JLab Isotope Production Procedures & Practices Manual

Kevin Jordan with inputs from entire Isotope Team January 23, 2020 Rev. C jordan@jlab.org 757-876-1742



Index

Chapter 1 - Overview 1.1 Overview

Chapter 2 – Boron Nitride Crucible

2.1 Procedure for filling Boron Nitride Crucible

Chapter 3 – Instrumentation

- 3.1 Thermocouples
- 3.2 Resistance Temperature Detectors
- 3.3 ADC Settings
- 3.4 Ozone Monitors

Chapter 4 – Alarm Setpoints

4.1 Anticipated Temperature Rise

- 4.2 Operator Instructions for alarm handling
- Chapter 5 Interlocks

5.1 Machine Protection System

Chapter 6

6.1 Procedure for Draining LCW Prior to opening water lines

Appendix A Technical Reference

- 1. P & I Diagram
- 2. Interfaces system print
- 3. Machine Protection System Interface
- 4. Ozone Monitor

Overview of Isotope Experiment

The Isotope Program at JLab partially addresses the national need for research radioisotopes, especially ⁶⁷Cu, by investigating photo-production using photo-nuclear reactions induced by bremsstrahlung photons from a high-power electron linac. Initially, we propose to investigate the production of useful quantities of ⁶⁷Cu.

We used FLUKA [3] to calculate 67 Cu yields in gallium. For the eventual production runs, we have settled on 50 KW beam power for reasons of target heating and heat removal. Calculated yields of 67 Cu for 40 MeV and 100 MeV electron beams at this power in thick 71 Ga and 20 MeV, 40 MeV, and 100 MeV in natural gallium (69 Ga – 60.1%, 71 Ga – 39.9%) targets are presented in Table 1. Total induced radioactivity in the targets is also considered. Calculations were for 10 radiation length targets.

Target	⁶⁷ Cu Production (mCi/h/50kW) E _e = 20 MeV	⁶⁷ Cu Production (mCi/h/50kW) E _e = 40 MeV	⁶⁷ Cu Production (mCi/h/50kW) E _e = 100 MeV		
⁷¹ Ga	10	43	53		
Natural Ga	4	17	22		

Table 1. Yields of ⁶⁷Cu per 50 kW beam in thick ⁷¹Ga and natural gallium targets calculated using FLUKA

For the R&D program, we narrowed the choice of energy to 40 MeV with up to 5kW of beam power. A run at 20 MeV produces much less undesirable spurious radioactivity in the target but the yield of ⁶⁷Cu is much lower. We chose 99.9999% chemically pure gallium for reasons of its low initial copper content at a reasonable cost.

The irradiation plan calls for a run at 20-40 MeV and 1 kW beam power to confirm the radiation shielding calculations before carrying out several runs at 35-40 MeV and 5kW beam power.

The gallium will be contained in a boron nitride crucible which will be removed from the irradiation area using long-handled tongs and placed in a lead pig. The pig will then be placed in a Class A shipping container and sent to VCU for radiochemical separation of the ⁶⁷Cu.

Procedure for Filling Gallium in Isotope Crucible

- 1. Don PPE; Lab coat, safety goggles & Nitrile (11 mil minimum thickness) Gloves
- 2. Lay down protective barrier in the fume hood in lab 1, ensure fume hood is on, and lower the sash as much as practical for splash protection.
- Heat the Gallium in a water bath (glass beaker) on a hot plate to 30 to 40 C, set crucible on hot plate as well to raise temperature to >30C (if exceed 44C, then use tongs to handle beaker.) (Fig. 1)
- 4. Set crucible on a scale & zero scale
- 5. Place 50 grams of Gallium in crucible
- 6. Screw Graphite plug into the end of the crucible. Place a label on the copper plate that holds the crucible to indicate that the crucible contains gallium.
- 7. Store filled crucible on the LCW stabilized copper plate until ready for use
- 8. Properly dispose of gloves in trash
- 9. If gallium is spilled, cleanup with spill pads and return used pads to ES&H for disposal



