Project No. DRTC-2008-HAZOP-02

NCFST Ozone Delivery System

Leader: Ted Rekart

Scribe: Ted Rekart

Meeting Location: NCFST Moffet
Center, Chicago, IL

Meeting Days: 2

Unit: Ozone Delivery System

June 2008

Air Liquide Ozone Delivery System for Destruction of Microorganisms. Collaborative effort between Air Liquide DRTC and NCFST

Table 1 Drawings Used in the Analysis

Type	Number	Title	Revision	Revision Date	Document Location
Procedure	MC-SA-105-SOP002-V01	Emergency Shutdown Procedures	V-01		
Procedure	MC-SA-105-SOP003-V01	Disconnecting the Oxygen and Nitrogen Cylinders	V-01		
Procedure	MC-SA-105-SOP004-V01	Ozone System General Safety Procedures	V-01		
Procedure	MC-SA-105-SOP005-V01	Setting Needle Valves for the Ozone System	V-01		
Procedure	MC-SA-106-SOP001-V01	Ozone System Startup and shutdown Procedure	V-01		
Drawing	P&ID	H1 Series Ozone Analyzer	-	6/20/2008	
Drawing	P&ID	Ozonia Ozat Model CFS-1A Ozone Generator	-	6/20/2008	
Drawing	PFD/P&ID	Ozone Design system	1	6/9/2008	

Table 2 Team Members

First Name	Last Name	Role	E-Mail	Phone	Job Title	Company
Bharat	Aluri		Baluri@iit.edu		Student	NCFST
Kerri	Cooper	Operator	kcooper4@iit.edu	803-669-3727	Student	NCFST
Todd	Diel		diel@iit.edu	703-563-8190	Safety Officer	NCFST
Steven	Fisher	Ozone Operations Expert	Steven.Fisher@AirLiquide.com	(708) 579-7709	Project Engineer	AL Consultant
Vishwesh	Kelkar		vkelkar@iit.edu		Student	NCFST
Vasuhi	Rasanayagam	AL Project Leader	vasuhi.rasanayagam@airliquide.com	302-286-5439	Scientist	American Air Liquide - DRTC
Ted	Rekart	RMR/HAZOP Leader	Theodore.Rekart@airliquide.com	302-286-5514	Safety Officer	American Air Liquide - DRTC
Claudia	Rodriguez	Co-PI	rodriguez@iit.edu	708-563-2052	Scientist	NCFST
Peter	Slade	PI	slade@it.edu	708-563-8172	Director of Education & Outreach	NCFST
Ed	Steiner	Lead Engineer	steiner@iit.edu	708-563-8273	Director of Facilities & Pilot Plant	NCFST
Lei	Wang	Operator	Lwang59@iit.edu		Student	

Table 3 Action Items

Type	No.	Action	Status	Responsibility	Date Complete	References
Recommendation	1	Add relief valve between NV1 and BV1 to outside building	Completed	Cooper/Slade	6/12/2008	1.1 High Oxygen Flow — Ozone Generator & Oxygen supply 1.4 High ozone generator temperature — Ozone Generator & Oxygen supply
Recommendation	2	Ensure standard operating procedure sets secondary side of regaulator to zero before opening oxygen valve.	Completed	Kerri Cooper, Lei Wang	6/17/2008	1.1 High Oxygen Flow — Ozone Generator & Oxygen supply 1.2 Low oxygen flow — Ozone Generator & Oxygen supply
Recommendation	3	Verify standard operating procedure precaition on adequate oxygen supply before startup	Completed	Cooper/Wang	6/17/2008	1.2 Low oxygen flow — Ozone Generator & Oxygen supply
Recommendation	4	Ensure standard operating procedure addresses venting and purging the buffer tank when experiment is completed and before any maintenance.	Completed	Coopr/Wang	6/10/2008	1.3 Reverse oxygen flow — Ozone Generator & Oxygen supply
Recommendation	5	Add flow meter to cooling water flow to ozone generator and verify that the standard operating procedure addresses verifying cooling water flow prior to ozone generator startup	Completed	Coopr/wang	6/10/2008	1.4 High ozone generator temperature — Ozone Generator & Oxygen supply
Recommendation	6	Verify standard operating procedure valve alignments	Completed	Cooper/Wang	6/10/2008	1.6 High ozone pressure at Generator Outlet — Ozone Generator & Oxygen supply
Recommendation	7	Verify standard operating procedure addresses nitrogen purging of anaerobic reaction tank with BV4 closed prior to inserting samples	Completed	Cooper/wang	6/10/2008	2.4 Misdirected ozone flow — Ozone Humidification Line
Recommendation	8	Outlet routinely checked for ambient ozone concentration	Completed	Cooper/wang	6/10/2008	3.1 High ozone concentration exiting column — MnO2 Ozone Destruction Column
Recommendation	9	Add MnO2 Destruct column thermocouple, determine operating limits and standard operating procedure response	Completed	Cooper/wang	6/10/2008	3.3 High destruct column temperature — MnO2 Ozone Destruction Column
Recommendation	10	Ensure standard operating procedure sets flow to ozone analyzer to recommended flow setting	Completed	Cooper/Wang	6/17/2008	4.1 High ozone flow — Ozone Analyzer

Type	No.	Action	Status	Responsibility	Date Complete	References
Recommendation	11	Add internal process flow diagram for ozone analyzer	Completed	Cooper/Wang	6/19/2008	4.8 Low ozone pressure — Ozone Analyzer
Recommendation	12	Add internal process flow diagram for ozone generator	Completed	Cooper Wang	6/13/2008	1.1 High Oxygen Flow — Ozone Generator & Oxygen supply
Recommendation	13	Standard operating procedure should contain steps to verify nitrogen flow	Completed	Cooper/wang	6/13/2008	5.2 Low/no nitrogen flow — Nitrogen supply
Recommendation	14	Add check valve at exit of ozone generator before BV7 & BV2	Completed	Cooper/Steiner	6/10/2008	5.4 Misdirected nitrogen flow — Nitrogen supply
Recommendation	15	Add to standard operating procedure procedure to calculate scrubber capacity and monitor ozone effluent gas stream accordingly	Completed	Cooper/Wang	6/17/2008	6.1 High ozone concentration exiting KI destruction tank — Anaerobic Reaction Tank
Recommendation	16	Evaluate hood set up and install velocimeter	Completed	Cooper/Steiner	6/16/2008	7.1 High flow — Fume Hood
Recommendation	17	Standard operating procedure needs to have emegency response to turn off all gas supplies	Completed	Cooper/Wang	6/16/2008	7.2 Low/no flow — Fume Hood
Recommendation	18	Verify nitrogen supply and room flooding calculations	Completed	Cooper/Steiner	6/17/2008	7.2 Low/no flow — Fume Hood
Recommendation	19	Standard operating procedure and P&ID's need revised	Completed	Cooper/wang/Slade	6/18/2008	

Table 4 List of Sections

No.	Type	Name	Description	Status	Drawings
1	Line/Pipe	Ozone Generator & Oxygen supply	Oxygen Cylinder, supply tubing and components including Ozone Generator		PFD/P&ID
2	Line/Pipe	Ozone Humidification Line	Line from Ozone Generator to the inlet of the Anaerobic Reaction Tank, including the Buffer Tank, Humidification tank and Excess Water Tank		PFD/P&ID
3	Tank/Vessel	MnO2 Ozone Destruction Column	Ozone destruction column (MnO2) and bypass lines to the column and valves BV2 & BV3		PFD/P&ID
4	Other	Ozone Analyzer	In-line Ozone Analyzer including valve NV2		PFD/P&ID
5	Line/Pipe	Nitrogen supply	Purge Nitrogen supplies for the anaerobic reaction tank and the ozone buffer tank through humidification train to the anaerobic reaction tank		PFD/P&ID
6	Tank/Vessel	Anaerobic Reaction Tank	Anaerobic reaction tank and KI destruction tank		PFD/P&ID
7	Other	Fume Hood	Fume hood containing experimental setup		

Table 5 for use in PHA Risk Matrix Used in Analysis

	0 No bodily injury, no damage to environment, no damage to eqiupment or production.	1 Minor injury, moderate damage with no durable effect on environment, or damage to small and medium-sized eqiupment, or a brief loss of production (several hours)	2 Serious injury, serious damage to environment but may be corrected, or damage to large equipment or lost of production (several days)	3 Potential victim, serious and durable damage to environment, or damage to very large items of equipment or extended loss of production (several weeks to months)	4 Major accident with potentiality of several victims, ecological catastrophe or massive destruction of facilities or total lost of production (permanent shutdown)
4 Frequent Event occur more than once every 10 years.	04 Low risk	14 Studies to minimize risk must be conducted	24 Unacceptable risk	34 Unacceptable risk	44 Unacceptable risk
3 Possible Event occurs between every 10 to 1000 years	03 Low risk	13 Low risk	23 Studies to minimize risk must be conducted	33 Unacceptable risk	43 Unacceptable risk
2 Rare Has happended to structures, static equipment, or redundant equipment	02 Low risk	12 Low risk	22 Low risk	32 Studies to minimize risk must be conducited	42 Unacceptable risk
1 Very rare The event requires the occurrence of two rare events	01 Low risk	11 Low risk	21 Low risk	31 Low risk	41 Studies to minimize risk must be conducted
0 Improbable The event has never occurred	00 Low risk	10 Low risk	20 Low risk	30 Low risk	40 Low risk

Company: DRTC-NCFST	**Plant:**	**Site:**	**Unit:** Ozone Delivery System	**System:**
Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)				

No.: 1	Oxygen Cylinder, supply tubing and components including Ozone Generator								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
1.1	High Oxygen Flow	Oxygen pressure regulator failure Valve misalignmemt	Loss of containment	2	3	23	2	22	Oxygen regulator Needle valve Hood & Ventilation
			Potential for internal damage to in-line component	2	3	23	2	22	
			High ozone pressure at Generator Outlet (see 1.6)	2	3	23	2	22	
			High ozone flow - Ozone Humidification Line (see 2.1)						
			High ozone pressure - Anaerobic Reaction Tank (see 6.5)						
1.2	Low oxygen flow	Closed valve High pressure downstream Low pressure upstream Oxygen supply depletion	High temp in Ozone generator	1	4	14	3	13	Automatic high temp shutdown of ozone generator
			High ozone generator temperature (see 1.4)						
			Low ozone pressure at generator outlet (see 1.7)						
			Low/no ozone flow - Ozone Humidification Line (see 2.2)						
			Low/no ozone flow - Ozone Analyzer (see 4.2)						
1.3	Reverse oxygen flow		Line Pressurized	0	4	04	4	04	
1.4	High ozone generator temperature	High ambient temperature Loss of cooling water No oxygen flow (see 1.2)	High temperature in ozone generator	1	4	14	3	13	Automatic high temp shutdown of ozone generator
			Low ozone pressure at generator						

No.: 1	Oxygen Cylinder, supply tubing and components including Ozone Generator								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
			outlet (see 1.7)						
1.5	Low ozone generator temperature	Low cooling water temperature	Condensation of water causing electrical arcing	2	2	22	2	22	Only tap water used for cooling
1.6	High ozone pressure at Generator Outlet	High Oxygen Flow (see 1.1)							
		Valve misalignment							
1.7	Low ozone pressure at generator outlet	Low oxygen flow (see 1.2)							
		High ozone generator temperature (see 1.4)							

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No.: 2 — Line from Ozone Generator to the inlet of the Anaerobic Reaction Tank, including the Buffer Tank, Humidification tank and Excess Water Tank

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
2.1	High ozone flow	humidity tank and excess water release tank glass stoppers loose High Oxygen Flow - Ozone Generator & Oxygen supply (see 1.1)	system depressurization	0	3	03	3	03	Experiment is in a ventilated hood
			release of ozone	2	3	23	2	22	
			High ozone pressure (see 2.5)						
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
2.2	Low/no ozone flow	Valve misalignment Low oxygen flow - Ozone Generator & Oxygen supply (see 1.2)	Loss of production and efficiency	1	2	12	1	11	Trained personnel & standard operating procedure
			Low/no ozone flow - Ozone Analyzer (see 4.2)						
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						
2.3	Reverse ozone flow	Humidity tank and excess water release tank glass stoppers loose	system depressurization	0	3	03	3	03	Hood and ventilation Isolation valve BV4
			release of ozone	2	3	23	2	22	
2.4	Misdirected ozone flow	valve misalignment	No safety consequences						
2.5	High ozone pressure	High ozone flow (see 2.1) High destruct column pressure - MnO2 Ozone Destruction Column (see 3.5) High ozone pressure - Anaerobic Reaction Tank (see 6.5)	Buffer tank relief valve releases ozone	2	3	23	2	22	Hood and ventilation
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
2.6	Low ozone pressure	Low ambient temperature Low temperature upstream	No additional safety related consequences						

No.: 2	Line from Ozone Generator to the inlet of the Anaerobic Reaction Tank, including the Buffer Tank, Humidification tank and Excess Water Tank								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
		Low ozone pressure at generator outlet - Ozone Generator & Oxygen supply (see 1.7)							
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						

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No.: 3 Ozone destruction column (MnO2) and bypass lines to the column and valves BV2 & BV3

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
3.1	High ozone concentration exiting column	Wet column causing channelling Water/water vapor in column (see 3.7)	Reduced ozone destruction	2	2	22	1	21	Check valve CV1 prevents wet gas backflowing to destruction column Dry gas feed used for ozone generation
			Water/water vapor in column (see 3.7)						
3.2	Low ozone concentration	No safety related issues							
3.3	High destruct column temperature	Normal operating situation as ozone destruction is exothermic	Fire and burn hazard	2	3	23	2	22	Themocouple on column exit gas to monitor temperature
3.4	Low column temperature	No safety related consequences	Freezing of water						
			Low pressure						
3.5	High destruct column pressure	Column inlet or exit screens plugged Water/water vapor in column (see 3.7)	High ozone pressure - Ozone Humidification Line (see 2.5)						
3.6	Low column pressure	No safety related consequences							
3.7	Water/water vapor in column	High ozone concentration exiting column (see 3.1)	High ozone concentration exiting column (see 3.1)						
			High destruct column pressure (see 3.5)						

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No.: 4	In-line Ozone Analyzer including valve NV2								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
4.1	High ozone flow	Valve misalignment High ozone pressure (see 4.7)	Inaccurate ozone analyzer readings	0	4	04	3	03	Trained Operator & SOP
			overpressure of ozone analyze with possible ozone release	2	2	22	1	21	
			High ozone pressure (see 4.7)						
4.2	Low/no ozone flow	Valve misalignment	No safety related consequences						
		Low oxygen flow - Ozone Generator & Oxygen supply (see 1.2)							
		Low/no ozone flow - Ozone Humidification Line (see 2.2)							
4.3	Reverse ozone flow	Valve misalignment	No safety related consequence						
4.4	Misdirected ozone flow	No credible cause							
4.5	High ozone temperature	No credible causes not already analyzed							
4.6	Low ozone temperature	No credible cause							
4.7	High ozone pressure	High ozone flow (see 4.1)	High ozone flow (see 4.1)						
4.8	Low ozone pressure	No credible safety cause							

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No.: 5 — Purge Nitrogen supplies for the anaerobic reaction tank and the ozone buffer tank through humidification train to the anaerobic reaction tank

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
5.1	High nitrogen flow	Valve misalignment (pressure regulator second stage set two high High gas flow - Anaerobic Reaction Tank (see 6.7)	lift relief in anaerobic reaction tank with possible ozone release	2	3	23	2	22	Hood and ventilation Training & standard operating procedure
			High nitrogen pressure (see 5.7)						
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						
5.2	Low/no nitrogen flow	Valve misalignment or regulator secondary stage not set properly Empty supply cylinder Low nitrogen pressure (see 5.8) Low/no gas flow - Anaerobic Reaction Tank (see 6.8)	Partial or no purge of ozone with potential ozone release	2	3	23	2	22	Hood and ventilation
			Low nitrogen pressure (see 5.8)						
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						
5.3	Reverse nitrogen flow	No credible cause							
5.4	Misdirected nitrogen flow	Operator error -- valve misalignment	Nitrogen backflow in to the ozone generator	1	3	13	2	12	Trained Operator & standard operating procedure Valve to be added to outlet of ozone generator
5.5	High nitrogen temperature	No credible cause							
5.6	Low nitrogen temperature	No credible cause							
5.7	High nitrogen pressure	High nitrogen flow (see 5.1)	Potential ozone release through the relief valve on buffer tank	2	3	23	2	22	Hood and ventilation
			Potential ozone release through the relief valve on anaerobic reaction tank	2	3	23	2	22	

No.: 5	Purge Nitrogen supplies for the anaerobic reaction tank and the ozone buffer tank through humidification train to the anaerobic reaction tank								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
			Lifting of glass stopper on humidity tank and excess water relaease tank releasing ozone	2	3	23	2	22	
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
5.8	Low nitrogen pressure	Low/no nitrogen flow (see 5.2)	Low/no nitrogen flow (see 5.2)						
5.9	Loss of Nitrogen containment	Cylinder or cylinder valve failure	Oxygen deficient atmosphere	3	2	32	1	31	Ambient oxygen monitor

