

# System to Test the Effects of Materials on the Electron Drift Lifetime in Liquid Argon and Observations on the Effect of Water

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## Abstract

A material test system (MTS) has been developed at FNAL to assess the suitability of materials for use in a large liquid argon time projection chamber. During development of the MTS, it was noted that controlling the cryostat pressure with a raining condenser reduced the electron drift lifetime in the liquid argon. The effect of condensing has been investigated using a series of passive materials to filter the condensate. We report the results of these studies and of tests on different candidate materials for detector construction. The inferred reduction of electron drift lifetime by water concentrations in the parts per trillion is of particular interest.

*Key words:* LArTPC, Liquid Argon, Purity

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## 1. Introduction

2     Liquid argon time projection chambers (LArTPCs) offer an opportunity for  
novel neutrino physics [1]. They can provide bubble-chamber quality event im-  
4     ages by drifting ionization electrons created by the passage of charged particles  
through the liquid to readout planes. Since argon is cheap and plentiful, one can  
6     conceive of detectors with multi-kiloton active volumes. A principal challenge  
for large LArTPCs is the removal of electronegative impurities that capture the  
8     ionization electrons. The Material Test System (MTS) has been built at FNAL  
to develop liquid argon purification techniques [2] and to qualify materials for  
10    use in a large LArTPC by measuring their effect on the electron drift lifetime.  
A schematic of the MTS cryostat is included as Figure 1.

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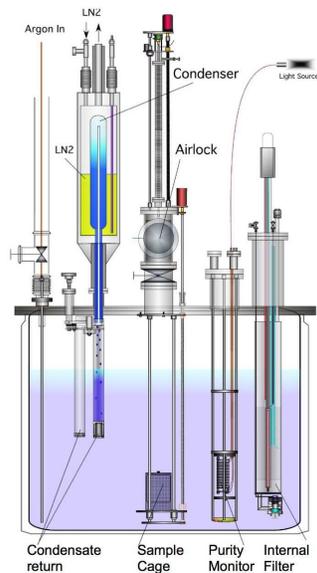


Figure 1: Schematic of the materials test system (MTS) cryostat at FNAL.

## 12 2. The Materials Test System

### 14 2.1. Argon Source

16 Commercial argon [3] is passed through molecular sieve [4] to remove water  
 18 and activated copper [5] to remove oxygen and other electronegative impurities  
 20 before entering the MTS cryostat. The liquid argon is supplied through vacuum-  
 22 jacketed  $\frac{3}{8}$  inch diameter tubing that consists of both stainless steel and copper  
 24 sections. Small diameter tubing was chosen to limit the system throughput to  
 26 match the capacity of the cryostat relief valve. The molecular sieve and activated  
 copper filter material are each housed in  $2\frac{3}{8}$  inch diameter stainless steel tubing  
 capped with ConFlat flanges. All valves in the delivery system are metal seal to  
 atmosphere to prevent the diffusion of oxygen through o-ring or stem packing  
 seals. Piping relief valves with o-ring seals have an argon purge on the exhaust  
 to prevent diffusion of ambient. The supply piping conforms to ASME B31.3  
 and the cryostat conforms to ASME Section VIII DIV 1. This setup provides  
 liquid argon with an electron drift lifetime of many milliseconds [6].

### 28 2.2. Internal Filter

30 This wholly-internal filter sits in the MTS cryostat and contains a combina-  
 32 tion of molecular sieve and activated copper. It is used to maintain the purity  
 of liquid argon in the cryostat and also to remove impurities that may be intro-  
 duced during materials testing. A description of filter operation can be found  
 in [2].

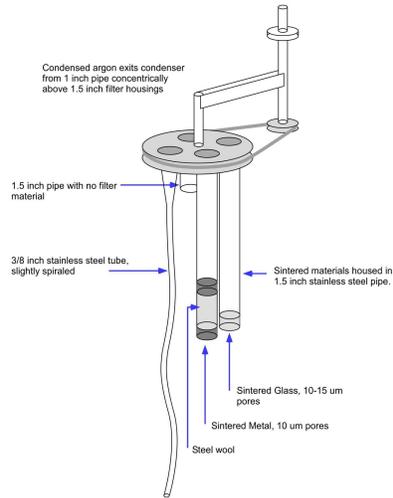


Figure 2: Detail of return mechanism. The return mechanism contains four return paths: a thin tube, a tube that contains sintered glass, a tube that contains steel wool and sintered metal, and a pipe stub with no filter media. The thin tube extends approximately 36 inches into the cryostat, which has a depth of 40 inches. The pipes for the sintered metal and sintered glass extend approximately 20 inches into the cryostat. A handwheel, fed through the top flange of the cryostat, is used to select the return path into which condensate drips.