Figure 1. Crucible and Gallium on hot plate

Instrumentation

Thermocouples & Resistance Temperature Detectors

The isotope experiment is instrumented with both thermocouples (TCs) and resistance temperature detectors (RTDs). Two different technologies are used to minimize any effect from the high radiation area. These are attached to the LCW supply and to each of the return water lines from the window frame cooling, the Tungsten radiator and the base plate cooling. Additional thermometers are located at the Beryllium vacuum window frame, the Tungsten radiator, the base plate and the rear support plug. (Fig. 2) There is a total of 12 thermometers instrumenting the entire isotope assembly.



Figure 2. Additional TCs on the Isotope assembly

The TCs & TRDs are readout using the Omega CNi3253 controllers. (Fig. 3). These have been repurposed from existing equipment already connected to EPICs. The cables have been extended to the rear of the LERF vault, the controllers are placed behind the concrete support column for additional radiation shielding.



Figure 3. Omega controllers for reading out thermocouples and RTDs, 12 channels

ADC Settings

The ADC is a differential input 64 channel VMIC MVME 3122, (Fig. 4 & 5)

Fig. 1 [01/2	22/2020 12:05:23]		/cs/opshome/edm/felmirror/mirror_adc_alarms.edl _					_ 0 3
	MITTOT ADC Readbacks		Mirror ADC Alarm Limits					
Chan.	Function	Value	Cha	n. Function	LOLO	LOW	HIGH	HIHI
1	ITQ1X00 - WINDOW	R2.868	1	ITQ1X00 - WINDOW	20.000	22.000	50.000	80.000
2	ITQ1X00A - TUNGSTEN	40.233	2	ITQ1X004 - TUNGSTEN	20.000	22.000	50.000	80.000
3	ITP1X00 - WINDOW	00.000	3	ITP1X00 - WINDOW	20.000	22.000	50.000	80.000
4	ITP1X00A - TUNGSTEN	40.565	4	ITP1X00A - TUNGSTEN	20.000	22.000	50.000	80.000
5	ITP1X00B - BASE	0.616	5	ITP1X00B - BASE	20.000	22.000	50.000	80.000
6	ITP1X00C - LCW SUPPLY	37.676	6	ITP1X00C - LCW SUPPLY	20.000	22.000	50.000	80.000
7	ITQ1X00B - BASE	36.065	7	ITQ1X00B - BASE	20.000	22.000	50.000	80.000
8	ITQ1X00C - LCW SUPPLY	43.552	8	ITQ1X00C - LCW SUPPLY	20.000	22.000	50.000	80.000
9	ITQ1X00D - Cu WINDOW	42.345	9	ITQ1X00D - Cu WINDOW	20.000	22.000	50.000	80.000
10	ITQ1X00E - Cu TUNGSTEN	00.710	10	ITQ1X00E - Cu TUNGSTEN	20.000	22.000	50.000	80.000
11	ITQ1X00F - Cu BASE	0.000	11	ITQ1X00F - Cu BASE	20.000	22.000	50.000	80.000
12	ITQ1X00G - Cu GRAPHITE	32.404	12	ITQ1X00G - Cu GBAPHITE	20.000	22.000	50.000	80.000
13	IFM1X00 - Window GPM	0.584	13	IFM1X00 - Window GPM	0.100	0.150	0.500	1.000
14	IFM1X00A - Tungsten GPM	0.725	14	IFM1X00A - Tungsten GPM	0.100	0.150	0.500	1.000
15	IFM1X00B - Base GPM	0.031	15	IFM1X008 - Base GPM	0.100	0.150	0.500	1.000
16	Ozone # 1; (PPB)	0.534	16	Ozone # 1: (PPB)	0.000	0.100	100.000	200.000
17	Ozone # 2; (PPB)	6.103	17	Ozone # 2: (PPB)	0.000	0.100	100.000	200.000
18	Spare	7.596	18	Spare	0.000	0.100	8.000	10.000
19	Spare	9.065	19	Spare	0.000	0.100	8.000	10.000
20	Spare	4.637	20	Spare	0.000	0.100	8.000	10.000
21	Spare	7.371	21	Spare	0.000	0.100	8,000	10.000
22	Spare	7.664	22	Spare	0.000	0.100	8,000	10.000
23	Spare	9.695	23	Spare	0.000	0.100	8,000	10.000
24	Spare	10.000	24	Spare	0.000	0.100	8,000	10.000
25	Spare	10.000	25	Spare	0.000	0.100	8.000	10.000
26	Spare	8.950	26	Spare	0.000	0.100	8.000	10.000
27	Spare	2.746	27	Spare	0.000	0.100	8.000	10.000
28	Spare	7.751	28	Spare	0.000	0.100	8.000	10.000
29	Spare	5.0.92	29	Spare	0.000	0.000	8.000	10.000
30	Spare	8.912	30	Spare	0.000	0.100	8.000	10.000
31	Spare	5.784	31	Spare	0.000	0.100	8.000	10.000
32	Spare	8.822	32	Spare	0.000	0.100	8,000	10.000
		Expert 🖪	02	opulo	0.000	0.100	0.000	10.000