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No.: 6	Anaerobic reaction tank and KI destruction tank								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
6.1	High ozone concentration exiting KI destruction tank	Depleted KI	ozone release	2	3	23	2	22	Hood and ventilation Ambient ozone analyzer Nitrogen flooding calculation shows 17% oxygen is possible in static room from one cylinder of nitrogen
6.2	Low ozone concentration	Valve misalignment	No significant safety consequences						
6.3	High ozone temperature	No credible cause	no significant safety consequences						
6.4	Misdirected ozone flow	Standard operating procedure not followed with nitrogen tank changeout not in proper sequence	Ozone release	2	3	23	2	22	Standard operating procedure to purge anaerobic reaction tank with BV4 closed Ventilation system and hood
6.5	High ozone pressure	High Oxygen Flow - Ozone Generator & Oxygen supply (see 1.1)	Potential release through the relief valve	2	3	23	2	22	Hood & ventilation
			High ozone pressure - Ozone Humidification Line (see 2.5)						
6.6	Low ozone pressure	No credible safety related issues							
6.7	High gas flow	High nitrogen flow - Nitrogen supply (see 5.1) High nitrogen pressure - Nitrogen supply (see 5.7) High ozone flow - Ozone Humidification Line (see 2.1) High ozone pressure - Ozone Humidification Line (see 2.5)	High nitrogen flow - Nitrogen supply (see 5.1)						
6.8	Low/no gas flow	Low/no nitrogen flow - Nitrogen supply (see 5.2) High nitrogen flow - Nitrogen supply (see 5.1) Low/no ozone flow - Ozone	Low/no nitrogen flow - Nitrogen supply (see 5.2)						

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No.: 6	Anaerobic reaction tank and KI destruction tank								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
		Humidification Line (see 2.2) Low ozone pressure - Ozone Humidification Line (see 2.6)							

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No.: 7	Fume hood containing experimental setup								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
7.1	High flow	Improper setup of hood openings	Backlfow and eddy currents into the room	2	3	23	2	22	Trained Operator and SOP
7.2	Low/no flow	Power failure	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI Ambient oxygen monitor
			High oxygen exposure to room occupants	2	3	23	2	22	
			nitrogen flooding of work space	3	3	33	1	31	
		Operator error, not turning in the hood blower	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI
		Mechanical failure of hood blower	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI Ambient oxygen monitor Hood velocimeter Limited nitrogen supply relative to room volume
			High oxygen exposure to room occupants	2	3	23	2	22	
			nitrogen flooding of work space	3	3	33	1	31	
		Operator error, PVC curtain misplacement	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI Ambient oxygen monitor Hood velocimeter Limited nitrogen supply relative to room volume

No.: 7	Fume hood containing experimental setup								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
			High oxygen exposure to room occupants	2	3	23	2	22	
			nitrogen flooding of work space	3	3	33	1	31	
7.3	Reverse flow	No credible cause							
7.4	High temperature	No credible cause							
7.5	Low temperature	No credible cause							

Ozone Delivery System Binder

By: Kerri C. Cooper

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Date: 5/9/08

Abbreviations

P + ID	Parts and Instrumentation Diagram
NCFPD	National Center for Food Protection + Defense
MnO_2	Manganese Dioxide
KI	Potassium ~~Dioxid~~ Iodide
BV	Ball Valve
NV	Needle Valve
PRV	Pressure Release Valve
PR	Pressure Regulator
CV	Check Valve
O_3	Ozone
SS	Stainless Steel
SOP	Standard Operating Procedure
PVDF	Polyvinylidene Fluoride
HAZOP	Hazard and Operability
H_2SO_4	Sulfuric Acid
BA	Bacillus anthracis (sterne)
BC	Bacillus cereus
BT	Bacillus thuringiencis

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Date: May 9, 2008

NCFPD Overall Project Plan

Abstract/Executive Summary: The proposed continuation of this project aims to optimize the cleaning process by comparison of existing methods with different cleaning agents (product and process specific) in conjunction with high-power ultrasound, to further facilitate removal of food residues to improve sanitizer/sterilant efficiency. Further steps will be taken to scale-up the application of the vaporized/gaseous sterilants (vaporized hydrogen peroxide, ozone, chlorine dioxide and formaldehyde) to eliminate remaining spores in food residues attached to processing surfaces after cleaning treatments.

Optimization of this potential non-invasive cleaning-sanitizing strategy for removal and inactivation of *B. anthracis* (Sterne strain) could provide a less destructive alternative for decontamination of food processing equipment in the event of a biological threat in food processing facilities. This application could potentially save both time for remediation and offset the potential financial impact caused by the loss of critical capital items.

Primary Goal: To validate the effects of various cleaning and liquid/gaseous sanitizing protocols for decontamination of food processing equipment and facilities with spores of several potential surrogate *Bacillus* species including *B. anthracis* Sterne strains.

Objectives: To compare the effects of various vaporized and gaseous sterilants to sanitize cleaned surfaces in scaled-up, simulated but near-real world situations.

Introduction and Highlights: In a previous NCFPD-funded project, we investigated: (1) the formation of "biofilms" consisting of *Bacillus* spores (*B. cereus* ATCC 21281 and *B. thuringiensis* ATCC 33680) embedded in complex food matrices on different food contact surfaces and the ability of cleaning regimens to remove spores in food (Xie *et al.*, 2007); and (2) the effects of liquid and gaseous/vaporized sanitizers (or sterilants) to inactivate spores on clean and non-clean surfaces (Oh *et al.*, 2007).

Sanitizing technologies to be included in this phase of the study include: vaporized hydrogen peroxide (Heckert *et al.*, 1997; Rogers *et al.*, 2005), ozone (Kim *et al.*, 1999; Aydogan and Gurol, 2006), chlorine dioxide (Kreske *et al.*, 2006b; Ryu and Beuchat, 2005), and paraformaldehyde (Ackland *et al.*, 1980). Although ozone has been used as an anti-fungal fumigant applied to stored cereal crops (Allen *et al.*, 2003; Wu *et al.*, 2006), its use for facility fumigation has been rarely reported. Pan *et al.* (1992) reported that application of 600ppm ozone for 6 hours might be effective for routine sterilization of cages, bedding, clothing, and other materials in laboratory animal facilities. Khadre and Yousef (2001) compared the sporicidal actions of hydrogen peroxide and ozone, and found the former to be less effective against *Bacillus* spores even at 10,000x higher concentration.

<u>Sanitizer challenge</u>

All gaseous sterilants will be prepared and applied to simulated food contact surfaces according to manufacturer's instructions. Various concentrations and contact/application times will be assessed to determine appropriate end points and limits for detection (as described below). The following gaseous sterilants, or their equivalents, will be considered: Ozone (American Air Liquide - proprietary on-site generation)

References

Peter J. Slade, Ph.D. Year 4 Project Proposal for: Validation of Methods for Decontamination of Food Processing Equipment and Facilities Deliberately Contaminated with *Bacillus* Spores (2007).

Funding: National Center for Food Safety and Protection (NCFPD)

Kimi C. Cooper 5/9/08

Date: May 9, 2008

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Abstract/Executive Summary: The proposed continuation of this project aims to optimize the cleaning process by comparison of existing methods with different cleaning agents (product and process specific) in conjunction with high-power ultrasound, to further facilitate removal of food residues to improve sanitizer/sterilant efficiency. Further steps will be taken to scale-up the application of the vaporized/gaseous sterilants (vaporized hydrogen peroxide, ozone, chlorine dioxide and formaldehyde) to eliminate remaining spores in food residues attached to processing surfaces after cleaning treatments.

Optimization of this potential non-invasive cleaning-sanitizing strategy for removal and inactivation of *B. anthracis* (Sterne strain) could provide a less destructive alternative for decontamination of food processing equipment in the event of a biological threat in food processing facilities. This application could potentially save both time for remediation and offset the potential financial impact caused by the loss of critical capital items.

Primary Goal: To validate the effects of various cleaning and liquid/gaseous sanitizing protocols for decontamination of food processing equipment and facilities with spores of several potential surrogate *Bacillus* species including *B. anthracis* Sterne strains.

Objectives: To compare the effects of various vaporized and gaseous sterilants to sanitize cleaned surfaces in scaled-up, simulated but near-real world situations.

Introduction and Highlights: In a previous NCFPD-funded project, we investigated: (1) the formation of "biofilms" consisting of *Bacillus* spores (*B. cereus* ATCC 21281 and *B. thuringiensis* ATCC 33680) embedded in complex food matrices on different food contact surfaces and the ability of cleaning regimens to remove spores in food (Xie *et al.*, 2007); and (2) the effects of liquid and gaseous/vaporized sanitizers (or sterilants) to inactivate spores on clean and non-clean surfaces (Oh *et al.*, 2007).

Sanitizing technologies to be included in this phase of the study include: vaporized hydrogen peroxide (Heckert *et al.*, 1997; Rogers *et al.*, 2005), ozone (Kim *et al.*, 1999; Aydogan and Gurol, 2006), chlorine dioxide (Kreske *et al.*, 2006b; Ryu and Beuchat, 2005), and paraformaldehyde (Ackland *et al.*, 1980). Although ozone has been used as an anti-fungal fumigant applied to stored cereal crops (Allen *et al.*, 2003; Wu *et al.*, 2006), its use for facility fumigation has been rarely reported. Pan *et al.* (1992) reported that application of 600ppm ozone for 6 hours might be effective for routine sterilization of cages, bedding, clothing, and other materials in laboratory animal facilities. Khadre and Yousef (2001) compared the sporicidal actions of hydrogen peroxide and ozone, and found the former to be less effective against *Bacillus* spores even at 10,000x higher concentration.

Sanitizer challenge

All gaseous sterilants will be prepared and applied to simulated food contact surfaces according to manufacturer's instructions. Various concentrations and contact/application times will be assessed to determine appropriate end points and limits for detection (as described below). The following gaseous sterilants, or their equivalents, will be considered: Ozone (American Air Liquide - proprietary on-site generation)

References

Peter J. Slade, Ph.D. Year 4 Project Proposal for: Validation of Methods for Decontamination of Food Processing Equipment and Facilities Deliberately Contaminated with *Bacillus* Spores (2007).

Funding: National Center for Food Safety and Protection (NCFPD)

Kim C. Coo[illegible] 5/9/08

Date: 5/9/08

Project Description

In 2001 a terrorist attack of distributing anthrax through the mail brought attention to the country that the use of spores as a weapon was feasible and the realization that steps have not been added in response to these possibilities. The food industry as a result began taking great strides in order to put methods in place that would allow for fast and easy response if any of these attacks were to ever affect their facilities. Anthrax which is the bacterial spore *Bacillus anthracis* can cause serious harm and even be deadly upon exposure. In case of any attacks within the food industry it is important to be prepared with steps such as a cleaning and sanitization process to quickly and efficiently eliminate the anthrax from any surfaces that it may come in contact with the spores. In order to simulate the contact surfaces, coupons made of stainless steel 316, glazed tile, Teflon, polypropylene, and rubber will be used as representatives of food contact surfaces. Due to *Bacillus anthracis'* extremely virulent nature, surrogate strains will be used in this study that may be similar to *Bacillus anthracis*. These strains are *Bacillus cereus* ATCC 21281, *Bacillus thuringiencis* ATCC 33680 , and *Bacillus anthracis* (Sterne strain). Food matrices that will be used are pancake mix, peanut butter, infant formula, vegetable oil, and a sucrose solution.

The use of ozone as a sanitizer for inactivation of *Bacillus* spores has been explored using different surfaces, but few studies have explored how it affects spores embedded in different food matrices. This is important because in most real world applications food matrices come in contact with many of the surfaces in the facilities and have the potential to become hardened and difficult to remove. Therefore a determination of the parameters, such as the contact time and ozone concentration required to inactivate the spores embedded in the food are very important in sanitizing. In order to determine this, an ozone delivery system must be designed to safely deliver and destroy the ozone.

Due to ozone's instability and potential deadly affect if inhaled at a high dosage, it is important to construct a process to safely deliver and destroy the ozone. A system must be built for controlled delivery and destruction of the ozone to the spores inoculated on the coupons.

Keri C. Cooper 5/9/08

Date: 5/9/08

Project Description

In 2001 a terrorist attack of distributing anthrax through the mail brought attention to the country that the use of spores as a weapon was feasible and the realization that steps have not been added in response to these possibilities. The food industry as a result began taking great strides in order to put methods in place that would allow for fast and easy response if any of these attacks were to ever affect their facilities. Anthrax which is the bacterial spore *Bacillus anthracis* can cause serious harm and even be deadly upon exposure. In case of any attacks within the food industry it is important to be prepared with steps such as a cleaning and sanitization process to quickly and efficiently eliminate the anthrax from any surfaces that it may come in contact with the spores. In order to simulate the contact surfaces, coupons made of stainless steel 316, glazed tile, Teflon, polypropylene, and rubber will be used as representatives of food contact surfaces. Due to *Bacillus anthracis'* extremely virulent nature, surrogate strains will be used in this study that may be similar to *Bacillus anthracis*. These strains are *Bacillus cereus* ATCC 21281, *Bacillus thuringiencis* ATCC 33680 , and *Bacillus anthracis* (Sterne strain). Food matrices that will be used are pancake mix, peanut butter, infant formula, vegetable oil, and a sucrose solution.

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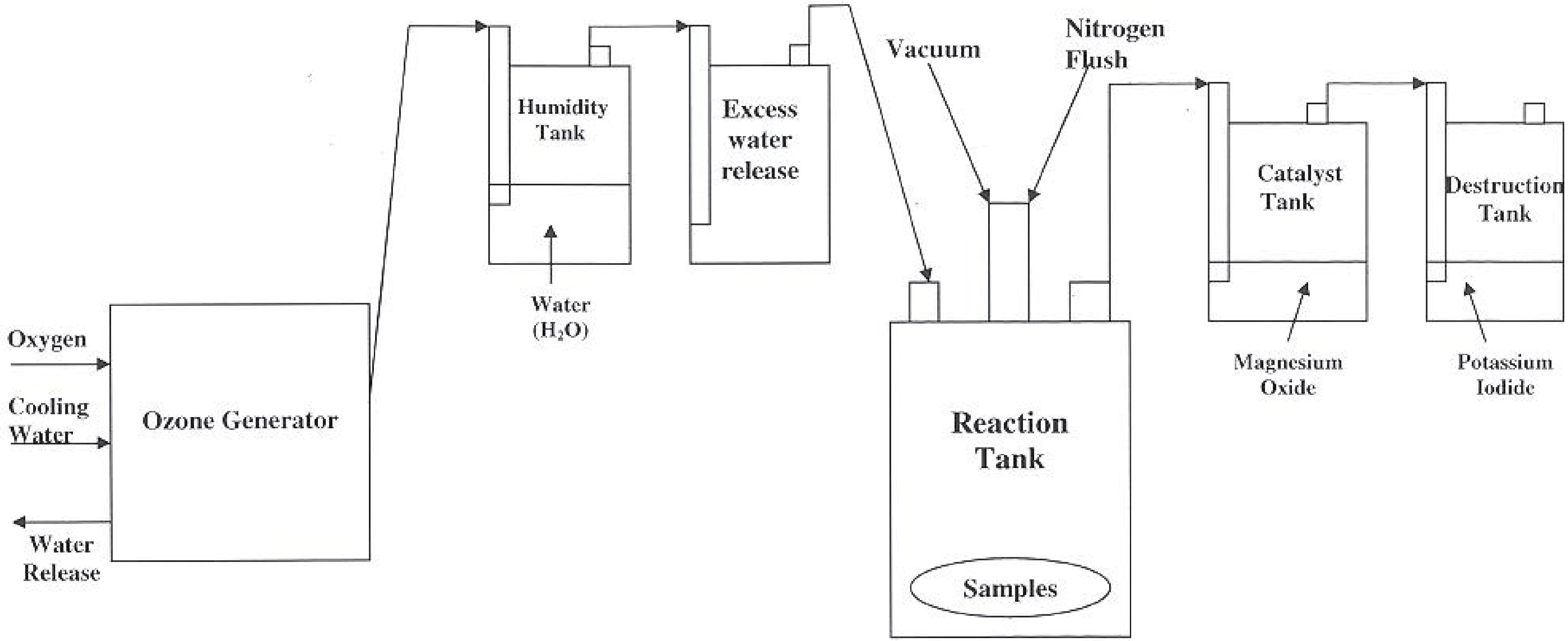
Keri C. Cooper 5/9/08