### 2.3. Lifetime Monitor

34 Modeled after the ‘purity’ monitors of the ICARUS Collaboration [6, 7], this device allows for the direct measurement of the electron drift lifetime.

### 36 2.4. Condenser to Control Cryostat Pressure

Argon vapor enters the condenser through a central tube and contacts sur-  
 38 faces cooled with liquid nitrogen maintained at 50 psia to prevent argon from  
 freezing on the contact surface. The condensed argon flows down the condenser  
 40 walls and drips into one of four condenser return paths before entering the bulk  
 liquid. When the condenser is not operating, argon is continuously vented. A  
 42 closed system is desirable during materials testing so that material-introduced  
 impurities remain in the cryostat and their effect on electron drift lifetime can  
 44 be observed.

### 2.5. Return Paths for Condensed Argon

46 A wheel below the condenser allows the selection of a return path for the  
 condensate. There were four paths available: a  $1\frac{1}{2}$  inch diameter tube with  
 48 stainless steel wool enclosed in sintered metal, a similar tube with a disk of  
 sintered glass, a thin spiral tube, and a hole which allows the condensate to fall  
 50 directly into the bulk liquid. Figure 2 shows details of this system. Other return  
 paths, described in Section 3.1, were used briefly.

52 *2.6. Mechanism for Material Insertion*

54 An airlock, separated from the cryostat by a large gate valve, sits above the  
cryostat. A sample material is placed into a sample cage inside the airlock and  
56 prepared for insertion by purging with argon or evacuation. The gate valve is  
then opened and the cage lowered into the cryostat via a rod attached to the  
top of the cage. Once in the cryostat, the cage is set on a lift platform equipped  
58 with an RTD to indicate temperature. The rod is then retracted, the gate valve  
closed, and the cage lowered further into the cryostat.

60 Since it may not be possible to evacuate the cryostat of a future large  
LARTPC, the MTS airlock has the ability to prepare materials for insertion  
62 by purging with argon. Samples may also be subject to evacuation, but this  
procedure is not routinely used since evacuation might remove contaminants  
64 that would otherwise remain.

*2.7. Data Acquisition and Control Systems*

66 The data acquisition system for the lifetime monitor consists of a Visual  
Basic program run on a Tektronix 5054NV digital oscilloscope. The system is  
68 fully automated and takes measurements at a user-specified interval. A commu-  
nication program sends the lifetime data to the Ifix interface where it is stored  
70 with the MTS system information.

The MTS controls are automated using a Beckhoff Programmable Logic  
72 Controller (PLC). The PLC reads out the pressure, liquid level, temperature,  
and gas analysis instrumentation. Based upon the monitored instrument val-  
74 ues, the PLC performs tasks such as opening and closing valves to control the  
cryostat pressure or sounding audible alarms that alert operators of undesir-  
76 able conditions. The PLC communicates with iFIX software run on a Windows  
PC. The iFIX software allows entry of temperature and pressure set points and  
78 other operational parameters, displays real time instrument values, and archives  
instrument values for historical viewing. The iFIX graphical user interface is  
80 included as Figure 3.

*2.8. Operation*

82 Operation of the MTS involves evacuating the cryostat, filling it with filtered  
commercial argon, inserting a sample material, and monitoring the electron drift  
84 lifetime. Upon evaluation, the sample may be removed and another sample  
material inserted. The condenser and internal filter are operated as needed.

86 **3. Effect of Condenser Operation on Electron Drift Lifetime**

After obtaining long electron drift lifetimes at FNAL [6], the condenser was  
88 first used to control MTS cryostat pressure in January 2008. The condensate  
was allowed to drip directly into the bulk liquid and it immediately became clear

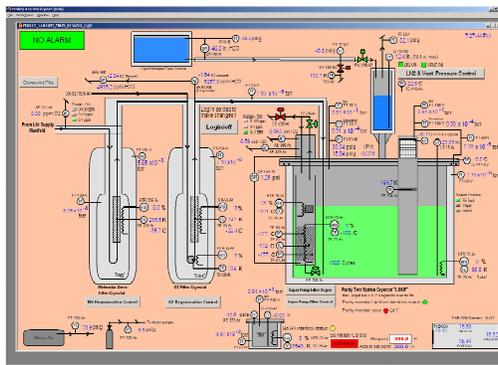


Figure 3: The iFIX graphical user interface for the MTS controls.