Figure 4 & 5. ADC readouts and alarm setpoints

Ozone Monitors

There are two ozone monitors in the vault, one near the concrete support post & the other in the GTS air plenum. (Fig 6 & 7). There is a nitrogen purge line fixed to the region where the electron beam passes through the air between the Beryllium window & the Tungsten target. This line is remotely controlled and interlocked to the Machine Protection System.



Figure 6. Ozone Monitor # 1

Figure 7. Ozone monitor # 2

Alarm Setpoints

Anticipated temperature rises for the various temperature sensors

POWER DISSAPATED IN ISOTOPE RIG kj 10/9/2019

Minimum Flow Rate

		Power				Resulting
Piece	Nomenclature	(W)	Factor	GPM	Delta C	Т
Window	1X00	5	265	0.1	0.1887	35.1887
Tungsten	1X00A	103	265	0.1	3.8868	38.8868
hBN/Gallium	1X00B	892	265	0.1	33.6604	68.6604
Total		1000				
Desired Flow Rate						
		Power				Resulting
Piece		(W)	Factor	GPM	Delta C	Т
Window	1X00	5	265	0.3	0.0629	35.0629
Tungsten	1X00A	103	265	0.3	1.2956	36.2956
hBN/Gallium	1X00B	892	265	0.3	11.2201	46.2201
Total		1000				

Once the exposure begins the temperatures should stabilize within an hour or so, the first data will be the commissioning run. This run will be with a solid hexagonal Boron Nitride crucible (no Gallium), but the remaining hardware is the same as for the irradiation experiment. At this point the final numbers will be entered into the alarm handler. These are anticipated to be:

Window Alarm at 40°C Tungsten alarm at 50°C Base (Crucible + Gallium) alarm at 60°C

If these alarm instructions are to call the experimental run coordinator: Kevin Jordan at 757-876-1742

Machine Protection System (MPS) Interlocks

Each of the three water cooling loops and the Nitrogen gas has a flow interlock switch that is connected to the LERF MPS. The MPS is designed around Beam Modes (allowable total beam power) and Machine Modes (beam destination), Twelve spare channels have been assigned for use with the isotope experiment. (Fig. 8) All of these channels must be green or the Beam Mode will drop from CW to Pulsed – low duty cycle. If any MPS channel trips, call the experimental run coordinator: Kevin Jordan at 757-876-1742