- Date '9/08

Ozone Design System - Draft 1



Kim C. Cooper 3/9/08

Date: 5/9/08

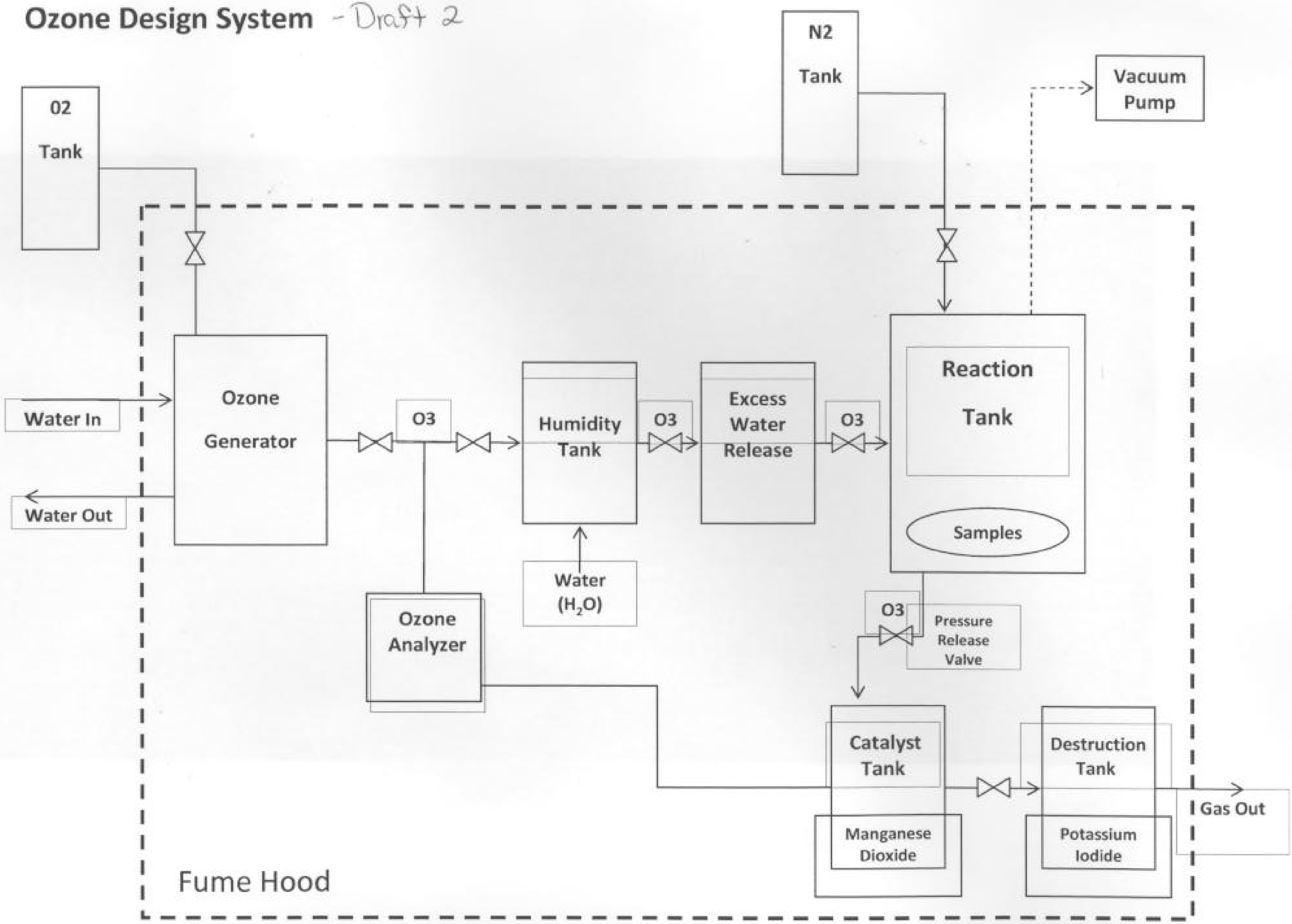
Changes for Draft 1

- Valves needed to be added
  - Still not sure what types are required
- Add fume hood to the design
  - All items that are producing ozone should be under the fume hood
- Draw oxygen and nitrogen tanks

Kurri C. Coa 5/9/08

Date: 5/9/08

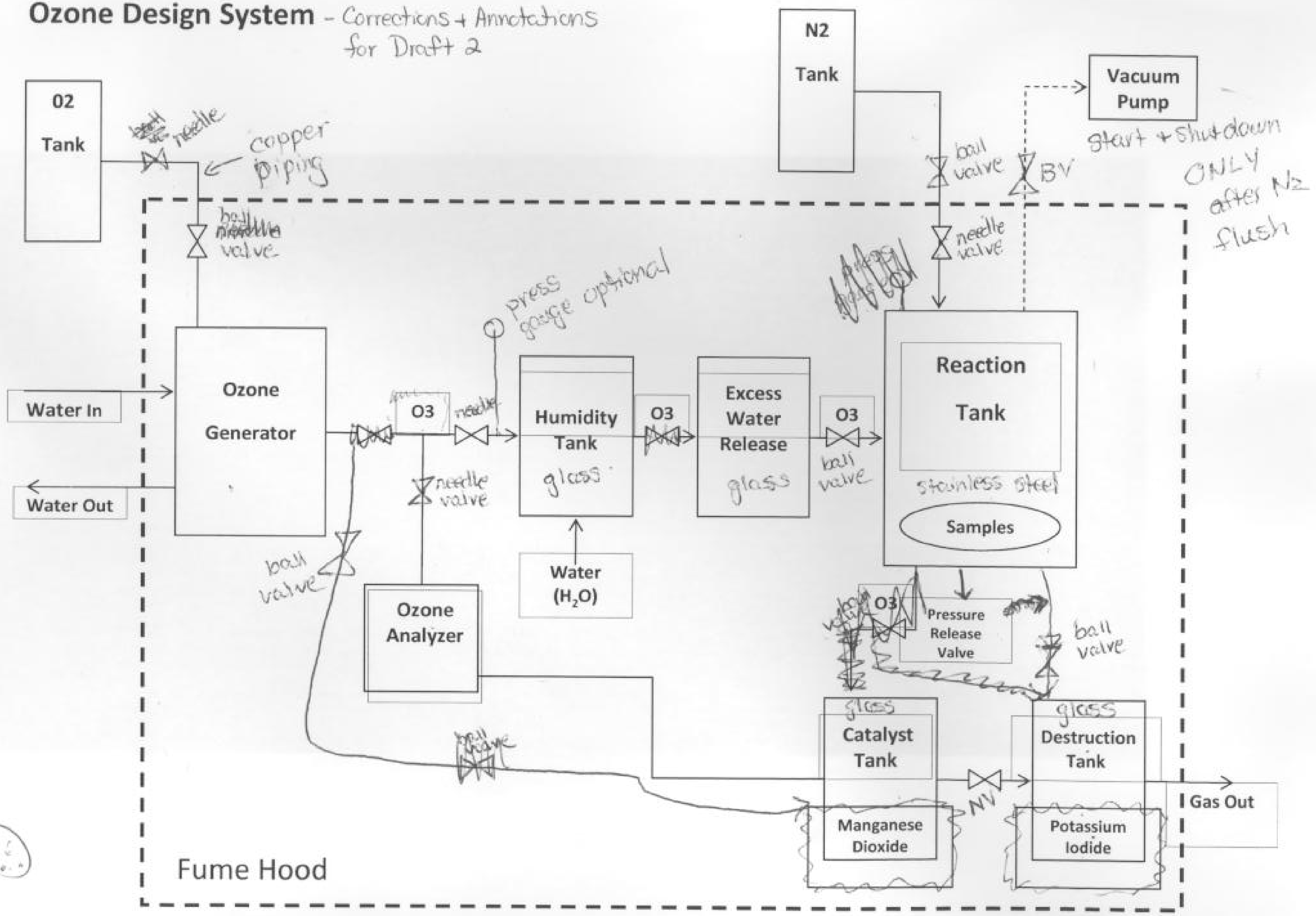
Ozone Design System - Draft 2



Kimi C. Cooper 5/9/08

Date 5/9/08

Ozone Design System - Corrections + Annotations for Draft 2



Kiri C. ~~Coo~~ 5/9/08

Stainless Steel 304 tubing throughout except between oxygen tank + ozone generator

Changes for Draft 2 of Ozone Design System

April 1, 2008

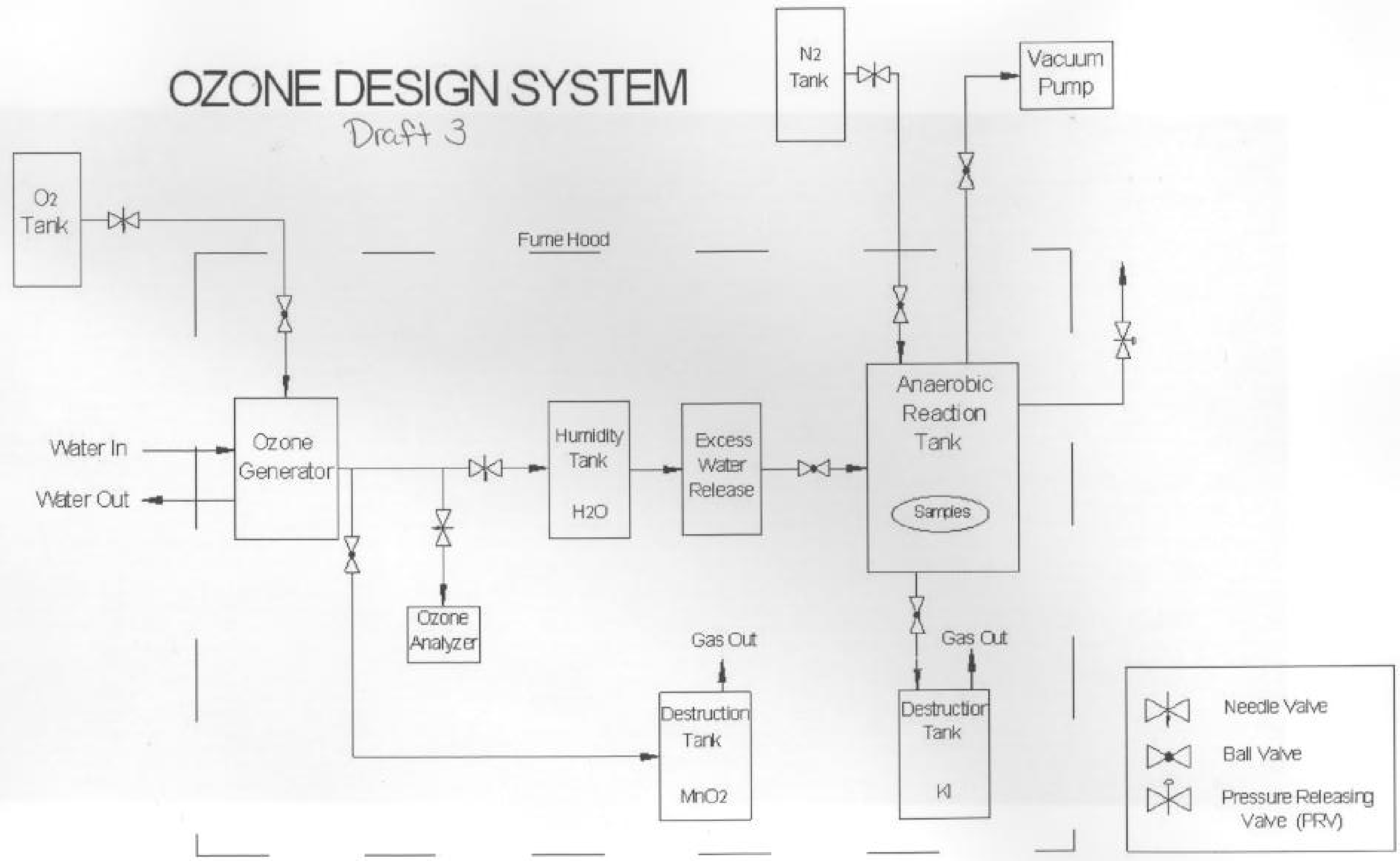
- Types of valves needed
  - Needle valves – controls the flow of the system
  - Ball valves – allows for and stops flow of the gas through the system
  - Pressure release valves – can be set for the maximum pressure allowed for the tanks and will pop and release pressure if the maximum pressure is reached
- Add pressure gauge to monitor pressures before the ozone flows into the humidity tank
  - Humidity tank can only withstand 5psig
- Add a line connecting the ozone generator to the MnO_2 destruction tank
  - This will be used to destroy the ozone until the desired concentration is achieved
- The line connecting the anaerobic tank to the MnO_2 destruction tank is removed
  - MnO_2 is in the powdered form and the ozone flowing from the anaerobic tank is humidified which would wet and destroy the MnO_2
- The line connecting the catalyst tank and the destruction tank is removed
  - If there is backflow from the destruction tank, the MnO_2 will become wet and destroyed
- Stainless steel tubing should be used throughout the system since it will withstand ozone
  - Check Valex company for tubing
- Copper tubing can be used to connect the oxygen and ozone generator
  - Cheaper and does not require ozone compatibility

Kimi C. Cooper 5/9/08

Date: 5/ /2008

OZONE DESIGN SYSTEM

Draft 3



Kimi C. Casey 5/9/08

Date: 5/9/08

Changes from Draft 3

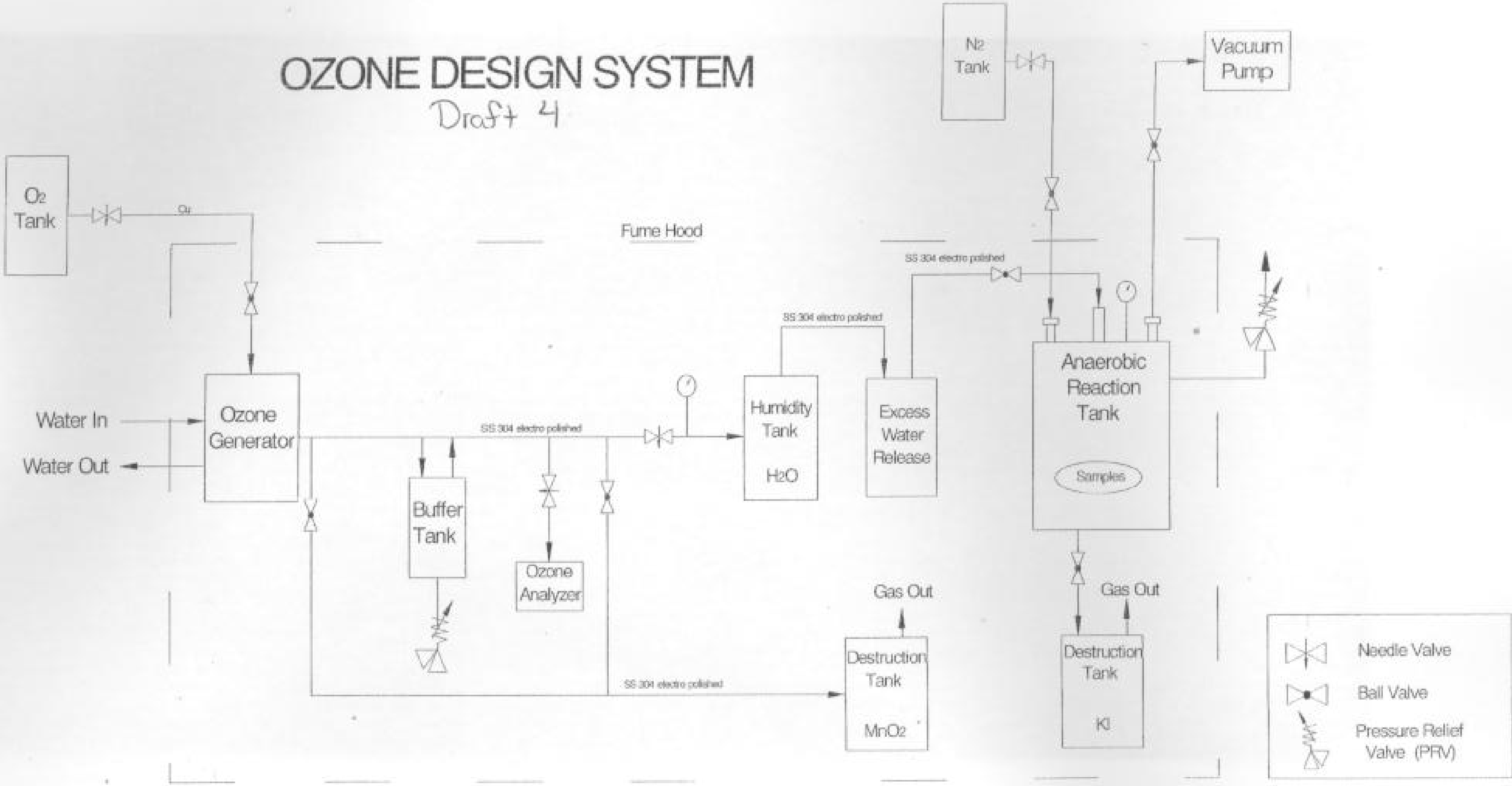
- The type of tubing used throughout the system needs to be identified
- The gauge is added before the humidity tank
- Addition of the buffer tank
    - When using the vacuum pump all the air will be removed from the anaerobic reaction tank. If the valve is opened to allow the ozone to flow into the reaction tank after vacuum pumping the ozone will flood into the tank and possibly over pressurize the tank.
    - The buffer tank will therefore act as a preventative measure by holding the ozone and then supplying the anaerobic tank with controlled flow
- Pressure release valve
    - Incorrect symbol so the PRV was modified on the anaerobic tank
    - Added to the buffer tank to prevent over pressurization

Kuri C. Coaz

Date: 9/08

OZONE DESIGN SYSTEM

Draft 4



Kimi C. Coop 5/9/08

Date: 5/9/08

Comments on Proposed P&ID/PFD for
Ozone Delivery System for NCFST
April 3, 2008

The draft P&ID/PFD and equipment list for the proposed ozone delivery system for NCFST was reviewed and the following comments resulted:

P&ID/PFD Diagram

1. The diagram need to annotate the nominal temperatures, pressures and flows which the various portions of the system will experience during normal operation.
2. Process line materials should be noted where the material of construction changes.
3. Relief valve and safety devices within the diagram should be annotated with their set points.
4. The relief valve on the anaerobic reactor should be shown to relieve into the fume hood.
5. If the vacuum pump is not necessary please remove it from the system, along with any other components which have been provided in support of the vacuum pump.
6. Show the two stage regulators on the oxygen and nitrogen tanks.
7. Move the pressure gauge immediately upstream of the humidifier vessel to upstream of the manual valve.
8. Number all valves, pressure, gauges, and components.
9. Indicate O3 analyzer in the environment

Equipment List

1. Provide the pressure rating and ozone production rates for the ozone generator.
2. Provide the dimensions and pressure rating of the buffer tank
3. Provide the pressure and flow rating for the ozone analyzer.
4. Provide the correct dimensions, and pressure rating for the anaerobic reactor.
5. Provide pressure ratings and flow limitations for the gas washing bottles.
6. The tubing, valves and fittings are of different sizes. Please verify the correct size for the tubing, valves and fittings.
7. Verify compatibility of all equipment and materials with the oxygen/ozone process stream.