90 that condensing reduced the electron drift lifetime dramatically, from ten mil-  
 92 liseconds to less than one millisecond, as shown in Figure 4<sup>1</sup>. This prompted us  
 to begin characterization of impurities introduced during condenser operation,  
 or condensing-associated impurities.

### 94 3.1. Characterization of Condensing-Associated Impurities

96 Since the cryostat had been evacuated to below  $10^{-6}$  Torr and there was  
 98 little material in the vapor of the cryostat other than the three coaxial lifetime  
 monitor cables, it did not seem likely that chemical impurities were introduced  
 100 into the liquid during condensing. It was initially thought that the decrease in  
 lifetime was caused by argon ions that formed as the condensate dripped from  
 the metal surface of the condenser down to the liquid [9].

102 Direct modification of the condenser would have been difficult since the  
 104 condensate return concentrically surrounds the gas inlet. Therefore, a pipe was  
 106 installed beneath the outlet of the condenser that contained a section filled with  
 stainless steel wool enclosed by sintered metal discs, the idea being to discharge  
 any ions. This addition to the system allowed for drift lifetimes of several  
 108 milliseconds. There was, however, still some uncertainty in our minds about  
 the action of this new feature. To confirm that ions were indeed the impurity  
 110 introduced during condensing, the steel wool and sintered metal section of the  
 return pipe was replaced with a section containing an electrically isolated metal  
 112 rod at its center. With a potential difference of  $1\frac{1}{2}$  kV between the rod and the  
 pipe, any argon ion would have plenty of time to reach an electrode given the  
 flow rate of the condensate through the pipe and the pipe's physical dimensions.

In practice we observed very little difference in lifetime whether the so-called

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<sup>1</sup>When the raining condenser was designed, we were not aware of relevant work done by the ICARUS Collaboration [8] that shows a high impurity concentration in the argon vapor relative to the liquid.

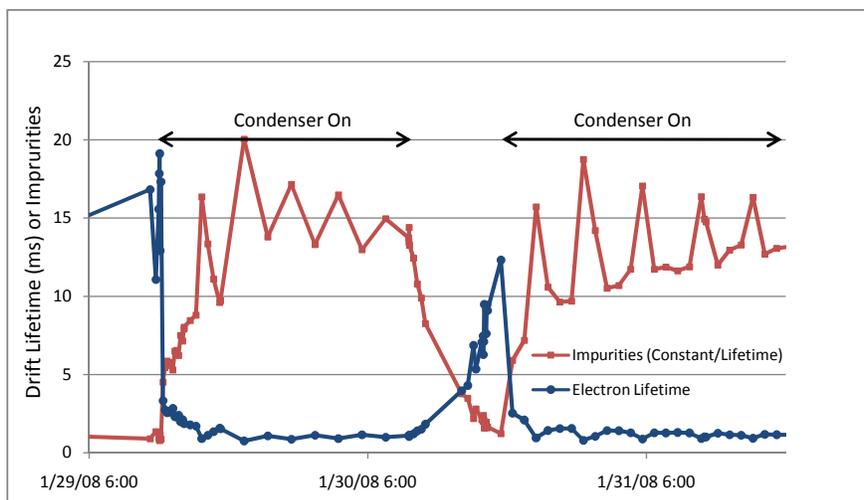


Figure 4: Effect of condenser operation on electron drift lifetime. The impurities, defined as a constant divided by the drift lifetime, represent the physical contaminants in the argon. When the condenser is off, the drift lifetime approaches 20 ms; when the condenser is on, the lifetime quickly degrades to 1 ms or less. The oscillations in the drift lifetime are related to cycling of the condenser.

114 ion rod was set to be a cathode or an anode or without any applied potential,  
 115 implying that the effect of the steel wool and sintered metal was not from  
 116 discharging ions. When the steel wool was examined under a microscope to see  
 117 if the effect was from trapping some particulate, the material was pristine—  
 118 suggesting that if the metal was trapping something, the trapped material had  
 evaporated when warmed to room temperature.

### 120 3.2. Characterization of Condensing-Associated Impurities with Return Paths

To better understand the effect of condenser operation, a mechanism was  
 122 installed beneath the outlet of the condenser that allowed one of four return  
 paths for condensate return. This device is shown in Figure 1 and detailed in  
 124 Figure 2.

