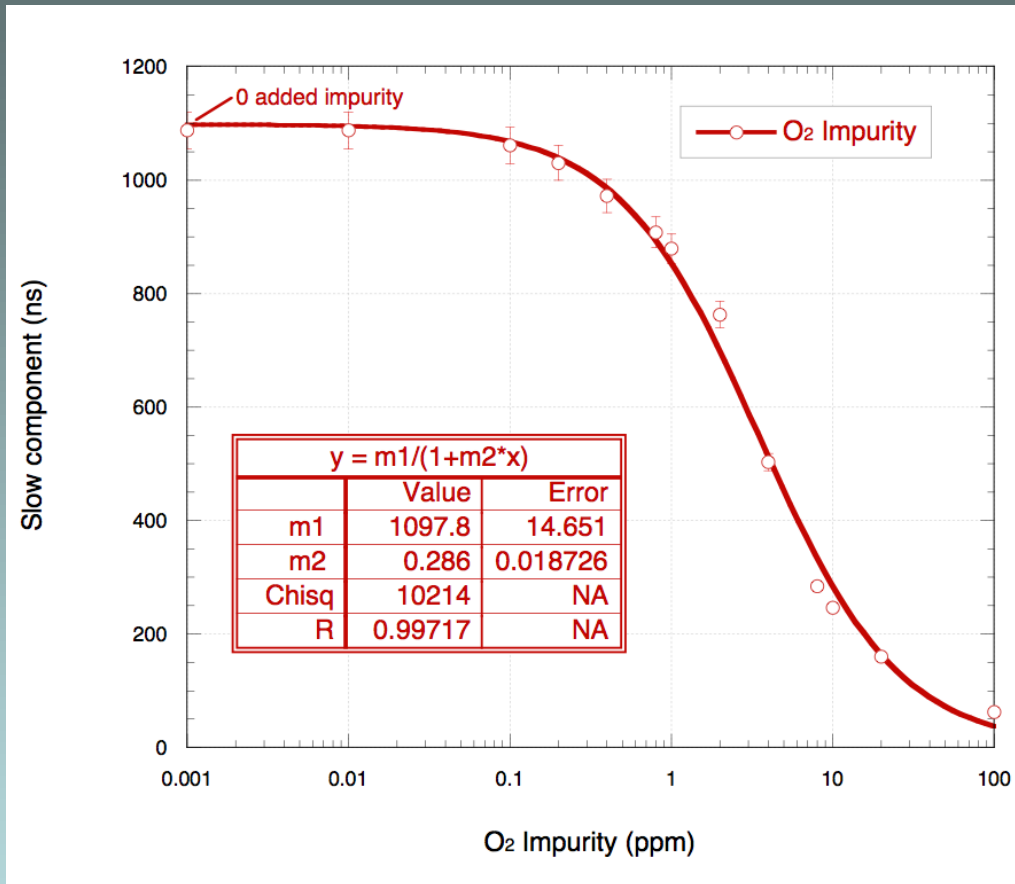
# LAr Recirculation and Purification Test



**Within 4 days continuous re-circulation at rate of 27 litres/hour, the slow component decay time was increased by 200 ns reaching ~1100 ns**



# Effect of 0.01 ppm to 100 ppm O<sub>2</sub> impurity on LAr scintillation light



Data collected using  
an Am-241 alpha  
source

Birks law type  
function fit  
 $y=m1/(1+m2*x)$ ;  
 $m1=1097\text{ns}$ ,  $m2=0.28$

Correlation of the LAr slow component decay  
time with part per million O<sub>2</sub> impurity

# Summary & Conclusions

**Effect of various impurities on the LAr scintillation –correlation of slow component decay time to ppm equivalent impurity level**

**Purification Chemicals -How they work**

-Molecular Sieves, Cu and  $P_4O_{10}$

**Efficiency of the chemicals at removing  $O_2$ ,  $H_2O$  and  $N_2$  found to be high**

**The Liverpool LAr Cell:**

- Construction of a novel cryogenic recirculation pump,
- Successful operation of the pump with a recirculation rate of 27L/hour over four days
- Successful purification of LAr over 4 days recirculation -slow component of LAr increased by 200 ns reaching ~1100 ns
- Slow component decay time of LAr scintillation light was correlated to ppm  $O_2$  equivalent contamination

# Thank You