Gas Analysis

1 Reasons for gas analysis

1. Check the quality of the argon delivered for water, oxygen and nitrogen content
2. Monitor the progress of the purging of the cryostat atmosphere
3. Check for the appearance of leaks during the gas phase of recirculation and purification
4. Ensure that the ultimate level of atmospheric contamination is low enough not to be a significant load on the purification system
5. Monitor the effectiveness of the liquid purification in its early stages before the purity monitors come on line.
6. Help diagnose problems should they occur during operation, including assessing contamination introduced in case of emergency removal/replacement of equipment

This list may not be complete but is based on our experience at Fermilab in using the purging process to remove the atmosphere in the Liquid Argon Purity Demonstration (LAPD) [1]. In particular, (item 3) we became aware of a significant leak during the recirculation and purification phase the first time we attempted to operate the LAPD, see Figure 1, and (item (6)) we could measure the contamination level produced and predict the time back to operation after we removed and replaced the feedthrough for the LAPD.

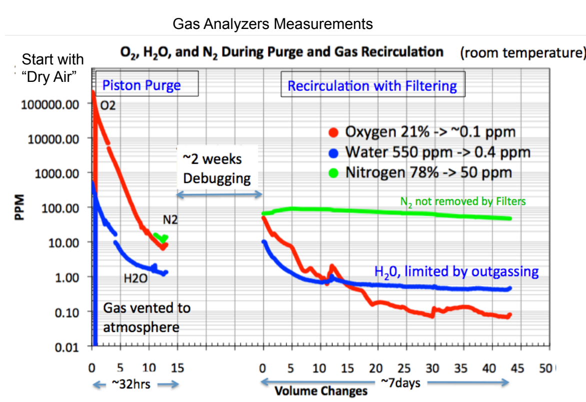

Figure 1: Analyzers during purge and recirculation when leak was identified

2 Equipment

The contaminants of most interest in argon are water and oxygen which affect the electron drift lifetime at the sub parts per billion (ppb) level, and nitrogen which affects light production and long-range propagation at the parts per million level.Figure 2 shows the analytic equipment made available at Fermilab first for the Liquid Argon Purity Demonstrator and then the 35 ton cryostat projects. The instruments are all commercial products and each identifies one contaminant. The dual purpose Tiger Optics unit is laser-based and measures water content by absorption of a water line - oxygen is measured by drying the incoming gas and then mixing the incoming gas with hydrogen to form water.

PC4 Gas Analyzers

Manufacturer	**Delta-F**	**Tiger Optics - one unit**		**Delta-F**	**Delta-F**	**Servomex/Kontrol Analytik**	**Tiger Optics**
Model	**NanoTrace II DF-560**	**LaserTrace O2**	**LaserTrace H20**	**DF-310**	**DF-310*E***	**K2001^{ns}**	**HALO H20**
Budget price	**$35k**	**$80k**		**$5k**	**$10k**	**$25k**	**$25k**
Species analyzed	Oxygen	Oxygen	Water	Oxygen	Oxygen	Nitrogen	Water
Ranges of operation	0-20 ppm	0-1.25 ppm	0-2.5 ppm	0-5,000 ppm	0-50 ppm	0 - 100 ppm	0-20 ppm
LDL (experience based)	1 ppb	1 ppb	1 ppb	1 ppm	100 ppb	100 ppb	4 ppb
Comments	The only high sensitivity oxygen meter in this set that is reliable. Used to monitor tank liquid purification and oxygen filter saturation. Long recovery time from oxygen upsets, typically use DF-310E above 1 ppm to protect it and prevent long recovery times.	These two analyzers are one unit that can't be separated. The oxygen analyzer is difficult to operate and at the moment is compromised. The water analyzer is the most sensitive water analyzer in the system. The water analyzer is used to check for water filter saturation and other careful measurements. The water analyzers take a long time to come to equilibrium because the water in the gas stream must come to equilibrium with the tubing along its entire length. Thus a water analyzer can't be switched between sources on a short time scale. It can often take days to reach a stable reading		High range of this oxygen meter is necessary for tank purge from air and to monitor tank vapor space during any tank extraction or insertion type repairs that introduce gross amounts of contamination.	Mid range oxygen analyzer. Used during the purge from air and gas recirculation phases until the NanoTrace can be brought online or any time the contamination is above 1 ppm to protect the NanoTrace.	Only N2 analyzer, necessary for light collection	Typically this analyzer monitors the tank vapor space so the water outgassing can be integrated over the entire run. The other water analyzer is then used to sample from other points in the system.

Figure 2: Hardware used for oxygen, water and nitrogen measurements at FNAL for LAPD and the 35 ton. Not included is the % sensitivity oxygen monitor used at the very beginning of the purge process.

For the protoDUNE single-phase (NP04), we propose a slightly less comprehensive set equivalent to a Halo 3 for water from 1 ppb to 9 ppm, a Servomex Servopro Nitrogen Plasma instrument with 0.1 ppm resolution, a Servomex DF550 for Oxygen with a sensitivity limit of 0.4 ppb and ranges with upper limits from 20 ppb to 10 ppm, a Servomex DF310E for cruder oxygen measurements up to 10,000 ppm, and a dew point meter for water from ambient to a few ppm. The total cost for these devices from present quotations is about $95k. In the almost certain case that such funding is not available, we would propose to borrow some equipment and/or omit the water analyzer. This latter instrument would be most interesting in its measurements of the water content in the feedthrough flow where the cable mass is warm and likely to be a major potential source of contamination. It is not, however, required for actual operations.