- These comments were submitted from Vasuhi Rasanayagam and Ted Rekart of Air Liquide as a result of our conference call for the P+ID.

Kuni C. Cogez 5/9/08

Date: 5/9/08

Conference Call with Air Liquide - Personal Notes
April 3, 2008

Discussion

1. Ozone Design
    a. Needs to be a Parts and Instrumentation Diagram (P&ID)
        i. Temperature, pressure, and flow rates displayed
        ii. Materials used for the process line (tubing type and size)
        iii. Set points for the relief valves
        iv. Move release valve within the fume hood
        v. Remove vacuum pump
        vi. Show pressure regulators on the oxygen and nitrogen tanks
        vii. Move pressure gauge to the buffer tank
        viii. Number the valves and gauges
2. Equipment List
    a. Ozone generator
        i. Pressure rating
        ii. Ozone production rates
    b. Buffer tank
        i. Dimensions
        ii. Pressure rating
    c. Ozone analyzer
        i. Pressure rating
        ii. Flow rate
    d. Anaerobic reactor
        i. Correct dimensions
    e. Gas washing bottles
        i. Pressure ratings
        ii. Flow limitations
        iii. Correct dimensions
    f. Correct the valve sizes
        i. Should be ¼ in
    g. Make sure all equipment is oxygen/ozone compatible
3. Hazard and Operability Study (HAZOP)
    a. Brief explanation was given on the purpose of conducting this study
    b. A P&ID with the correct information is required in order to correct develop the plan
4. Fittings
    a. Air Liquide will provide the fittings to be oxygen cleaned
    b. Submit a list of needed fittings
5. Air Liquide will send an example P&ID

Next Meeting

- Will determine the date of the next meeting upon progress of the P&ID and equipment ordering
- Submit updated P&ID before next meeting

Kevin C. Coap 5/9/08

Date: 5/9/08

Changes for Draft 4

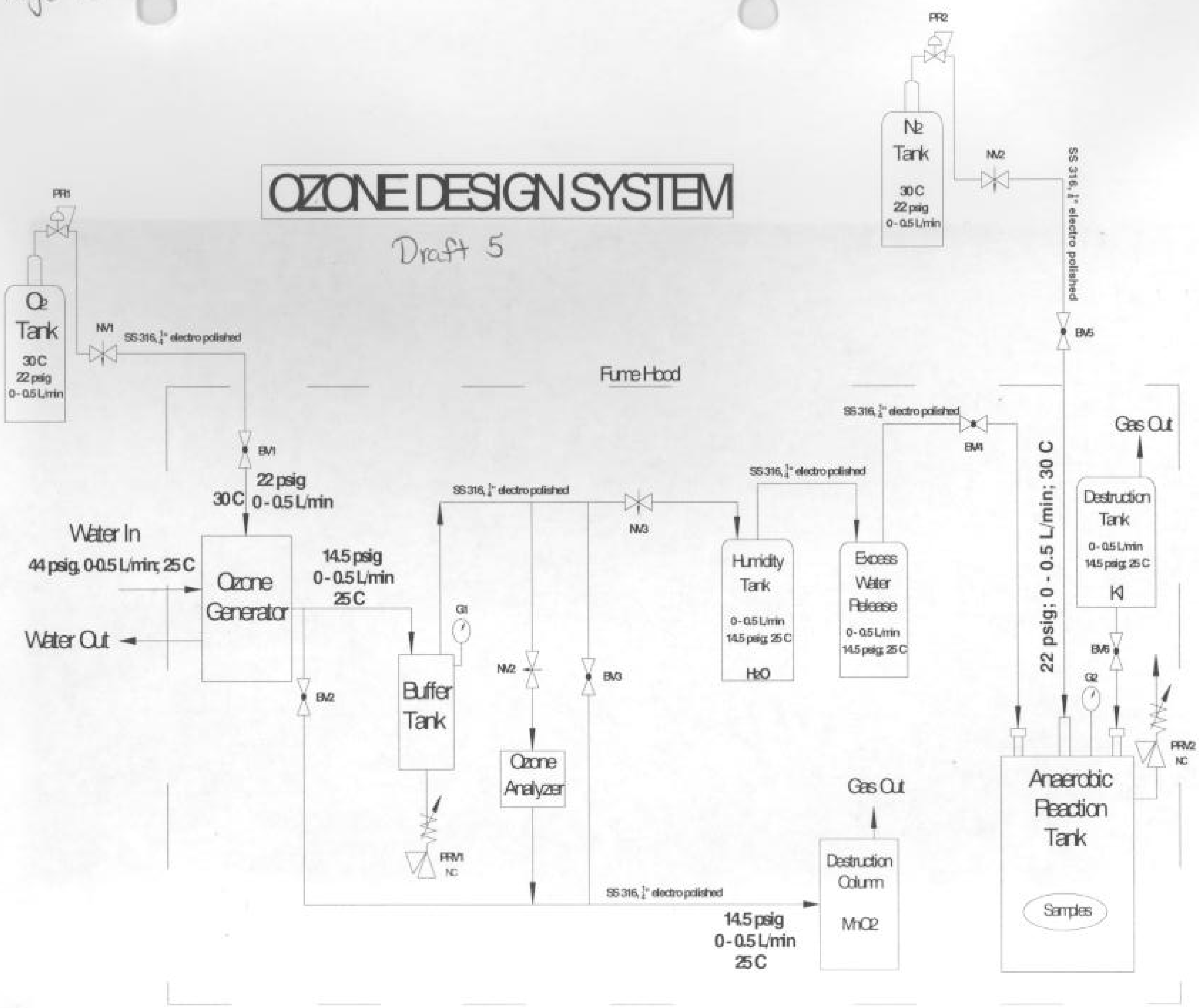
- Removed the vacuum pump
  - From past studies it was found that spores are not affected by vacuum pumping therefore it was removed because it was more of a hazard to the system (oxygen compatibility of oil, over pressurizing the anaerobic reaction tank)
- Buffer tank still kept in place to effectively monitor the flow of the system
- Pressure gauge moved to the buffer tank
  - Pressure can be monitored in the tank and adjusted to specified flow by the needle valve placed before the humidity tank
- The PRV connected to the anaerobic tank is releasing outside the fume hood.
  - Moved to release under fume hood
- Line added for the ozone passing through the ozone analyzer and sent to the MnO_2 destruction tank
- The temperatures, flow rates, and pressures were added to indicate parameters along the system line (determined by the maximum conditions allowed for the parts)

Kerri C. Coop 5/9/08

Date: 5/9/08

OZONE DESIGN SYSTEM

Draft 5



	Needle Valve (NV)
	Ball Valve (BV)
	Pressure Relief Valve (PRV)
G1, G2	Pressure Gauge
	Pressure Regulator (PR)
NC	Normally Closed

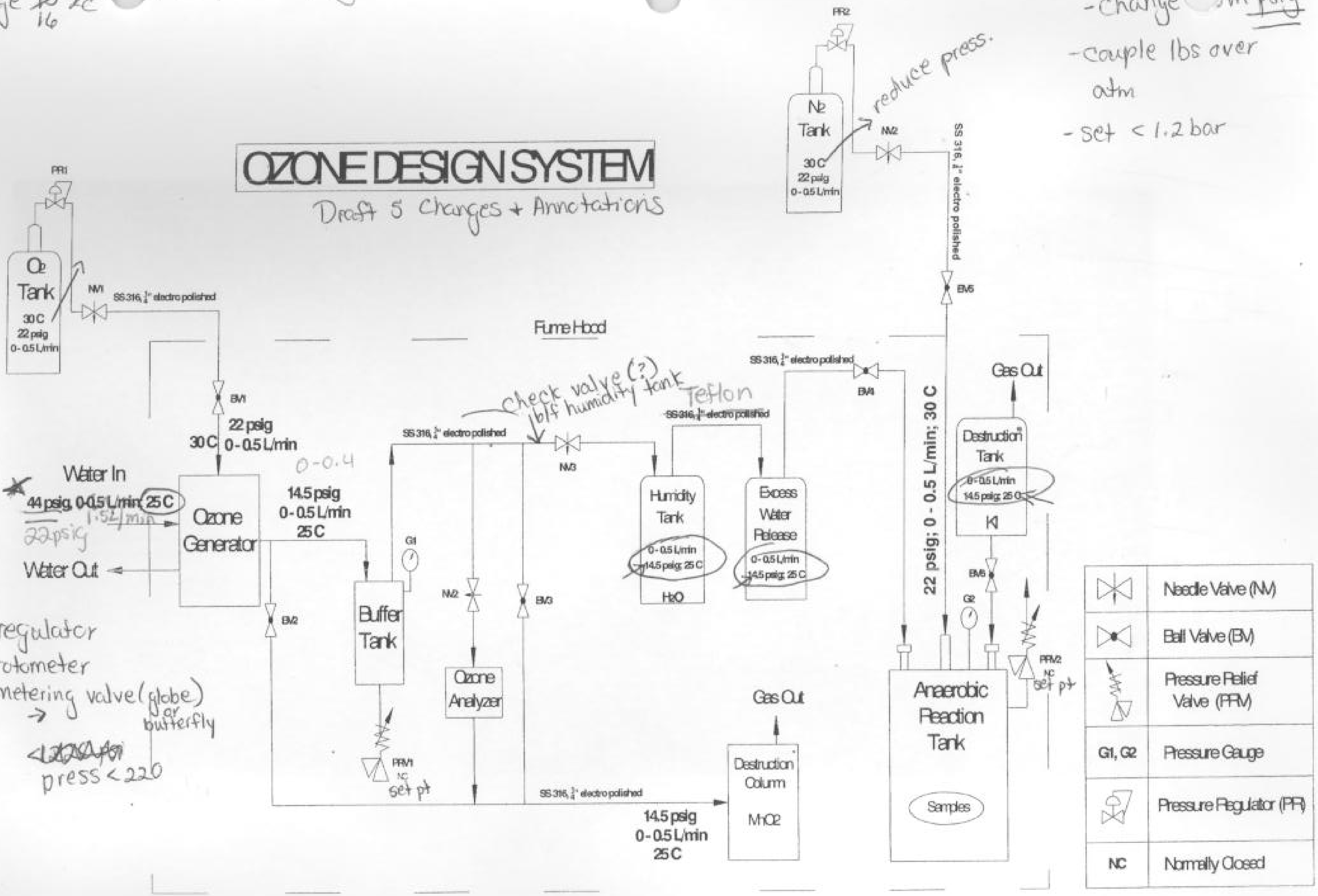
Kuii C. Ooga 5/9/08

Page ~~15~~ 16 ~~zc~~

14.5 psi → 1 psig

Date: 5/9/08

- change [illegible] m psig
- couple lbs over atm
- set < 1.2 bar



- regulator
- rotometer
- metering valve (globe or butterfly) →

~~<[illegible]~~ press < 220

Kui C. Cog 5/9/08

Date: 5/9/08

Comments on Proposed P&ID/PFD for
Ozone Delivery System for NCFST
April 15, 2008

The revised P&ID/PFD and equipment list for the proposed ozone delivery system for NCFST was discussed and the following comments were made:

P&ID/PFD Diagram

1. The pressures, temperatures and flows indicated inside the compressed gas cylinders for oxygen and nitrogen should be moved to the appropriate lines downstream of the pressure regulators.
2. A check valve or some device should be placed downstream of the buffer tank and upstream of valve NV3 to prevent moisture from back flowing into the ozone analyzer.
3. The line and vessel pressures should be changed to psia vice psig to reflect the actual operating conditions.
4. The relief valve set points should be indicated for PRV1 and PRV2.
5. Verify the ozone generator flow rates of 0.04 – 0.96 Nm^3/h are compatible with the desired system flow rate of 0.5 L/min.

- Comments submitted by Vasuhi Rasanayam and Ted Rekart of Air Liquide.

Kevin C. Cooper 5/9/08

Date: 5/9/08

Conference Call with Air Liquide - My Personal Notes
April 15, 2008

Discussion

1. Changes to be made to the P&ID
    a. Move the pressures and flow indicated inside the gas tanks to the line
    b. Check on the flow rates of the gas coming from the generator → conversions do not seem to be correct
    c. Add a check valve before needle valve (NV3) to prevent back flow of the humidified ozone into the MnO_4 destruction tank
    d. The pressures are not correct and should be labeled psia or converted correctly to psig
    e. Indicate pressure release valve set points (PRV1, PRV2)
2. Equipment list
    a. Modify symbols for pressure and temperature ranges
    b. No removal or additions
3. Anaerobic Jar Lid
    a. Requested for Air Liquide to make adjustments to the jar lid
        i. Fill original holes and make them ¼ in NPT threads
        ii. Need an oxygen clean guage as well
    b. Air Liquide agreed to make the changes and we will ship the item to them
4. Oxygen cleaning of the parts
    a. Buffer tank is dusty and needs to be cleaned
    b. Anaerobic tank needs to be cleaned
5. All items will be shipped to Air Liquide
    a. Buffer tank
    b. Anaerobic tank (cleaning and thread changes)
    c. Stainless Steel anaerobic tank (Chlorine dioxide unit)

Next Meeting

- Will meet again in 2 weeks (April 30, 2008)
- Submit updated P&ID before meeting
- Make changes to equipment list
- Continue ordering supplies
- Ship items to Air Liquide

Kuni C. Cooper 5/9/08

Date: 5/9/08

Changes for Draft 5

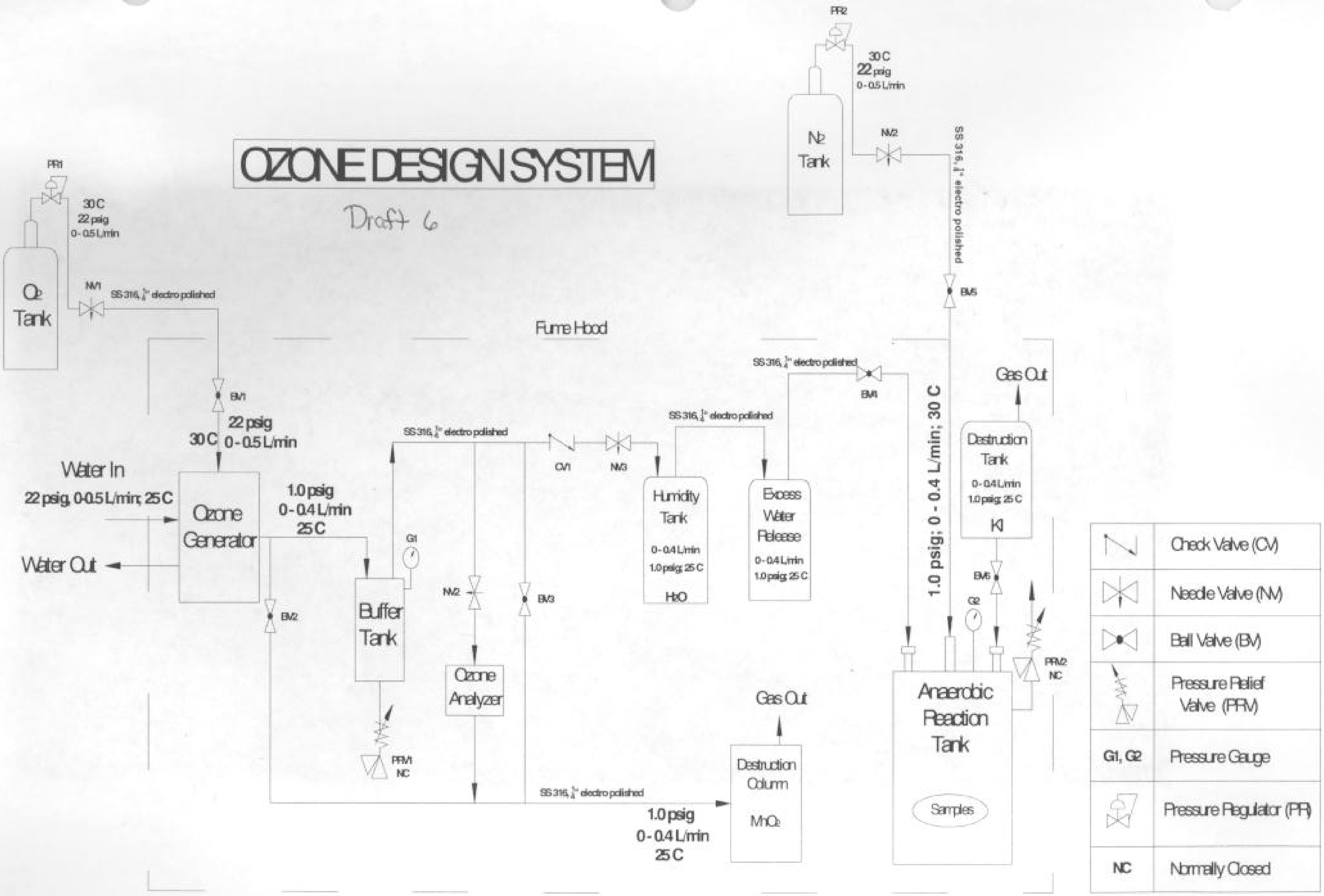
- Parameters inside the oxygen and nitrogen tank are incorrect
  - Those are the desired parameters for flow through the tubing
- The pressures are incorrect
  - The readings are atmospheric pressure and not gauge pressure
    - 14.5psia = 1.0psig
- All valves are labeled and numbered accordingly
  - This is to help for easy references during building, discussions, and operations
- Check valve added into the system before valve NV3
  - this is to prevent backflow of the humidified ozone to the MnO_2 destruction tank
- Pressure regulator for the nitrogen and ozone tanks shown in the line