The different return paths were chosen for their ability to selectively remove  
 126 ions or particulate from the condensate. The thin, spiraled tube was designed  
 to stop condensed argon from dripping into the bulk liquid and so prevent  
 128 the generation of ions. The sintered glass was chosen for its ability to remove  
 particulate, but not discharge any ions generated as the condensate dripped from  
 130 the condenser into the return path. The sintered metal and steel wool return was  
 used because it had prior success at removing condensing-associated impurities  
 132 (see Section 3.1), presumably because it removed both ions and particulate.  
 The hole was chosen to provide a baseline to which to compare the effects of the  
 134 other return paths. Furthermore, the length of the sintered glass and sintered

136 metal returns was chosen to allow the ends to be uncovered if the argon depth  
 in the cryostat was below 18 inches—thus forcing the condensed argon to once  
 again drip out of the return and splash into the liquid.

138 The cryostat was initially filled with 29 out of 40 inches of argon, enough to  
 cover the outlets of all the return paths except the hole. The effect of filtering  
 140 the condensate through each of the returns was observed and results are shown  
 in Figure 5.

142 In order to understand the effects of the return paths and internal filter  
 operation, the impurity concentration in the cryostat was modeled. It was con-  
 144 venient to use three ‘types’ of impurities, each with different behavior. The  
 first type of impurities, base impurities, provides a constant impurity con-  
 146 centration that limits the maximum electron drift lifetime (1). While not a  
 physical type of impurity, this impurity concentration combines any non-ideal  
 148 or not-understood behavior of the MTS into one quantity. The second type,  
 condensing-independent impurities, accumulates as surfaces slowly outgas into  
 150 the liquid and is removed by internal filter operation (2). The rate of surface  
 outgassing decreases over time in a way similar to a surface under vacuum. The  
 152 third type, condensing-associated impurities, accumulates at a rate proportional  
 to condenser activity and is removed by internal filter operation and another  
 154 passive mechanism that is clearly present (see Figure 5) and attributed to the  
 gettering ability of cold metal surfaces. The gettering effect applies only to  
 156 condensing-associated impurities. Also, the rate at which the condensing asso-  
 ciated impurities are added to the liquid is affected by the return path. It was  
 158 assumed each return path removed a constant fraction of the impurities from  
 the condensate before returning it to the bulk liquid (3).

160 The sum of the three impurity concentrations gives the total impurity con-  
 centration in the liquid and determines the electron drift lifetime. Table 1 gives  
 162 the parameters of the model. Unknown parameters were found using a least-  
 squares fit to the observed electron drift lifetimes.

$$[I1] = \textit{Base Impurities} \quad (1)$$

$$\frac{d[I2]}{dt} = (\textit{Cond. Indep. Source}) \cdot t^{-1/2} - (\textit{Int. Filter Const.}) \cdot [I2] \quad (2)$$

$$\begin{aligned} \frac{d[I3]}{dt} = & (\textit{Cond. Assoc. Source}) \cdot (1 - \textit{Frac. Removed by Return}) \\ & - (\textit{Int. Filter Const.} + \textit{Gettering Const.}) \cdot [I3] \end{aligned} \quad (3)$$

164 The sintered metal and steel wool return path removed a large fraction  
 ( $\approx 90\%$ ) of the condensing-associated impurities, but the performance of the  
 166 other returns did not conclusively differentiate between ions or particulate.  
 Our preferred explanation for the observed effects of the return paths is that  
 168 condensing-associated impurities desorb from ‘warm’ metal surfaces and mix