		MPS Beam	Mode Card 3			
Machine Mode In: MM HB: Cur: OK	LTC: OK		Beam Mode (BM HB: Cur:	Dut: <mark>0 </mark> OK LTC	: ОК	
Device ITV7F00B ITV8F03 UV Happek ITV8F04 ITV8F06 ITV8F09 ITV8F09 ITV8F10 ITV8F12 UV Happek Mirror OXM1V00E OXM2V00 OLT3V00 ITV3V00 - - - - - - - - - - - - - - - - - -	Brode C L Z I I Z I I Z I I I Z I I I I Z I I I I I Z I <td>Mask</td> <td>Device VBV2V00E VBV2V00F VBV3V00A VBV3V00B VBV3V00C - - - - - - - - - - - - -</td> <td>Bmode 7</td> <td></td> <td>Mask</td>	Mask	Device VBV2V00E VBV2V00F VBV3V00A VBV3V00B VBV3V00C - - - - - - - - - - - - -	Bmode 7		Mask
VBV9F06 VBV8F01 VBV8F05 VBV8F07 VBV8F13 VBV2V00 VBV2V00A VBV2V00A VBV2V00B VBV2V00D UBV2V00D			N.C. N.C. N.C. Default	7 7 7 0	•	

Figure 8. MPS EPICS Screen with 1X interlocks. One channel is tripped due to the nitrogen flow being off.

There is an interface box in the tunnel near the isotope 1X beamline. The interface has 12, 2-pin connectors that tie into the flow switches, the spare channels have a jumper (short) on each of those cables. (Fig. 9)



Figure 9. MPS Interface box. The nitrogen flow is not turned on, so the LED is off for that channel.

Procedure for Draining LCW from Isotope Rig

- 1. Close IMV1X00C Supply, then IMV1X00C Return. These are the DUMP LCW cooling connections to the isotope rig (Fig. 10)
- Close the supply first then return for the following valves: IMV1X00, IMV1X00A, IMV1X00B. These are the three cooling circuits for the window, tungsten, and base plate. (Fig. 11)
- 3. Place the ¼" drain line connected to IMV1X00D securely in a bucket. Open this valve slowly to avoid any splashing of the LCW. (Fig. 12)
- 4. Open both the Supply & Return ball valves of the LCW circuit you wish to drain, then slowly open IMV1X00D Nitrogen Supply valve to push the LCW out of this circuit, repeat as needed. Be careful not to splash or spill the Dump LCW water. (Fig. 13)



Figure 10. Dump LCW Supply & Return Valves



Figure 11. Three branch cooling circuits



Figure 12. ¼" Nylon drain line



Figure 13. Nitrogen supply to blow circuits dry

Appendix A Technical Reference

- 1. P & I Diagram
- 2. TC/RTD Routing to ADC
- 3. MPS Interface
- 4. Ozone Monitor Manual









ECO SENSORS, INC. Ozone Monitor and Controller

Model C-30ZX

INSTRUCTIONS FOR USE

Before you read further! Be sure that the C-30ZX warms up for at least one hour during your first test; thereafter warm-ups of about 5 minutes should be sufficient if the instrument has recently been used. It is a good precaution to warm-up the instrument overnight to burn off any absorbed chemicals that affect the calibration.

GENERAL DESCRIPTION

The Eco Sensors® model C-30ZX makes ozone monitoring and system control simple and inexpensive. The instrument is not a primary standard and is solid for general monitoring rather than precise measurements. The C-30ZX can be used outdoors under certain moderate temperature and relative humidity conditions.

OPERATION

There is no off-on switch. To power up the instrument, simply plug in the AC adapter or connect 12-24 volts DC. At least one bar on the display will light up. The first green bar is the "on" pilot light; any additional illuminated LED color bars indicate ozone in the new. the area.

It is recommended to test the instrument for ozone response using an ozone generator. Eco Sensors offers hand-held battery-operated test generators.

DATA READOUT

Data can be read out in several ways from the C-30ZX: (See wiring diagram on the other side of this sheet)

1 - LED Display - This is a ten segment "bar graph" or "color bar" display that reads 4 green segments, three yellow segments, and three red segments. The first green segment serves as a pilot light to indicate that the instrument is receiving power. The second green segment comes on at .02 ppm of ozone, the first yellow at .05 ppm, the first red at .1 ppm, and the final red at over .14 ppm of ozone. Complete calibration is shown in the Bar Graph Calibration chart.