Kun C. [signature] 5/9/08

Date: 5/9/08

OZONE DESIGN SYSTEM

Draft 6



Kimi C. Cooper 5/9/08

Date: 5/9/08

Conference Call with Air Liquide - My Personal Notes
May 2, 2008

Discussion

1. The P&ID is fine and there are no immediate changes to be made
    a. Vasuhi and Ted (Air Liquide) will proceed with the HAZOP
2. 3-A sanitary piping from McMaster Carr is fine to use
    a. As long as it is for a short length
    b. The item will be ordered immediately
3. Jar lid threads
    a. Email sent to Vasuhi concerning increasing the thread size to ½ in NPT for one connection → she will need to look into this in order to determine if it is possible
    b. If the thread will be ½ in then they will work to find the ½ in to ¼ in fitting (oxygen cleaned)
4. Oxygen cleaned parts
    a. Vasuhi is currently collecting the fittings
    b. Will hopefully ship next Friday (May 9, 2008)
5. Steve Fisher will be the local contact for the HAZOP
6. Need basic SOPs to write the HAZOP (startup and shutdown procedures)
    a. Email to Air Liquide on Friday, May 9, 2008
    b. Will receive their comments on May 14
7. Ozone Training
    a. Will be done the first week of June, either June 2 or 3
    b. Steve Fisher will be conducting the training
8. Team sheets should be done for the HAZOP
    a. Submit to Vasuhi with the names of the participants and their roles

Next Meeting

- Conference call on May 14, 2008
- Discuss the SOPs
- Finalize the date for the ozone training

Kerri C. Ca[illegible] 5/9/08

Date: 5/9/08

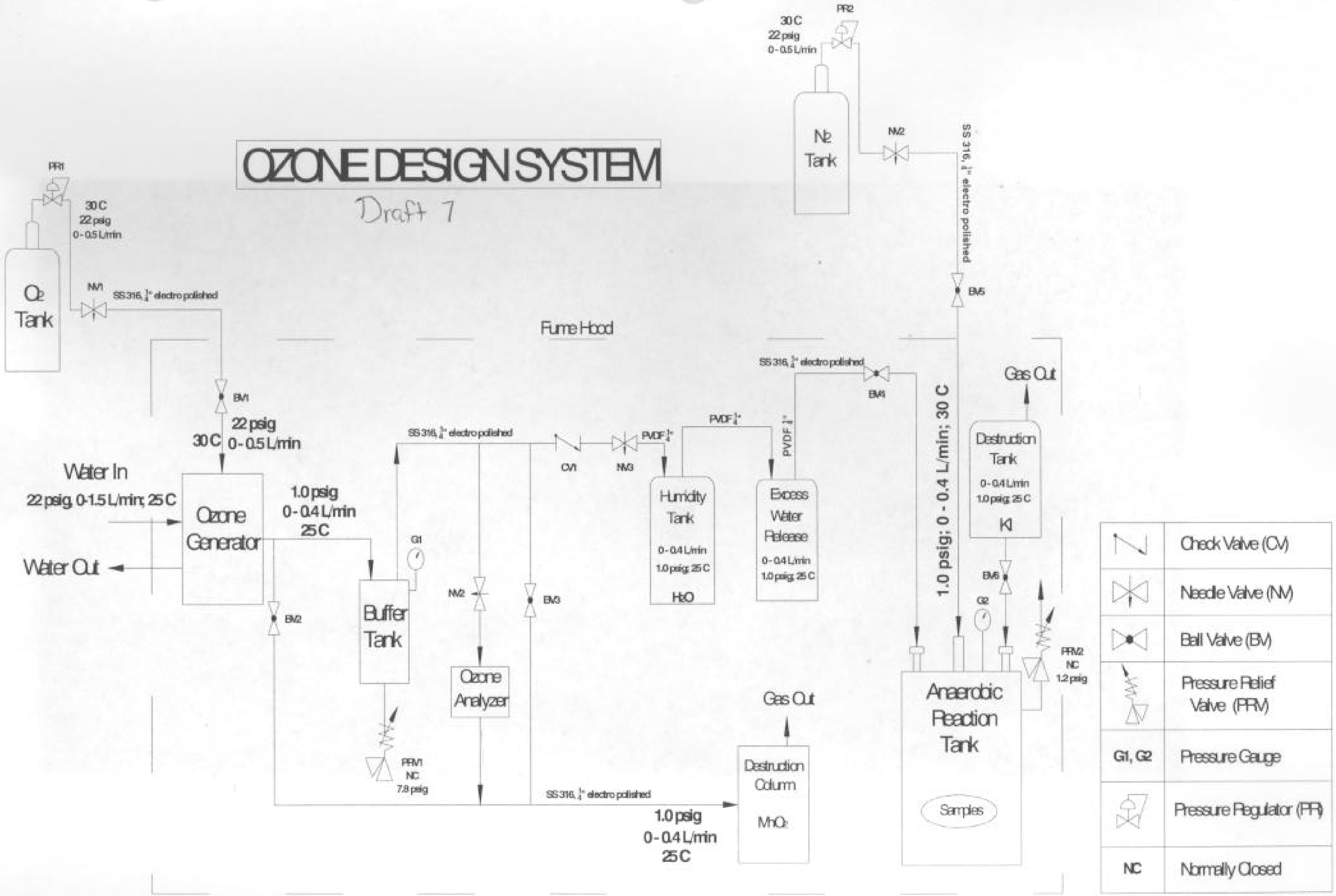
Changes for Draft 6

- Set points for PRV1 and PRV2 are added
- Water flow into the ozone generator increased to 1.5L/min
    - Correct conversion from the ozone generator manual
- Polyvinylidene fluoride (PVDF) tubing will be connected to the humidity tank and excess release tank
    - Tanks are glass and needs to be connected to flexible tubing
    - This tubing should also be ozone compatible as well

Kuri C. Cooper 5/9/08

Page ~~22~~ 23 KC

Date 5/9/08



Kimi C. Coo[illegible] 5/9/08

Page 24 Date: 5/9/08

MOFFETT CENTER	Page 1 of 2
Title: **Ozone System Shutdown Process**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP002-V01	Effective Date: May 9, 2008

1.0 Purpose and Scope

1.1 To safely shutdown and purge the ozone from the system

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Ozone is toxic to humans and should be contained and destroyed effectively

3.2 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned

3.3 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere

4.0 Equipment and Supplies

- Oxygen tank
- Nitrogen tank
- Ozone Generator (Ozonia/CFS-1A)
- Buffer tank (10gal pressure vessel)
- Ozone Analyzer (IN USA/H1)
- Ambient Ozone Analyzer (IN USA/IN2000L2-LC)
- Anaerobic Reaction Tank (Schütt Labortechnik/stainless steel)
- MnO_2 Destruction tank
- Humidity tank, excess water tank, and destruction tank (Ace Glass/500ml Gas washing bottle)
- Stainless Steel piping (Valex 316L, Specification 301, ¼ in)
- Ball Valves (Swagelok/SS-42GS4-SC11)
- Needle Valves (Swagelok/SS-4MG-SL-SC11)
- Pressure Release Valve (Swagelok/SS-RL3S4/SC11)
- Pressure Gauge (Blue Ribbon Sales & Service Corp/BR4001-4LD, oxygen cleaned)
- PVDF Tubing (McMaster-Carr/5390K342, 3-A sanitary tubing)
- Butterfly Valve (McMaster-Carr/4682K74, ½ in connections)
- Check Valve (Swagelok/SS-4C-1-SC11)
- Rotameter (Mcmaster-Carr/8051K17)
- Nylon Tubing (McMaster-Carr/5112K653, 3/8in OD)
- Oxygen Detector (McMaster-Carr/18995T14)
-

Kerri C. Cooper 5/9/08

Date: 5/9/08

MOFFETT CENTER	Page 2 of 2
Title: **Ozone System Shutdown Process**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP002-V01	Effective Date: May 9, 2008

5.0 Operational Procedure

5.1 Stopping Gas Flow

5.1.1 Turn off ozone generator by hitting the OFF command for POWER SUPPLY
  5.1.1.1 Screen should indicate: PSU OFF and PURGING time
  5.1.1.2 The background screen should also be orange
5.1.2 Allow for the equipment to purge the system with the feedgas and remove the residual ozone in the generator
5.1.3 Once this is completed the screen will turn green and read: PSU OFF and POWER 00000kw
5.1.4 Stop O_2 and water flow into the ozone generator
  5.1.4.1 Close valves BV1 and turn off water supply
5.1.5 Close valve BV4 to stop flow into Anaerobic Reaction Tank
5.1.6 Open valves to allow for ozone destruction
  5.1.6.1 Valves BV3, BV6
5.1.7 Allow ozone to flow into destruction tanks for predetermined time
  5.1.7.1 Monitor oxygen analyzer to make sure O_2 concentration is less that 23%
5.1.8 Close valves BV3

5.2 Flushing the System

5.2.1 Open valves to allow for nitrogen flush of the system
  5.2.1.1 BV 3-4, BV8-9 and NV3
5.2.2 Allow nitrogen to flush through the system for the specified time
5.2.3 Close all valves before the nitrogen tank
  5.2.3.1 BV3-4, BV8-9, NV3
5.2.4 Open valves BV5 and set NV4 to remove humidity in the anaerobic reaction tank
5.2.5 Allow nitrogen to flush through the tank for a specified time
5.2.6 Close valves BV5-6 and NV4

6.0 References and Supporting Documents

Ozone Material Safety Data Sheet (MSDS). <http://www.ozoneapplications.com/info/ozone_msds.htm>.

Ozone Safe Working Practices. Work SafeBC. <http://www.worksafebc.com/publications/health_and_safety/by_topic/assets/pdf/ozone_bk47.pdf>.

Steiner, Ed. National Center for Food Safety and Technology Engineer.

The Hazards of ozone & Ozone Gas. <http://www.inspect-ny.com/sickhouse/OzoneHazards.htm>.
Wikepedia. Ozone. 2007. <http://en.wikipedia.org/wiki/Ozone>.

Kerri C. Cooper 5/9/08

MOFFETT CENTER	
Title: **Ozone System Startup Process**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP001-V01	Effective Date: May 2008

1.0 Purpose and Scope

1.1 To safely startup the ozone system for delivery to the anaerobic reaction tank

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Ozone is toxic to humans and should be contained and destroyed effectively

3.2 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned

3.3 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere

4.0 Equipment and Supplies

- Oxygen tank
- Nitrogen tank
- Ozone Generator (Ozonia/CFS-1A)
- Buffer tank (10gal pressure vessel)
- Ozone Analyzer (IN USA/H1)
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- Anaerobic Reaction Tank (Schütt Labortechnik/stainless steel)
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- Pressure Gauge (Blue Ribbon Sales & Service Corp/BR4001-4LD, oxygen cleaned)
- PVDF Tubing (McMaster-Carr/5390K342, 3-A sanitary tubing)
- Butterfly Valve (McMaster-Carr/4682K74, ½ in connections)
- Check Valve (Swagelok/SS-4C-1-SC11)
- Rotameter (Mcmaster-Carr/8051K17)
- Nylon Tubing (McMaster-Carr/5112K653, 3/8in OD)
- Oxygen Detector (McMaster-Carr/18995T14)

Kerri C. Cooper 5/9/08

5.0 Operational Procedure

5.1 Flushing the Anaerobic Reaction Tank

5.1.1 Ensure all valves are closed

a. PR 1-2, NV 1-4, BV 1-6, PRV 1-2

5.1.2 Set valve NV2 to desired flow

5.1.3 Set PR2 to 1.0 psig

5.1.4 Open BV5 and BV6

5.1.5 Allow nitrogen to flow through the Anaerobic Reaction Tank to flush any excess ozone into KI destruction tank

5.1.6 Close valves PR2, NV2, and BV5

5.1.7 Close valve BV6

5.2 Setting Parameters for the Ozone Generator

5.2.1 Turn water supply on

5.2.2 Switch on (MAIN-ON) ozone generator and wait until the screen turns green

a. Screen should read: MAINS ON→ SYSTEM CHECK

5.2.3 Select desired language (English, German, Latin, French, Italian, Spanish)

a. Continually press SET POINT until desired language is displayed

5.2.4 Wait 5 seconds to allow the ozone generator to set the language

5.2.5 Ensure SET POINT is set to LOCAL

a. Screen should read % LOCAL, PSU OFF

5.2.6 Set valves for correct operating conditions

a. PR1: 22psig

a. NV1: 0.5 L/min

5.2.7 Set generator to POWER SUPPLY ON

a. Screen should read PSU ON

5.2.8 Open valve BV1 and BV2

5.2.9 Turn oxygen supply on

5.2.10 Set gas flow by adjusting the HCV 301 dial located on the front of the generator

5.3 Achieving Desired Concentration

5.3.1 Close valve BV2

5.3.2 Open valve BV3 and BV7

5.3.3 Allow ozone to flow through until desired concentration is achieved

a. Refer to the H1 ozone analyzer reader in the system

5.3.4 Set valve NV2

5.3.5 Close valves BV8 and NV2

5.3.6 Allow buffer tank pressure to reach 5psig

5.3.7 Open valves BV4 and BV8, set valve NV3 and allow ozone to flow into the anaerobic tank for desired time

Kerri C. Cooper 5/9/08

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Date: 5/9/08

5.3.8 Close valve BV3 once desired concentration is achieved
*Steps 5.3.7 and 5.3.8 should be done quickly and almost simultaneously

6.0 References and Supporting Documents

Ozone Material Safety Data Sheet (MSDS). <http://www.ozoneapplications.com/info/ozone_msds.htm>.

Ozone Safe Working Practices. Work SafeBC. <http://www.worksafebc.com/publications/health_and_safety/by_topic/assets/pdf/ozone_bk47.pdf>.

Steiner, Ed. National Center for Food Safety and Technology Engineer.

The Hazards of ozone & Ozone Gas. <http://www.inspect-ny.com/sickhouse/OzoneHazards.htm>.

Wikepedia. Ozone. 2007. <http://en.wikipedia.org/wiki/Ozone>.

Kerri C. Cooper 5/9/08

Date: 5/1~~3~~14/08

Changes for Draft 7

- Additional line added connecting the nitrogen tank to the buffer tank
  - This will allow for flushing the system with nitrogen upon completion of the experiment
  - If ozone is left in the system it could possibly corrode the pipes over time and allow for humidity buildup in the system which would adversely affect the material and the experiment
- Addition of valves
  - BV7 – while obtaining the desired ozone concentration the ozone will need to flow only into the MnO_2 destruction tank. This valves will control the flow of the ozone into the buffer tank
  - BV 8 – stops and allows the flow of the ozone from the buffer tank
  - BV9 and NV5 – controls the flow of the nitrogen for flushing the system
- Additional line connecting to the MnO_2 destruction column
  - Safety measure to make sure the ozone does not backflow into the generator or ozone analyzer

Kevin C. Ca~~oo~~ 5/14/08

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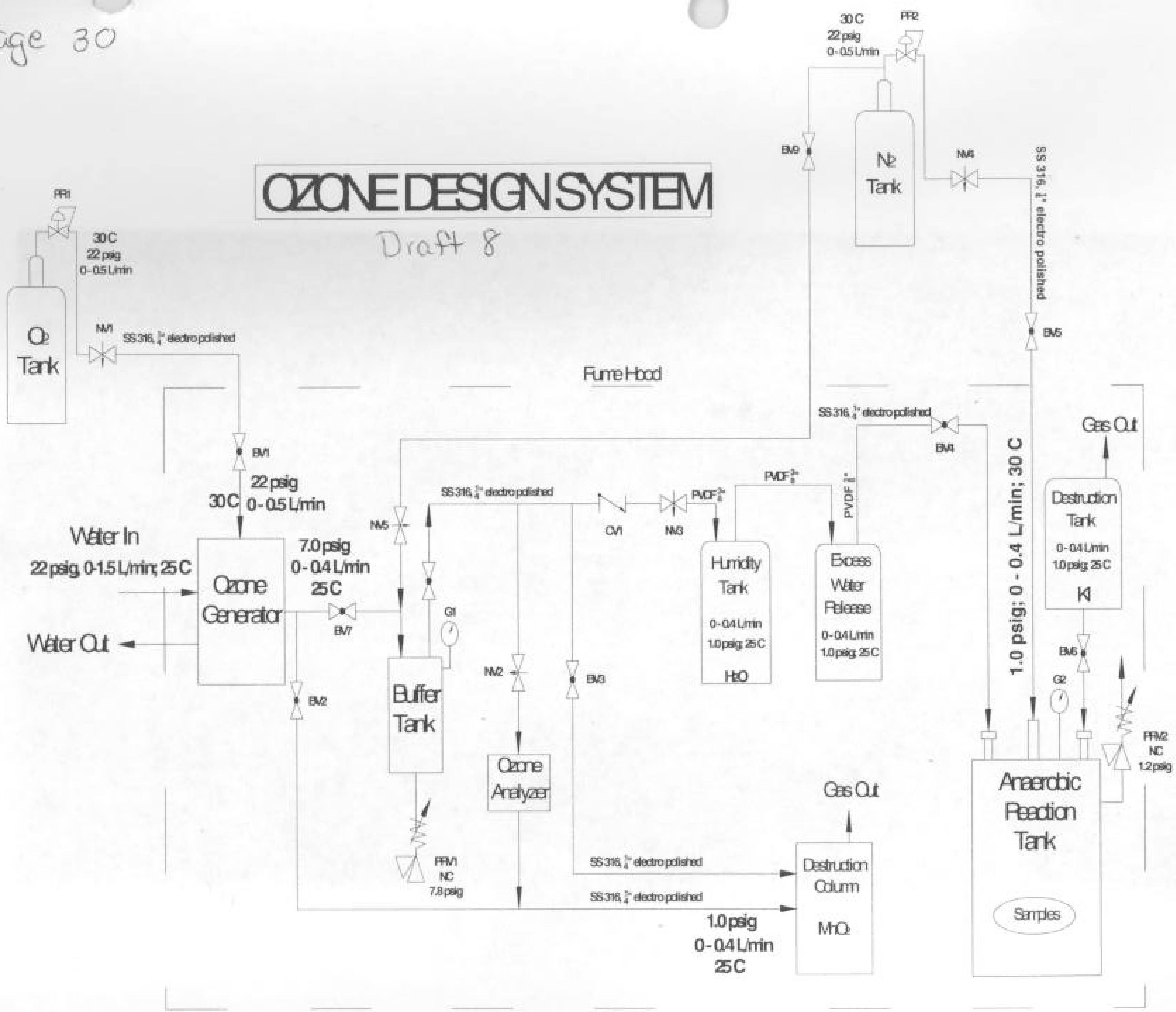
Date 5/~~9~~ 14/08