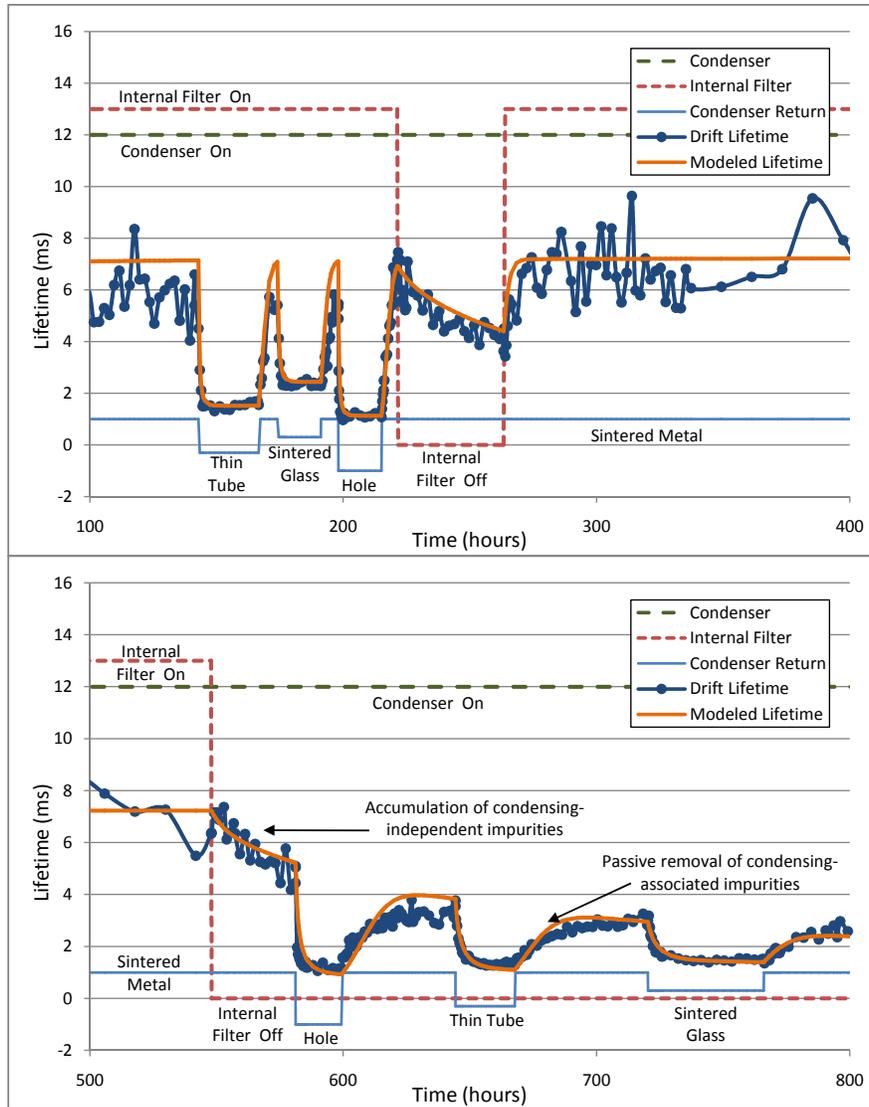


Figure 5: Electron drift lifetime as a function of return path and internal filter operation. The dashed red line and the dashed green line indicate, respectively, internal filter and condenser operation: high for on, low for off. The light blue line and its associated labels indicate which condenser return was in use. The dark blue and orange lines show, respectively, the observed drift lifetime and the modeled drift lifetime. The figure shows the effect of the different return paths on the drift lifetime with the internal filter both on (first part of Figure 5) and off (second part of Figure 5). For reference, when the condenser is off, the drift lifetime is 10–20 ms.

Constant		Value	Units	Comment
Base Impurities		0.07		Determined from drift lifetime while venting with internal filter on.
Condensing-Independent Source		0.027	1/hour	Determined from fit.
Condensing-Associated Source		0.16 or 0.64	1/hour	0.16 if int. filter is off, 0.64 if on. Ratio determined by condenser LN2 consumption.
Fraction Removed by Return	Hole	0		Defined as zero.
	Thin Tube	0.28		Determined from fit.
	Sintered Glass	0.58		Determined from fit.
	Sintered Metal	0.92		Determined from fit.
Internal Filter Constant		0.63	1/hour	Determined from internal filter speed.
Gettering Constant		0.16	1/hour	Determined from fit.

Table 1: Constants for modeled impurity concentration in the liquid of the MTS cryostat. The electron drift lifetime in milliseconds equals  $1/([I1]+[I2]+[I3])$ . Some constants were estimated from operational parameters; others were determined from a least-squares fit to the observed drift lifetime.

with the argon vapor but adsorb to ‘cold’ metal surfaces and exit the liquid  
170 argon. Return path behavior depends only on the amount of cold metal surface  
172 area presented to the condensate. This explanation accounts for differences  
174 in return path performance and also accounts for the passive removal of  
176 condensing-associated impurities from the bulk liquid as they attach to the walls  
178 of the cryostat. As a check on this explanation, the amount of cold metal surface  
180 area presented by the return paths to the condensate was decreased by lowering  
182 the liquid level in the cryostat to 16 inches. The return paths removed fewer  
condensing-associated impurities in this new operating condition, as shown in  
Table 2. The continued success of the sintered metal and steel wool return also  
eliminates the possibility of ions as the condensing-associated impurity. With  
the lower level of argon in the cryostat, ions would have been generated as  
the condensate dripped from the sintered metal return into the bulk liquid and  
decreased the electron drift lifetime—an effect which was not observed.