2 - Audio Alarm - Comes on at .1 ppm (first red bar) if the alarm switch is on. This trigger threshold can be changed to detect at any other LED color bar point down to .03 ppm (third green bar) as shown on the other side of this sheet.

3 - External Outputs - As can be seen on wiring diagram on the other side of this sheet, a terminal block inside the C-30ZX allows connections to external functions as follows:

Terminals	Function
1 and 2	0-3 volts DC signal output to data logger, RAP-7800, or control systems.
2 and 3	DC power supply input if the AC adapter is not used. These connections can also be used to supply power to the RAP- 7800.
4 and 5	Relay contacts normally open.
5 and 6	Relay contacts normally closed.

There is a 2-circuit 3.5 mm jack is on the bottom of the C-30ZX. This is to be compatible with the accessory plugs of the C-30Z, and also as a jack for the plug connection of the Eco Sensors DL-2 data logger.

SPECIFICATIONS

The C-30ZX has the following specifications

- Sensor: Heated metal oxide semiconductor .
- Sensitivity to ozone:
 - 0-,14 ppm on the LED display 0-,30 ppm by external voltage measurement Detection threshold: .02 ppm

Response time: 10's of seconds to respond and somewhat longer to

- recover
- Accuracy: 10-20% depending on the conditions. Recommended general function check: every three months. Recommended celibration check every year. Temperature range: At least 15-27 degrees C (80-80 degrees F).

- Temperature range: At least 15-27 degrees C (60-80 degrees F). Size: 85 X 35 K 60 mm. (3 1/4" X 1 3/8" X 2 3/8") Weight: 140 grams (5 oz.) Power requirement: 12-24 volts DC at 300 ma. (120 volt 60 Hz. AC adapter supplied to North American customers). Fusing: Self-resetting fuse that triggers at 250 ma. current or more than approximately 24 volts input. Has overvoltage protection diodes to eliminate supply voltage splikes and surges. Enclosure material: flame retardant ABS. Mounting: Wengengtic had the finance of the supplied
- Mounting: by magnetic backing or hanging adapter supplied

ACCURACY AND CALIBRATION

The C-30ZX calibration is shown on the other side of this sheet. The response is linear with the scaling of 1 volt = .1 ppm.

The C-30ZX is most accurate in the .05-.14 ppm range and should be 10-20%. Below .05 ppm, the accuracy can be affected by electronic and chemical interferences. .03 ppm is the lowest reading that generally should be used.

If the LED reads high (all red) with no ozone present, the possible causes are: 1-Broken sensor, such as from dropping the instrument 2-A strong chemical interference such as from acid gases or NOx.

3-The sensor calibration has changed.

- OZONE SENSOR \square CE 2002 OZONE PPM LEVEL ALARM 6 MCDEL C-30ZX Czone Sensur JACK FOR DATA LOGGER 6.4mm HOLE FOR WIRING TO ACCESSORY TERMINAL BLOCK INSIDE THE INSTRUMENT P-20 AG POMER A OUTFORT 12 VEC 500 MA, GTR +

SERVICE AND MAINTENANCE

Please contact your distributor about service. In emergencies, the factory service number is (800) 472-6626, the fax is (505) 988-1315 and the e-mail is sales@ecosensors.com. We recommend that the instrument be maintained and recalibrated annually.

Sensor - The sensor is the only component of the instrument that is likely to fail or to Sensor - The sensor is the only component on the insudirent that is alkely to fail of to require checkups. We recommend that the sensor be checked for satisfactory operation every three months, or more often in environments that are dusty, have high levels of chlorine or halogen compounds, or where there is dust or water spray. The sensor should be observed to see if it is responsive to changes in ozone level and if the ppm readings are reasonable. This can be done with the Eco Sensors model OG-2 hand-held ozone generator.

Indications of sensor problems are:

•The instrument reads full scale all the time. • The ozone concentration readings seem too high and tend to get even higher. . The instrument doesn't respond at all.

Some of these problems can be compensated for by circuit adjustments or simple maintenance. Sensor replacement, however, it is not easy and must be done by Eco Sensors or its authorized service representative.