OZONE DESIGN SYSTEM

Draft 8



	Check Valve (CV)
	Needle Valve (NV)
	Ball Valve (BV)
	Pressure Relief Valve (PRV)
G1, G2	Pressure Gauge
	Pressure Regulator (PR)
NC	Normally Closed

Kerri C. Cog 5/14/08

 Date: 5/14/08

Conference Call w/ Air-Liquide Notes

Date: May 14, 2008

Attendees

Claudia Rodriguez
Kerri Cooper
Dr. Peter Slade
Ed Steiner

Absent

Vasuhi Rasanayagam
Ted Rekart

Discussion

- Need to finalize dates for ~~ele~~ KC training → June 2nd or 3rd
- Email Vasuhi and ask for comments on SOPs
- June 3rd best day for training
  → will it be all day training
  → what equipment is needed for training
- Pilot plant spot
  → steel toe covers (contact Todd)
  → wear hard hat
- Obtain ~~wire~~ KC pipe cutter and bender from Ed Steiner
- Electrical parts for setting up the system in Lab 105
  → will discuss with Ed what is needed

To Do List

1. Email Vasuhi
   → SOP comments
   → training date
   → equipment for training
2. Contact Todd concerning steel toe covers

Kerri C. Cooper 5/14/08

Date: ~~5~~ 6/9/08

Conference Call w/ Air-Liquide Notes
May 19, 2008

Attendees

Kerri Cooper
Dr. Peter Slade
Ed Steiner
Vasuhi Rasanayagam
Ted Rekart

Discussion

- combine shutdown and startup procedure
- add flowmeters into system for setting needle valves
- add procedure for setting needle valves
- verify flush time with ambient analyzer
- Vasuhi has shipped O_2 cleaned parts
  - → ferrules will be sent at the end of week
  - → destruct column contains MnO_2 already
- Training: June 2 + 3rd
  - → HAZOP will require 1 day
  - → Dr. Slade will send list of other trainings he would like to have
  - → Vasuhi will email a list of items she will need for training
- Ted reminded me of safe handling of O_2 cleaned parts
  - → don't leave out in atmosphere
  - → no oil or grease to be used
  - → will send list of O_2 compatible lubricants

To Do List

- update SOPs - discuss with Dr. Slade + Ed
- update P+ID
- email information to Air-Liquide

Kerri C. Cooper 8/19/08

~~5/27/08~~ KC
Date: 6/9/08

Meeting with Todd Diel
- Safety Officer Introduction

Date: 5/27/08

Submissions for Committee

- SOP → approval form
- P+ID

~~map~~
- attachments
  → HAZOP
- operational SOP (PPE, hazards, chemicals) ★ - reference startup + shutdown SOP
  - lab workers
  - leakage

Training - June 2 + 3, 2008
- cylinder
- O_2 cleaning
  → ~~cath~~ Dr. Slade will contact Vasuhi for materials list

- 1 wk for review after submission
- Attendees

Kerri Cooper
Dr. Peter Slade
Ed Steiner
Todd Diel

Kerri C. Cooper 5/27/08

Date: 6/9/08

General Safety Procedures
May 27, 2008
- discussed w/ Dr. Slade

1. Curtains for drawing O_2 through hood
2. Fan turned on + off
3. All components O_2 cleaned
4. PPE requirements
5. Lab access during experimentation
6. Monitor ambient analyzer
   → inhalation limits (OSHA)
7. Leak test
   → run N_2 through initially
8. Contingency procedures - what happens if leak occurs
9. Emergency Contacts
   → Dr. Slade
   → Ed Steiner
   → Kerri Cooper
10. Training
11. Handling O_2 cleaned parts
12. Chemical handling
    → disposal
    → interactions

Kerri C. Cooper

6/9/08

Training with Air Liquide

June 2, 2008

I. Cylinder Handling (presented by Ted Rekart)
 - Safe Storage, Handling + Use of Compressed Gas
- 3 types of gases
1. compressed
2. liquified
3. dissolved (acetylene)
- 3 classes of gases
1. flammable (acetylene, H^+, propane, butane)
2. non-flammable (N_2, argon, He, Air, CO_2, O_2)
3. poison gas (toxic + corrosive)

A. Cylinder Use
- know which gas you're using
- never use an adapter to make a cylinder fit a regulator
 → CGA stamp should match
- open valve slowly!
- leak check w/ soapy solution
 → NEVER use flame!
- close valves when not in use
- OSHA most cited violations
 → O_2 + flammable gases too close

B. Cylinder Storage
- storage based upon:
 → types of gases
 → full + empty cylinders
- Secure cylinder before removing valve
- Store in well venilated areas (<125°F)
- keep away from electrical circuits
- avoid excessive moisture

Kerri C-Cargo 6/9/08

6/9/08

Training with Air Liquide

C. Cylinder Handling
- do not roll or drag cylinders
- use hand truck to move cylinders
- keep valve protection caps in place while in storage, during handling, and transportation

D. Special Precautions for Flammable Gas
- no smoking near cylinder
- use spark proof tools
- flammable gases should be stored 20ft away from:
  - → oxidizers
  - → combustibles
  - → ignition sources
- Oxygen
  - ★ → keep away from oil + grease

E. Summary
- never use a gas unless you are trained in its use + understand the hazards
- use MSDS

II. Oxygen Cleaning (presented by Ted Rekart)
- Levels 1 + 2

A. Scope
- list cleaning agents

B. HSE considerations
- hydrocarbon contamination in presence of O_2 → explosion, fire, serious injuries
  - → handle with clean cotton white gloves
  - → could occur at any time
- entry into any confined space for inspection, cleaning shall require a Confined Space Entry Permit
- O_2 concentrations below 19.5% by vol. reduce the capacity to sustain life
- ~~s~~ KC

Kini C. Cage 6/9/08

6/9/08

Training w/ Air-Liquide

- solvent or cleaner spill emergencies
  - → notify management immediately
  - → refer to MSDS

B. Terminology

- O_2 component or sytem: any component or system that contains O_2 at a conc. of 23.5% or greater
- qualified person:
- aqueous cleaner: chemical composition containing any of the following
  - → diluted to a specified conc. w/ potable H_2O
- solvent: chemical not miscible in water + does not require dilution
- contaminants: loosely adherent rust, dirt, grit, grinding dust, metal filings
- preliminary cleaning: used to remove large amts of particles

C. O_2 Service + Oils + greases

- greases + oils must be compatible w/ service equipment

D. Air-Liquide Policy

- Level 1 - cleaned for O_2 service + oxidizing gases + mixtures
  - → cleaning solutions + solvents

E. Packing - Recommendations

- protect + labeled as ~~such~~ KC O_2 cleaned immediately to prevent contamination
- should not be in a resealable package

~~F. Labeling~~ KC → do not use duct tape

- if bag is broken must reclean item

F. Storage

- all components must be separated from dirty or uncleaned items

G. Documentation

- cleaning + inspection check sheet
- can use checklist to inspect incoming items

H. Level 1 + 2 Cleaning Solution

- Blue ~~C~~ KC J Gold

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Training w/ Air-Liquide

I Cleaning Techniques
- hand wiping
- swabbing
- immersion cleaning
- ultrasonic cleaning
- flushing
- hand tool cleaning
- power tool cleaning
- brushes w/ steel bristles are only permitted for use on carbon steel

J ~~H~~. Preliminary Cleaning Methods
- steam
- white-blast cleaning...

K ~~J~~ Minimum inspection
- odor test + visual inspection
- odor test + wipe test

★- only qualified persons

III. Oxygen Enrichment (given by Ted Rekart)

A. Properties of O_2
- normal conc. in air is 21%
- can breath 50-60% O_2 enriched atmosphere for several hrs
  → dangerous to do without knowing associated risk
- not flammable but supports combustion
  → materials burn fiercly
- concentrations above 23% in air the situation becomes dangerous
- colorless, odorless + tasteless
- heavier than air → goes to low lying areas
- 3 elements needed for explosion
  ⇒ O_2, combustible material, ignition sources

Kerri C. Cooper 6/9/08

Training w/ Air-Liquide

- never use oil or grease to lubricate O_2 equipment
- leaking equipment is very dangerous
  - → perform a leak check before operation
- be careful using air within a system with O_2 cleaned parts
  - ★ → O_2 only in system ★
- no flames in area
- risk areas for atmosphere
  - → low enclosed areas
  - → around tank filling
  - → closed rooms, insufficient ventilated areas
- no work before checking atmosphere
- in case of entry in enriched atmosphere
  - → ventilate clothing in open air for at least 15 min before smoking or going near ignition source

B. Temperature effect

C. Adiabatic compression effect
- opening a valve, gas downstream is quickly compressed
- use validated compression effect: explosion of a press. gauge

D. Flow friction effect
- velocity of gas

IV. Ozone Safety (presented by Ted Rekart)

A. Advantages
- not stored in bulk
- generator shutdown eliminates supply of ozone
- not explosive or flammable

B. Important concepts
- automatic warning → can smell before harm
- effects of exposure are a function of time + conc.

Kim C-Cagye 6/9/08

6/9/08

Training w/ Air-Liquide

- first aid
  - → low level - fresh air
  - → high levels - seek medical care

C. Reactions to Exposure

- irritant to eyes, throat, nose, upper respiratory tract + lungs
- headaches, nausea, wheezing, or couching
- pulmonary edema
- no evidence will cause cancer or harm to unborn
- check w/ doctor if you have special respiratory or heart conditions
- discharge according to OSHA
  - * check requirements
  - → record discharge conc. according to analyzer

~~V HA~~ KC

Kuui C. Cooper 6/9/08

HAZOP Training with Air-Liquide

June 2, 2008

Attendees
- Kerri Cooper
Vasuhi
Ted Rekart
Steve Fisher
Lei Wang
Bharat Aluri
Todd Diel
Peter Slade
Ed Steiner

- Risk matrix used to determine severity

Nodes
1. Between O_2 cylinder and O_3 generator
   → add PRV to outside building
   → valve misalignment: ~~at~~ KC dial reset inappropriately while not being attended
   ★ SOP for ~~:~~ checking O_2 cylinder volume
   ★ SOP for removing O_2 line
   ★ look at dew pt. temp vs cooling H_2O temp
2. Ozone humidification line
   → Everything between BV7 + BV4 (except N_2 lines + MnO_2 column)
   ~~O~~ ★ mini operating procedure → complete purge w/ ambient analyzer
   – SOP for checking concentration
   – channeling through MnO_2 → agglomeration of powder ~~and~~ KC causing ~~bits~~ KC O_3 not to come in contact with the powder and destruct
   – in startup SOP check MnO_2 reaction

Node 3

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6/9/08

3. Node 3: MnO_2 destruct column
   - → check efficacy of column routinely (startup SOP)
   - → monitor KCO $\cancel{O_2}$ exit temp.
   - → column pressure:

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HAZOP Training
June 3, 2008

4. Node: Ozone Analyzer
   - add flow meter to P+ID (black box)
   - make sure SOP includes setting appropriate flow
   - 1

5. Nitrogen Supply
   - initial valve alignment
   - check press on tank (50psi)
   - verify N_2 amt during purge → flow meter + ambient analyzer
   - need check valve coming out of generator to avoid backflow ~~into~~ of N_2
   - No oil N_2 needed

6. Anaerobic Tank + KI destruction
   - glass wool at outlet to capture KI
   - SOP for checking KI strength
   - pre-req → check all tanks before beginning
   - ~~SOI~~ KC

7. Fume Hood
   - SOP in case of hood failure → turn off cylinders
   - calculate O_2 percentage with full release
     L × atm   150 × 49L

Kerri C. Cooper 6/9/08

 6/9/08

Calculations

1. Anaerobic Tank Volume

$V = \pi \left(\frac{117mm}{2}\right)^2 (270mm)$

$= 2.9\times10^6 mm^3$

$\boxed{V = 2.9L}$

2. Maximum flow rate of O_3 released from generator

$\frac{0.96m^3}{hr} \cdot \frac{1000L}{m^3} \cdot \frac{1hr}{60min} = 16$

$\boxed{\text{max flow rate} = 16 L/min}$

3. Cooling medium pressure for O_3 generator (max press)

- $6\,barg \cdot \frac{1.43\,bar}{barg} = 8.6\,bar$
- $8.6\,bar \cdot \frac{14.5psi}{1\,bar} = \cancel{125.7psi}$ KC 124.7 psi
- $\boxed{124.7psi = 124.7psig}$

4. Volume of Buffer tank

$10\,gal \cdot \frac{3.79L}{1gal} = \boxed{37.9L}$

5. Required time to flush Anaerobic tank

$t = \frac{V}{Q}$

$= \frac{2.9L}{16L/min}$

$\boxed{t = 0.18125 min = 10.9secs}$

Kari C. Cooper 6/9/08

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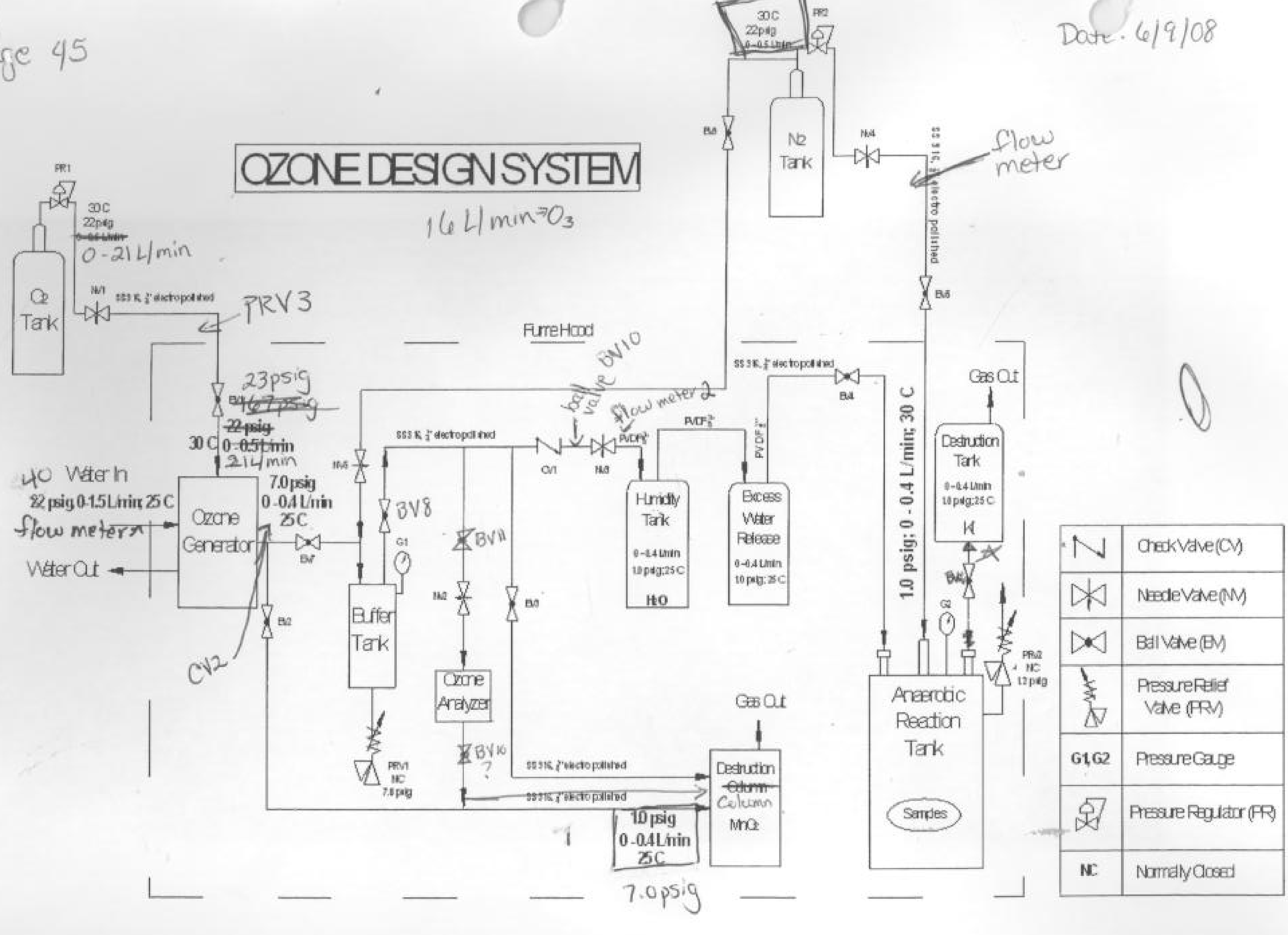
Date. 6/9/08