### 3.3. Water as a Candidate for the Condensing-Associated Impurity

184 Condensing-associated impurities that appear in the liquid are removed by  
the internal filter. This suggests that the argon cannot be the long-term source  
186 of these impurities. Since the cryostat is evacuated before filling with argon,  
the source is unlikely to be in the gas-phase. Water, however, is well known to  
188 remain on metal surfaces in vacuum [10] and has an affinity for cold surfaces.

In order to further investigate the effect of water, a Tiger Optics moisture  
190 analyzer [11] with a 2 ppb detection limit and a 1 ppb resolution was used to

Return Filter	Cold Metal Surface Area Presented to Condensate (cm <sup>2</sup> )		Drift Lifetime (ms)	
	29" LAr	16" LAr	29" LAr	16" LAr
Hole	0	0	1.1	1
Thin Tube	150	70	1.5	1.3
Sintered Glass	300	Near 0	2.4	1.2
Sintered Metal	≈5000	≈5000	5 to 8	5 to 8
N/A (Venting)	N/A	N/A	10–20	10–20

Table 2: Electron drift lifetime as related to return path and liquid level. The sintered glass return path had less metal surface area that contacted the condensate and removed fewer impurities at the lower liquid level. This supports the explanation that return path performance depends on the amount of cold metal surface area presented to the condensate.

monitor the water concentration in the MTS cryostat. The argon vapor was  
192 monitored for moisture content because the moisture analyzer was not sensitive  
to concentrations in the liquid. For example, when using the sintered metal  
194 return and operating the internal filter, we estimate the water concentration in  
the liquid is  $\approx \frac{1}{500}$  of that in the vapor. The sintered metal leaves only  $\frac{1}{10}$  of the  
196 impurities in the condensate; this ratio is further reduced by the internal filter,  
which filters liquid 50 times faster than the condenser adds liquid. The final  
198 ratio of the concentrations depends on operational parameters of the cryostat  
and associated apparatus and on the condition of the sintered metal return.

200 The airlock volume was connected to the cryostat volume to see if warm  
metal surface area would add water to the system and reduce the electron drift  
202 lifetime. During this test, shown in Figure 6, the internal filter was operated  
and the sintered metal return path was used. The water concentration in the  
204 argon vapor increases when the airlock is connected to the cryostat and also  
is an indicator of drift lifetime: the product of the drift lifetime and water  
206 concentration remains roughly constant. This provided indication that water  
may be the condensing-associated impurity.

#### 208 4. Material Tests and Inferred Effects of Water on Electron Drift Lifetime

210 A series of material tests, summarized in Table 3, was performed to deter-  
mine the effect of various materials on the drift lifetime and the role of water.  
212 Test materials were inserted into the sample cage in the airlock and then evac-  
uated and/or purged with argon from the cryostat. The cage with the material  
214 was then lowered into the liquid argon and subsequently raised into vapor. The  
RTD attached to the platform supporting the cage recorded the temperature  
216 of the sample. Material tests were conducted while operating the internal filter  
and using the sintered metal return.

218 During materials testing, it was noted that the water concentration in the  
argon vapor was correlated with the electron drift lifetime in a way similar to