AC Power Adapters - Replacements are available from your distributor or Eco Sensors. Outside of North-America, AC adapters that satisfy local codes and conditions should be purchased locally using electrical and mechanical specifications supplied by Eco Sensors and found at the web site www.ecosensors.com "products" and then "AC adapters and Power sources."

PRECAUTIONS

- Allow at least 5 minutes warm up, and better, an hour or more. If the instrume hasn't been used for days or weeks, an overnight warm up will be helpful to 'burn off' any contaminants that may have been absorbed by the sensor. Read all instructions in this manual. Keep instrument dry. Never let water or other liquids into the sensor.
- Do not drop the instrument or subject it to continuous vibration.
- Do not store or operate the instrument in high levels of dust. Do not store or operate the instrument in high levels of dust. Do not attempt to service the instrument yourself this will void the warranty. Do not clean the instrument with cleaning chemicals or solvents. Clean it with a damp cloth or with Armor Alt¹¹⁴.
- Do not operate the instrument near heavy aerosols (spray) usage or where oxygen is being administered. Do not operate the instrument or rely on its reading where there are high
- concentrations of:
 - Chlorine or other halogen compounds Sulfer compounds Nitrides of nitrogen (NOx)

 - 0
 - Urine residues or ammonia compounds
 - Acid gases such as sulfuric or nitric acid fumes

When in doubt, operate the instrument at least 24 hours in your worst case environmental condition to see if it will operate properly.

WARRANTY

This product is warranted against defects in materials and workmanship for one year following the date of purchase by the original owner. This warranty does not include damage to the product as a result of misuse, accident, damage, modifications or alterations, and it does not apply if the instructions in this manual are not followed.

If a defect develops during the warranty period, Eco Sensors at its election will repair the instrument or will replace it with a new or reconditioned model of equivalent quality. In the event of replacement with a new or raconditioned model, the replacement unit will continue the warranty of the original unit. To return the instrument, contact your distributor, or call Eco Sensors at (800) 472-6626 or email to sales@eccosensors.com to receive return instructions and a Return Authorization (RGA) number.

Except as provided herein, Eco, Sensors makes no warranties, express or implied, including warranties of merchantability and fitness for a particular purpose. Eco Sensors shall not be liable for loss of use of this instrument or other incidental or consequential damages, expenses or economic loss, or claims for such damage or economic loss. This warranty gives you specific rights, and you may also have other rights, which vary from state to state.

RECORD YOUR SERIAL NUMBER HERE

KEEP THIS MANUAL AND WARRANTY FOR YOUR RECORDS.

Eco Sensors is a registered trademark of Eco Sensors, Inc.

© Eco Sensors, Inc., 2000. Revision 1. For C-30ZX REV 1.

CALIBRATION

Eco Sensors Model C-30ZX



Voltage Output: Linearly proportional to the Ozone Concentration with a scaling of 1 volt = .1ppm, 2 volt = .2ppm, etc.



WIRING TO THE C-30ZX

The C-30ZX internal relay is a low power device. It will handle up to 30 volts AC or DC at up to 1 amp so it will switch low voltage alarms directly. For switching power loads an intermediate relay and power supply should be used such as shown above.

*Note: The Eco Sensors data logger is wired to plug into the jack on the bottom of the C-30ZX.

RECOMMENDED ACCESSORIES

OG-2 Ozone Source Calibrator Pocket-size battery-operated micro generator produces .1 ppm for calibration checking purposes.

EE-2 Environmental Enclosure For dust and water protection in industrial and agricultural environments and where the public may be near the instrument. $120 \times 180 \times 90 \text{ mm} (5" \times 7" \times 3")$ with a clear polycarbonate gasket front cover.

DL Series Data Loggers Tiny battery-operated data loggers record data for later analysis in PCs.

RAP-7800 Remote Alarm Panel In rugged PVC enclosure. Feature 90 dB audio alarm, red alarm strobe light, 4-20 mA analog signal loop (for building control systems), and digital readout.

More information about our products and applications at: www.ecosensors.com