OZONE DESIGN SYSTEM

16 L/min → O_3



Version #

Draft #

Khui C. Coop 6/9/08

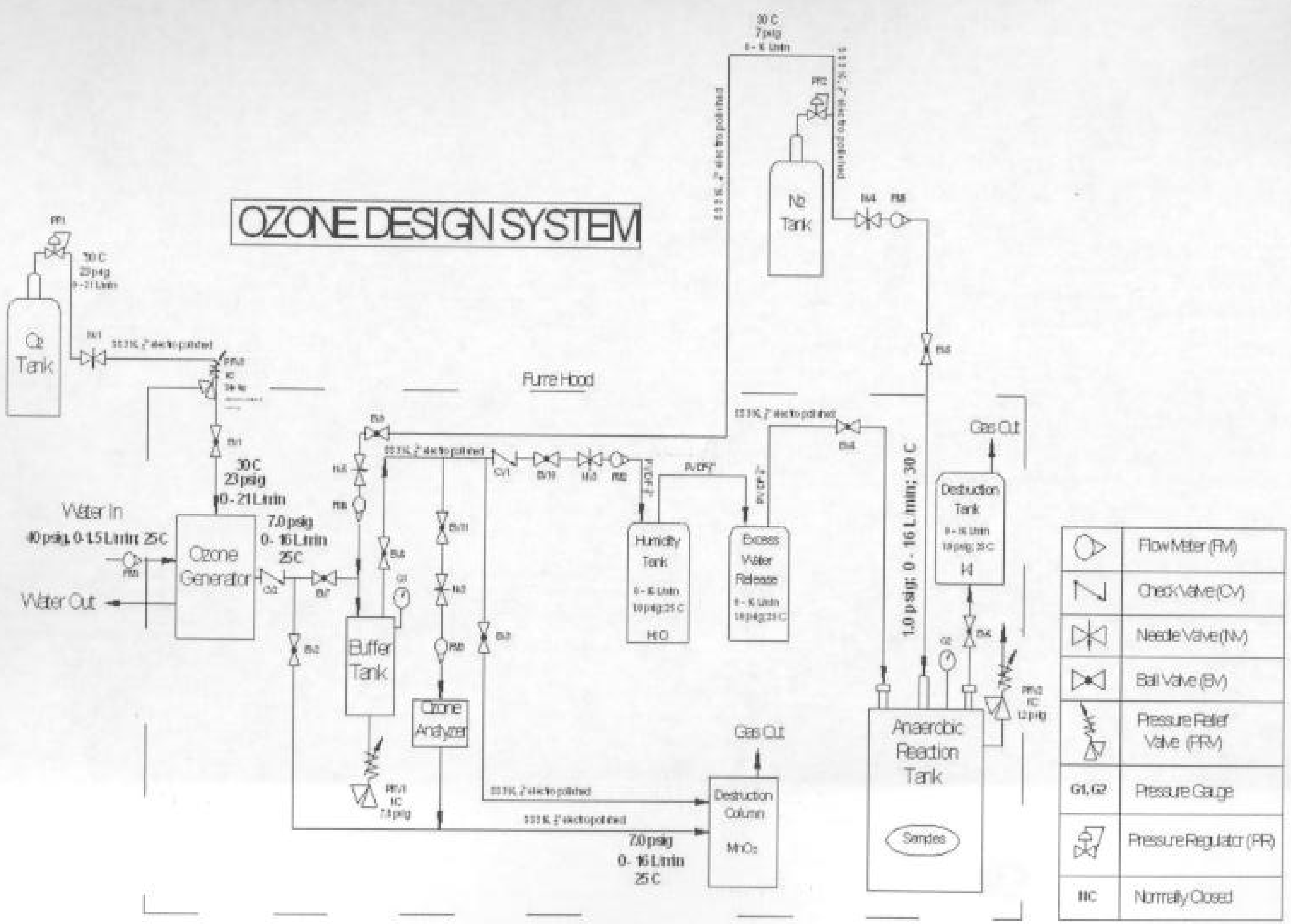
Date: 6/9/08

Changes from Draft 8

- Recalculate flows and pressures
- Addition of PRV between the O2 cylinder and the O3 generator
  - This prevents too high pressure of oxygen from flowing into the ozone generator
  - A safety measure in case the pressure regulator fails or is misaligned
- Addition of valves
  - CV2 – check valve prevents backflow of ozone into the generator which could cause over pressurizing
  - BV11 – allows for ozone to flow freely into the humidity tank without being routed to the MnO2 destruct column after the correct concentration has been established
  - BV10 – closes off the line to the humidity tank while the ozone concentration is being achieved and stabilized
  - Flow meters – reference for setting the needle valves for a particular flow
- Changed the direction of the arrow of the connection between the Anaerobic Reaction Tank and the KI Destruction Tank to flowing into the destruction tank
- Moved the line connecting the nitrogen tank to the buffer tank so that it is connected after PR2

Keri C-Coop 6/9/08

Date: 6/12/08



Draft 9
Version 1
Date: 06/9/08

Kevin C. Cooper 6/12/08

Date: 6/24/08

Changes from Draft 9, Version 1

- Added in references for the ozone generator and ozone analyzer (H1) P&IDs
- Changed PRV 2 pressure rating
    - Miscalculation of the Anaerobic Reaction Tank maximum pressure
    - 0.2bar = 2.9psi
    - Pressure set to release at 2.5 for safety aspects
- Changed sizes of text for easier reading

Kimi C. Cooper 6/24/08

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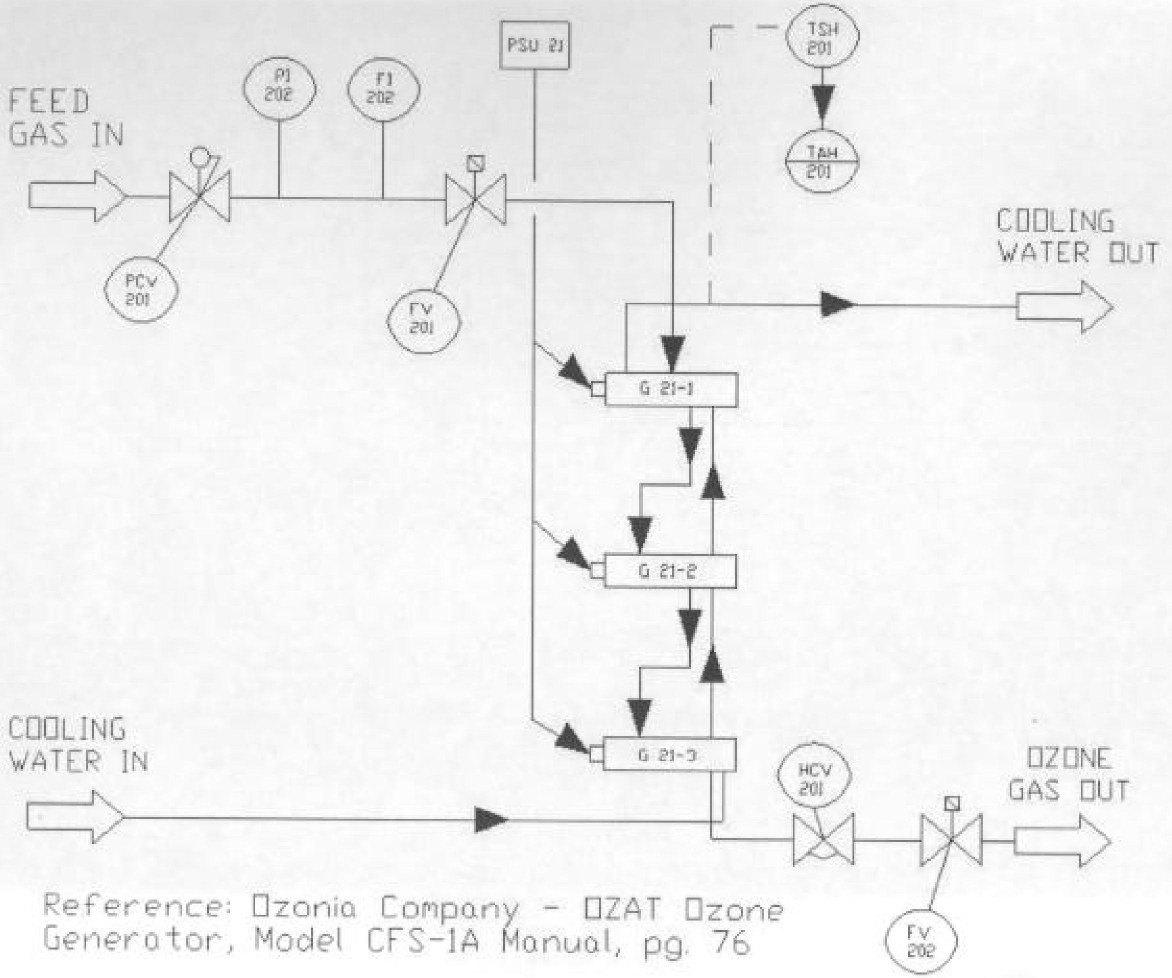
6/24/08

Ozone Generator P&ID



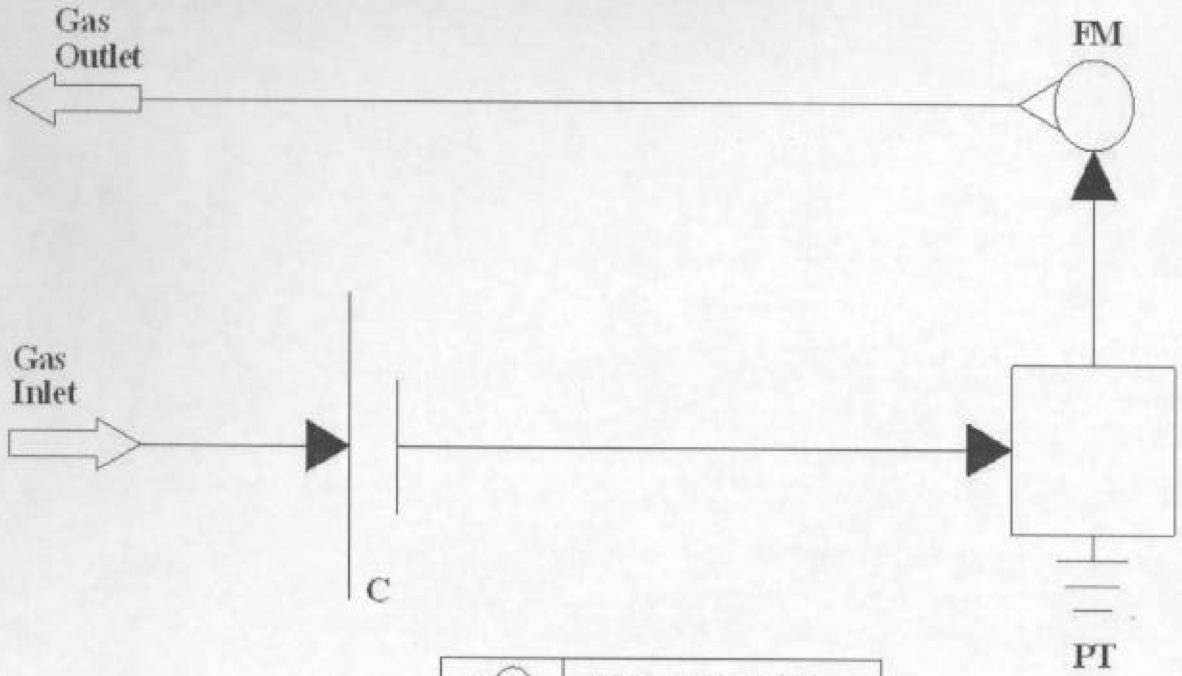
PCV201	Pressure Control Valve
FV201/ FV202	Solenoid Valve
PI202	Gas Pressure Display
FI202	Gas Flow Display
G21-1..3	Ozone Generator
TSH201-1	Temperature Monitor Switch
TAH 201-1..3	Temperature Alarm, high
HCV201	Hand Operated Control Valve
PSU21	Electrical Power Supply
	Pressure Control Valve
	Solenoid Valve
	Hand Control Valve

Reference: Ozonia Company - OZAT Ozone Generator, Model CFS-1A Manual, pg. 76

Kim C. Cooper 6/24/08

Date: 6/24/08

H1 Series Ozone Analyzer P&ID



	Flow Meter (FM)
	Pressure Transducer (PT)
	Cell (C)

Kim C. Coar 6/24/08

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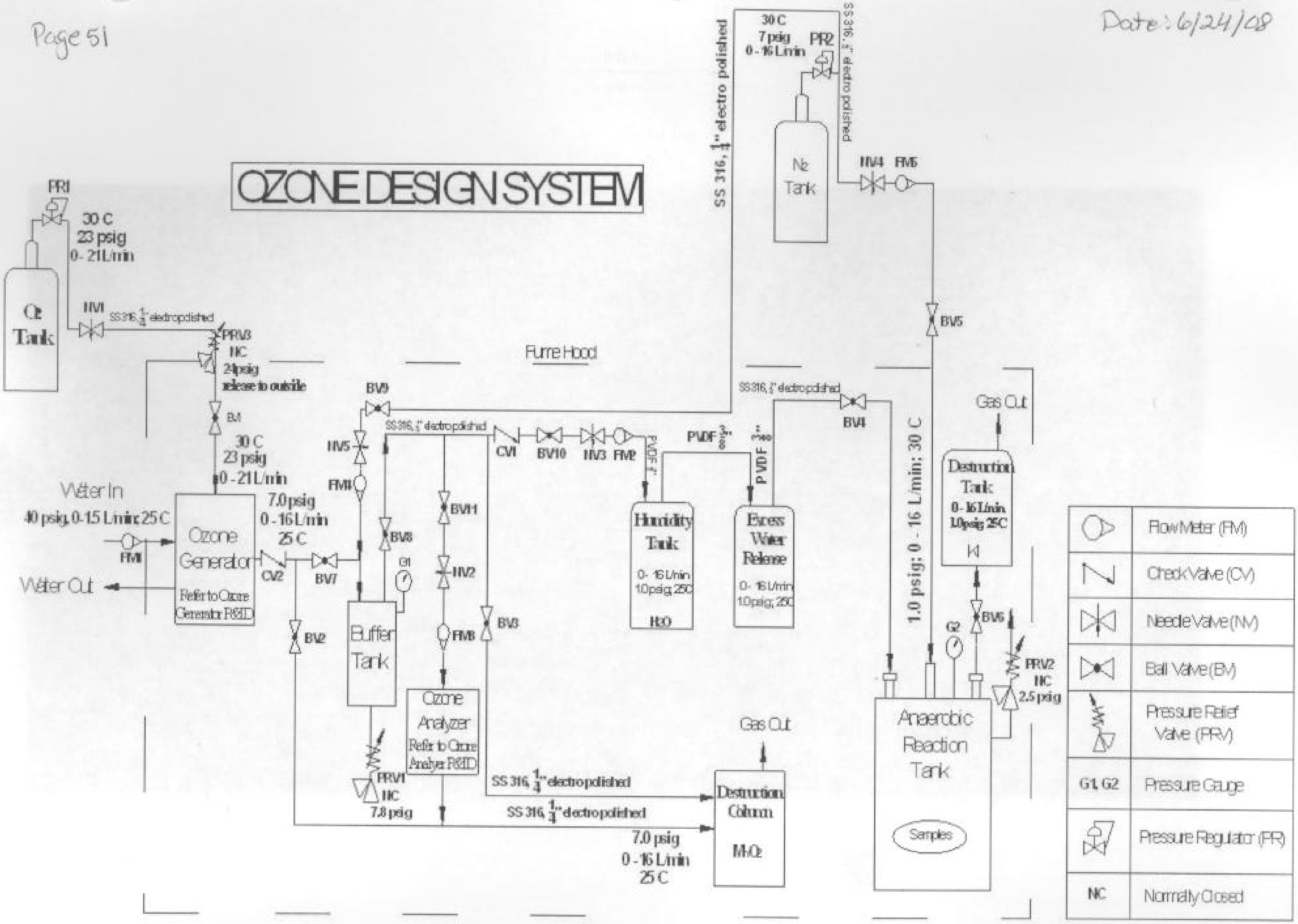
Date: 6/24/08