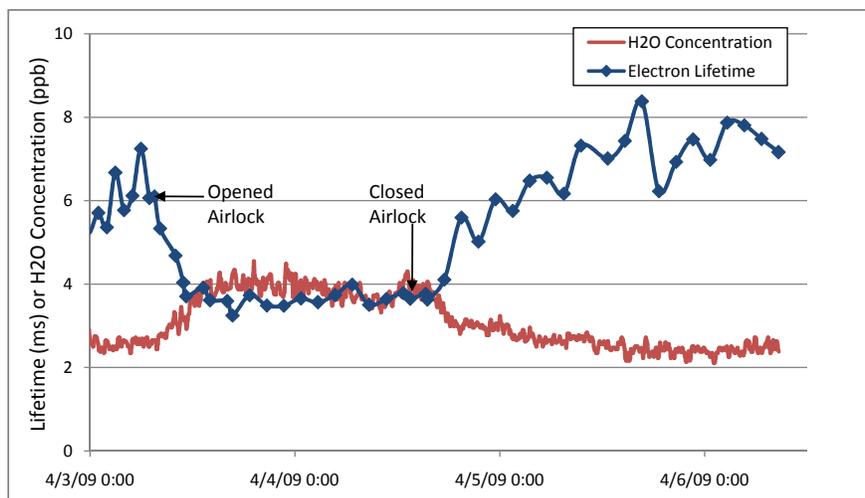


Figure 6: Effect of connecting cryostat and airlock volumes. The cryostat was connected to the airlock by opening the gate valve that typically separates the two. Prior to opening, the airlock was under vacuum. The increase in water concentration is attributed to the additional warm metal surface area in contact with the argon vapor. The relationship between water concentration and drift lifetime is similar to the relationship observed during materials tests (e.g. Figure 7). The material test was performed with 15 inches LAr in the cryostat.

220 that of joining the cryostat and airlock volumes. See Figure 7. The product of  
 222 the drift lifetime and the water concentration was a constant, independent of  
 material:  $(\text{Drift Lifetime in ms}) \cdot (\text{H}_2\text{O Concentration in ppb}) \approx 17$ . It was also  
 224 noted that when evacuated in the airlock for a few days prior to testing, PC  
 board materials had little effect on the water concentration in the argon vapor.  
 See Figure 8. These observations suggest that water may be the sole significant  
 226 electronegative impurity introduced by various materials.

228 While there are more materials of interest to test and our characterizations  
 are not complete, the results presented in Table 3 indicate a variety of materials  
 230 outgas water when warm ( $\approx 225$  K) and that water, at least with our current  
 condenser and condensate return, reduces the drift lifetime in the MTS cryo-  
 232 stat. Also, it appears water may be removed from materials by evacuation and  
 perhaps by leaving the material in argon vapor for an extended period. Further  
 investigation is required.

## 234 5. Summary and Conclusions

236 We have built a system (the MTS) to test materials for use in a large liquid  
 argon TPC. The current system uses a raining condenser with paths for conden-  
 238 sate return. We have found that materials inserted into the liquid argon have  
 very little effect on the electron drift lifetime. We have observed a direct rela-  
 tion between the water concentration in the vapor above the liquid argon and

Material	Sample Surface Area (cm <sup>2</sup> )	Effect of Material on Electron Drift Lifetime (LT)			Comments
		94 K liquid	≈120 K vapor	≈225 K Vapor	
Red-X Corona Dope	100	None	None	LT Reduced from 8 to 1 ms; recovery observed.	H <sub>2</sub> O concentration not monitored.
Deactivated Rosin Flux	200	None	Not Tested	LT reduced from 8 to 1.5 ms recovery observed	H <sub>2</sub> O concentration not monitored.
FR4	1000	None	Not Tested	LT reduced from 8 to <1 ms	Outgassed enough H <sub>2</sub> O at 225 K to saturate sintered metal return.
Taconic	600	None	Not Tested	LT reduced.	Sample outgases water at 225 K.
Hitachi BE 67G	300	None	Not Tested	LT reduced; recovery observed	Sample outgases water at 225K; outgassing reduced over time.
TacPreg	200	None	None	LT reduced; recovery observed	Sample outgases water at 225 K; outgassing reduced over time.
FR4, y-plane wire endpoint for uBooNE	225	None	None	LT reduced from 8 to 3 ms	Sample outgases water at 225 K.
FR4, y-plane wire cover for uBooNE	225	None	None	None	Sample was evacuated in airlock before before testing
Devcon 5-min epoxy	100	None	None	LT reduced from 10 to 6 ms; some recovery observed	Sample outgases water at 225 K.

Table 3: Summary of material test results. Materials were inserted into the liquid argon then subsequently raised to different temperatures in the argon vapor. The water concentration in the argon vapor and the electron drift lifetime (LT) were monitored during material tests. Most materials began outgassing water and reduced the drift lifetime when raised to 225 K. When maintained at this temperature for several days, outgassing decreased for some materials and there was a corresponding increase in the drift lifetime. The water concentration of the argon vapor was not monitored for the first two material tests. In all of the material tests in which the water concentration was monitored, it was related to the drift lifetime by  $(\text{Drift Lifetime in ms}) \cdot (\text{H}_2\text{O Concentration in ppb}) \approx 17$ .