In addition to the analytic equipment itself, a valve switchyard is needed to allow different sampling points to be connected to a given instrument. Figure 3 shows the R & D switchyard developed for the LAPD. The system allows any combination of eight instruments to be connected to any of seven sources.

In our situation, we need to be able to sample:

Input	Output to
Received liquid	Halo, DF310E, Servopro N2, fast purge
Cryostat during purge	DF310E, N2 meter, Dew Point Meter, Halo water meter, fast purge
Cryostat Liquid	Halo, DF 550, Servopro N2, fast purge
Cryostat Ullage	Halo, DF 550, Servopro N2, fast purge
Feedthrough flow	Halo, fast purge
Nitrogen Calibration	Servopro N2, fast purge

Table 1: Gas switchyard inputs and outputs.

Our experience of nitrogen monitors from both Servomex and LDetek has shown that they require frequent calibration and in case of a critical measurement, we would want to calibrate the instrument immediately beforehand.

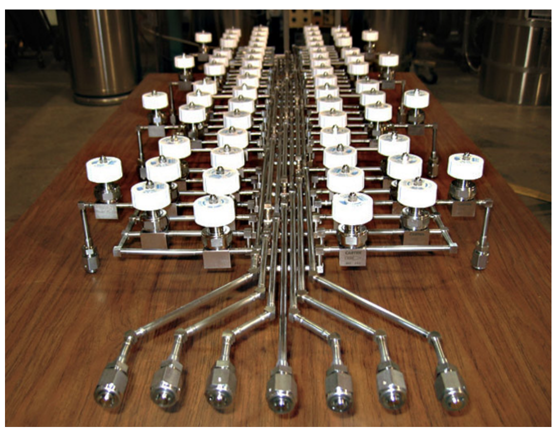

Figure 3: Switchyard to connect sampling points with analytic instruments

3 Interfaces

The gas analyzer system involves interfaces with the cryogenics and with the controls system. The active equipment is outside the main cryostat and its operation is not expected to interfere with the apparatus inside the cryostat.

Cryogenics
The cryogenics system provides the access points to the argon, and the piping from the access points to the switchyard just before the analytic instrumentation. Access is proposed to the line receiving argon from the vendor, to the liquid and to the ullage

in the cryostat, and to the vent line at the feedthrough ports. The total flow of argon through the analyzers will be of the order of 4 l/m (gas at room temperature) which translates to about 1.6 kg/day. If there is a make-up system, it would be helpful to sample th quality of the argon therein.

It is assumed that DUNE will provide the switchyard, pressure regulators, metering valves and any pressure-booster pump needed to satisfy the input pressure requirements on the analyzers. It is hoped that the Neutrino platform group will provide a small dry pump for evacuation of lines as needed.

Controls
We will wish to record the outputs from the analyzers every minute or so in some database. The standard readout provided is 4 to 20 mA but we would exploit options for ethernet or RS 232 data transfer and control where available (e.g.m on the Halo water meter). The actual implementation will be decided by discussion with the DCS group. It is proposed that the state of the switchyard (which source is connected to which analyzer) will be recorded by hand on an electronic shift form.

The analyzers plus switchyard will take about 1 standard relay rack of space whose location will be decided by discussion with DCS and the cryogenics group

4 Charge Status

In response to the charge:

1. *meet requirements* The set of instrumentation proposed will cover the needs for gas analysis from delivery to purge to operations

2. *development towards DUNE* It is envisaged that the same type of instrumentation is appropriate for DUNE. The length of DUNE may require several sets of instrumentation distributed along the length of the cryostat.

3. *schedule, QA/QC, transportation, commissioning* The major item that is not a stock commercial device is the valve switchyard. There is experience both building such a device in house and in purchasing one so the times can be estimated and there is plenty of time. The analyzers come with commercial warranties and the switchyard will be constructed and qualified at Fermilab. These items can be shipped to CERN as ordinary commercial objects. Calibration/span gases will be ordered and used for initial commissioning.

   It is assumed that installation of the piping to the analyzer rack will be done by the Neutrino Platform.

4. *installation plan* There is no installation plan as yet. (April 17 2017). This clearly needs to be resolved with the Neutrino platform.

5. *interfaces* The connections to the cryogenics system are defined in the Piping and Instrumentation Diagram (EDMS). The location of the analyzer rack and switchyard are yet to be determined.

6. *interfaces with slow control* It is expected that the measurements from the analytic equipment will be recorded in a data-base and accessible in some historian. The readout interfaces have not been discussed with the slow control system. This is something that need to be understood and agreed in the next 6 months.

7. *grounding and shielding* We expect that the electrical grounding issues presented by the external piping used for sampling will be resolved by the electrical engineers when they define the grounding for the external piping in general.

8. *operation conditions* The equipment is all external to the cryostat and is not expected to interfere with detector operations. We will check if the pump which may be used to boost the sample line pressures produces any electrical noise that affects the detector readout.

9. *safety of installation and operation* The analytic instrumentation carries electrical certification. Installation of the switchyard and the instrumentation requires careful leak-checking but is otherwise straight forward. Once installed and commissioned, the operations burden is limited to setting the appropriate sources on the appropriate devices and so recording, and periodic calibrations of the N2 sensor. When vacuum pumps are used, procedures will be followed to ensure that we do not pull a vacuum where it is not wanted.

10. *QA,QC,testing, lessons learned* The proposal is based on experience at Fermilab where these instruments have been used extensively. We have consulted the designers at Fermilab and intend to continue this consultation and to incorporate the results of their and local (CERN) experience.

References

[1] M. Adamowski *et al.*, The Liquid Argon Purity Demonstrator, JINST **9** (2014) P07005