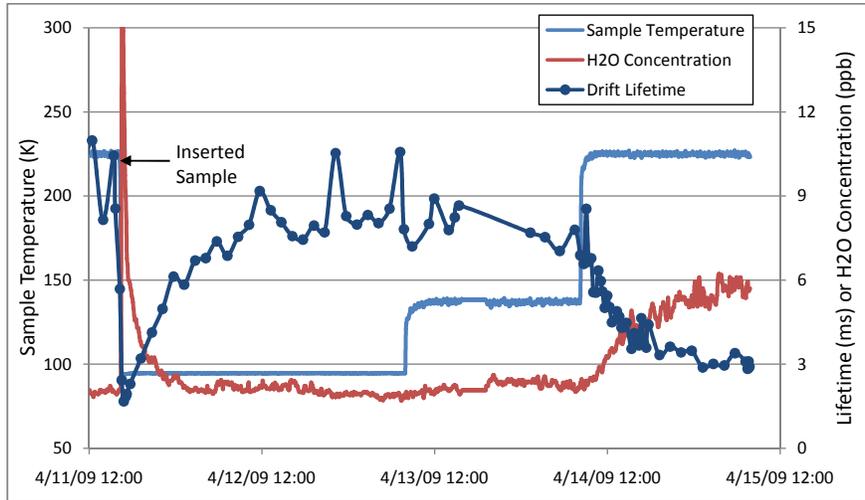


Figure 7: Material test of FR4 y-plane wire endpoint. The sample was first lowered into the liquid argon then raised so that the temperature of the sample was increased. When moved to 225 K, the sample began to outgas and the effect on water concentration and drift lifetime can be seen in the figure. A similar relationship between water concentration and drift lifetime was observed during other material tests, including the metal surface area test documented in Figure 6. The material test was performed with 17 inches LAr present in the cryostat.

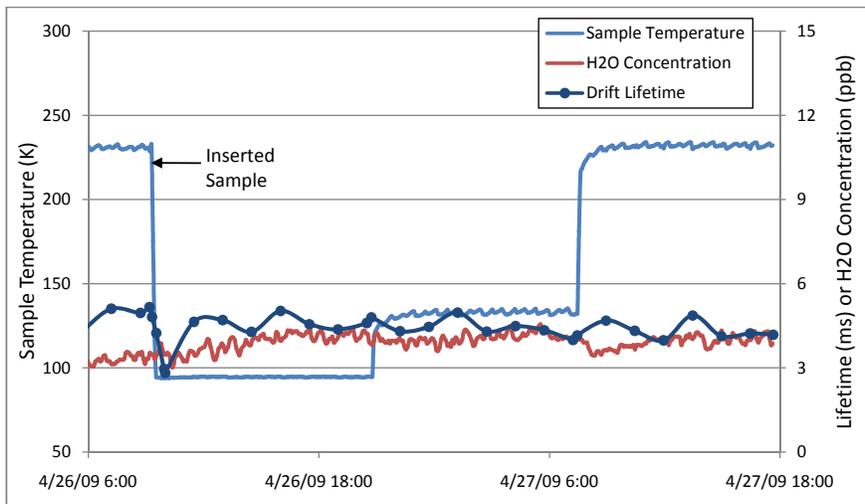


Figure 8: Material test of y-plane wire endpoint after evacuation. The sample was placed in the airlock and evacuated to 1 mTorr for a few days prior to testing. The sample did not outgas any water and had no effect on the drift lifetime. The material test was performed with 13 inches LAr present in the cryostat.

240 the electron drift lifetime of the form (Drift Lifetime)·(Water Concentration)=a  
constant. We can affect the water concentration by introducing different ma-  
242 terials into the vapor space and the constant is independent of material. We  
have not directly measured water concentrations in the liquid but we infer that  
244 concentrations at the level of tens of parts per trillion affect the drift lifetime.  
Based on our observations, we think water moves through our system in the  
246 following way. Warmer metal surfaces and unevacuated, warm, and perhaps  
recently-introduced materials release water into the argon vapor. Condenser  
248 operation introduces the water-contaminated argon vapor into the liquid of the  
cryostat where water naturally exits the liquid because of its affinity for cold  
250 metal surfaces. It is also removed by operation of an internal filter. The equilib-  
rium concentration of water in the liquid determines the electron drift lifetime.  
252 We find that exposing the condensate to a large cold metal surface before entry  
to the bulk liquid can remove much of the water from the condensate. Water  
254 may also be prevented from entering the liquid by filtering the condensate. A  
condenser system allowing the condensate to return directly to the liquid will  
256 ruin the electron drift lifetime unless the water concentration in the vapor is  
well below one part per billion.

## 258 6. Acknowledgements

For Stephen to fill out.

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