Draft 9
Version 2
Date: 06/24/08

Kim C. Cooper 6/24/08

Date: 7/28/08

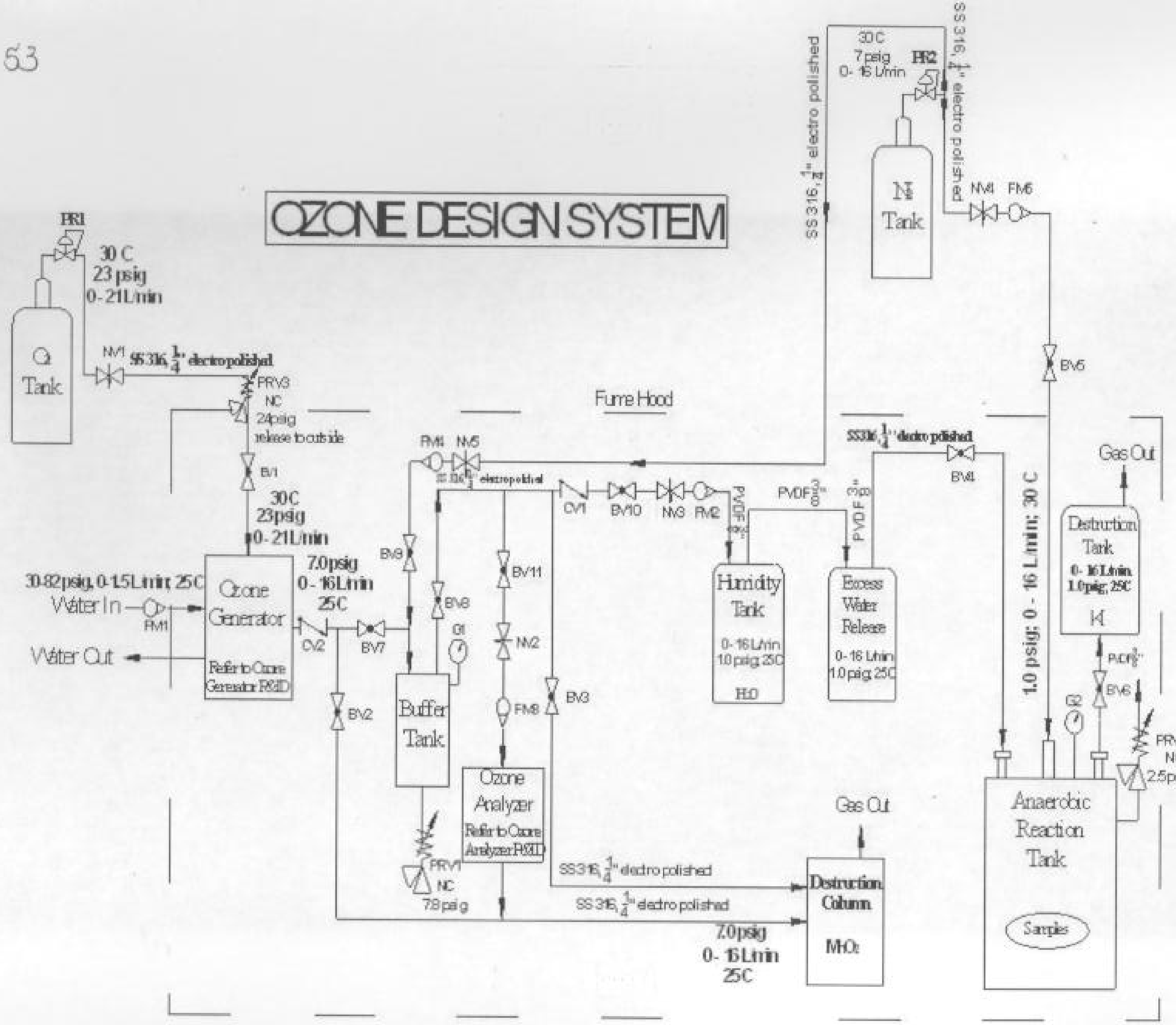
Changes from Draft 9, Version 2

- Changed pressure of cooling water entering the ozone generator
  - There is an actual range that the generator can withstand
  - 38-82psi
- Added in material change leading into the KI tank
  - Stainless steel is connected between the anaerobic reaction tank and valve BV6
  - PVDF connects BV6 to the KI tank

Keri C. Coaz 7/28/08

Date: 7/28/08

OZONE DESIGN SYSTEM



	FlowMeter (FM)
	Check Valve (CV)
	Needle Valve (NV)
	Ball Valve (BV)
	Pressure Relief Valve (PRV)
G1, G2	Pressure Gauge
	Pressure Regulator (PR)
NC	Normally Closed

Draft 9
Version 3
Date: 07/28/08

Kurt C. Cogg 7/28/08

Date: 9/25/08

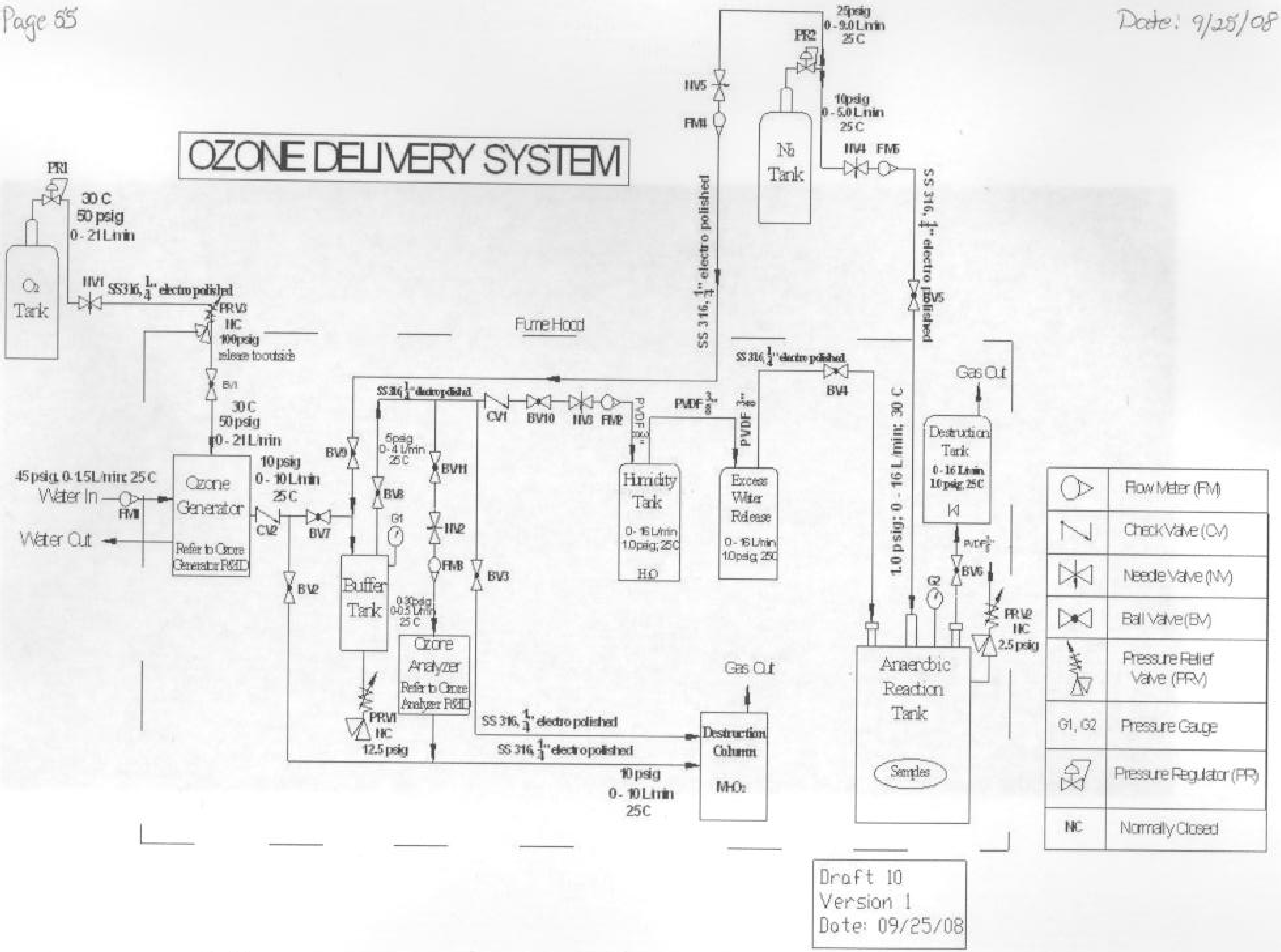
Changes from Draft 9, Version 3

- Oxygen tank exit pressure
  - Changed to 50psi due to the range rated for the ozone generator. The middle pressure was chosen for safety aspects
- Cooling water inlet pressure
  - Changed the pressure to 45psi to prevent overpressurization of the gauge and the generator
- Exit pressure from the ozone generator
  - Changed pressure to 10psi to prevent blowing PR1 which is set to 12.5
- Pressure of exit gas from the buffer tank
  - Changed to 5psi to prevent overpressurizing the gas washing bottles and causing the stoppers to pop open
- Inlet pressure of nitrogen tanks
  - 25psi for lines flowing into the buffer tank
  - 10psi for purge line into the anaerobic reaction tank

Kim C. Coa 9/25/08

Page 55

Date: 9/25/08



Kerri C. Coop 9/25/08

Page 56

Date: 12/2/08

Changes from Draft 10

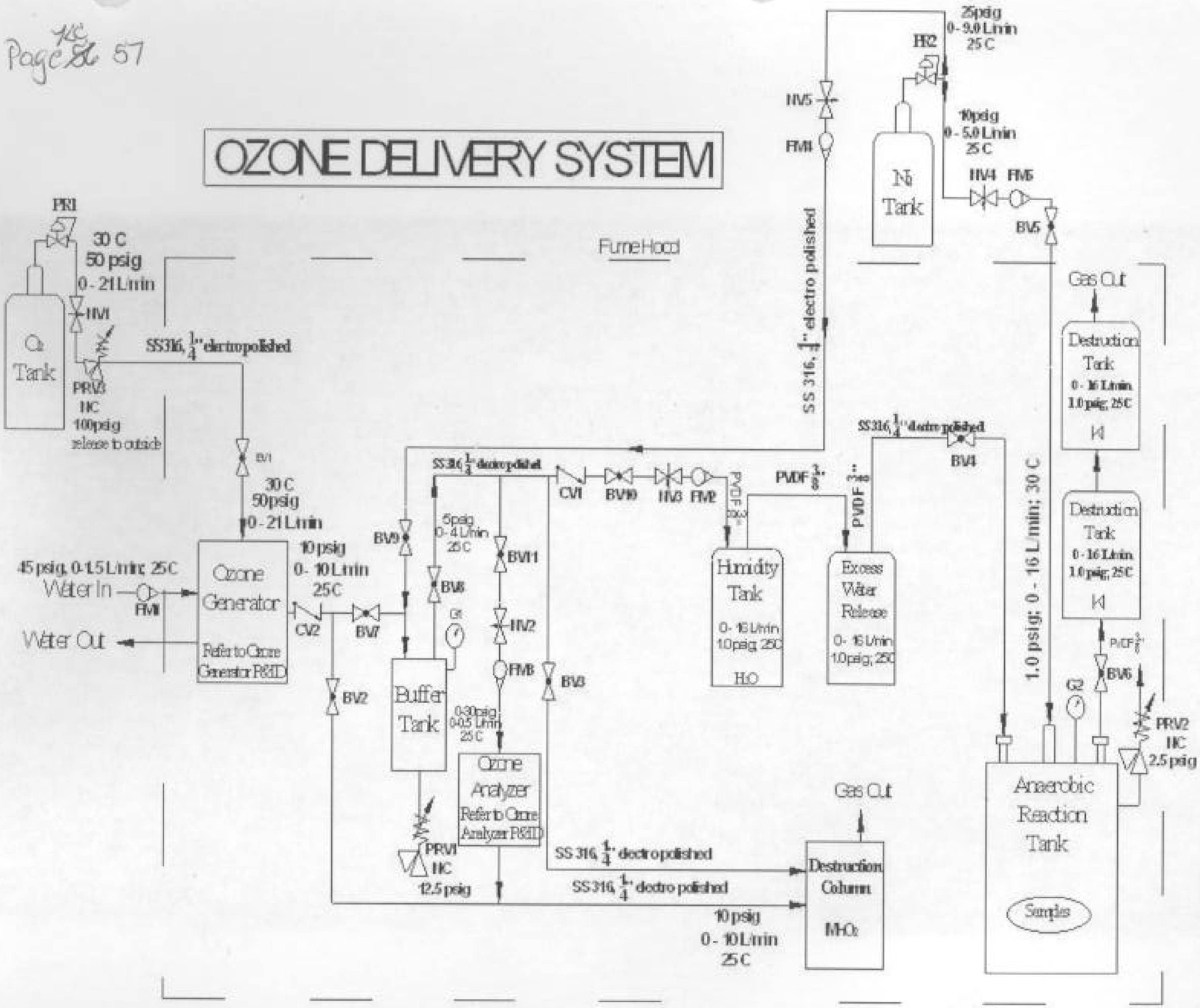
- Added in an extra KI Destruction Tank
  - This will help destroy more ozone without depleting the solution too fast

Kerri C. Cooper 12/15/08

Page ~~56~~ 57 KC

Date: 12/2/08

OZONE DELIVERY SYSTEM



(symbol)	Flow Meter (FM)
(symbol)	Check Valve (CV)
(symbol)	Needle Valve (NV)
(symbol)	Ball Valve (BV)
(symbol)	Pressure Relief Valve (PRV)
G1, G2	Pressure Gauge
(symbol)	Pressure Regulator (PR)
NC	Normally Closed

Draft 11
Version 1
Date: 12/2/08

Kumi C. Ong 12/15/08

MOFFETT CENTER	Page 1 of 11
Title: **Ozone System Startup and Shutdown Procedure**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP001-V01	Effective Date: May 2008

1.0 Purpose and Scope

1.1 To safely startup and shutdown the ozone system for delivery and destruction to the anaerobic reaction tank

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Ozone is toxic to humans and should be contained and destroyed effectively

3.2 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned

3.3 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere

4.0 Equipment and Supplies

- Oxygen tank
- Nitrogen tank
- Ozone Generator (Ozonia/CFS-1A)
- Buffer tank (10gal pressure vessel)
- Ozone Analyzer (IN USA/H1)
- Ambient Ozone Analyzer (IN USA/IN2000L2-LC)
- Anaerobic Reaction Tank (Schütt Labortechnik/stainless steel)
- MnO_2 Destruction tank
- Humidity tank, excess water tank, and destruction tank (Ace Glass/500ml Gas washing bottle)
- Stainless Steel piping (Valex 316L, Specification 301, ¼ in)
- Ball Valves (Swagelok/SS-42GS4-SC11)
- Needle Valves (Swagelok/SS-4MG-SL-SC11)
- Pressure Release Valve (Swagelok/SS-RL3S4/SC11)
- Pressure Gauge (Blue Ribbon Sales & Service Corp/BR4001-4LD, oxygen cleaned)
- PVDF Tubing (McMaster-Carr/5390K342, 3-A sanitary tubing)
- Butterfly Valve (McMaster-Carr/4682K74, ½ in connections)
- Check Valve (Swagelok/SS-4C-1-SC11)
- Flowmeter (Mcmaster-Carr/8051K17)
- Nylon Tubing (McMaster-Carr/5112K653, 3/8in OD)
- Oxygen Detector (McMaster-Carr/18995T14)
- Vaneometer

5.0 Operational Parameters

- Humidity tank volume: 250ml
- KI Destruction Tank volume: 300mL KI solution (40g/L)
    - 12g KI / 250ml H_2O

6.0 Operational Procedure

6.1 Turn on the following:

6.1.1 Fume Hood

a. Refer to vaneometer to ensure that air is flowing upward into the fume hood

6.1.2 Ambient ozone analyzer

6.1.3 In-line ozone analyzer

6.2 Ensure needle valves are set correctly according to SOP: MC-SA-SOP005-V01

6.3 Calculating the Volume of Gas in the O_2 and N_2 Cylinders

6.3.1 Determine pressure within the cylinder by referring to the gauge connected to the cylinder

6.3.2 Calculate the volume of the cylinder using this ratio by solving for x:

$$\frac{2600\,psi}{300\,ft^3} = \frac{\text{gauge pressure (psi)}}{x\ \text{ft}^3}$$

6.3.3 Record the gas cylinder volumes in the log book located in Lab 105

6.3.4 If volume is not sufficient for experimental run then refer to the disconnecting O_2 and N_2 cylinders SOP (MC-SA-105-SOP003-V01)

***This should be done before every experimental run**

6.4 Flushing the Anaerobic Reaction Tank

6.4.1 Ensure the following valves are closed

a. PR 1-2 and BV 1-11

6.4.2 Open BV6

6.4.3 Open BV5

a. Switch the orange lever to the open position

6.4.4 Set PR2 to 10 psi

a. Flow rate: 5 L/min

6.4.5 Allow nitrogen to flow through the Anaerobic Reaction Tank into the KI Destruction Tank

6.4.6 Verify flush time with ambient ozone analyzer by placing tubing perpendicular to the exit port of the KI Destruction Tank

a. Ozone concentration should be zero

6.4.7 Stop nitrogen flow by setting PR2 to zero

6.4.8 Close valve BV5
- a. Switch the orange lever to the shut position

6.4.9 Close valve BV6

6.5 <u>Setting Parameters for the Ozone Generator</u>

6.5.1 Turn water supply on
- a. <u>Constraints:</u>
  - Flow: 1.5 L/min
  - Pressure: not to exceed 50psi
  - Butterfly valve open to 40°

6.5.2 Switch on (MAIN-ON) ozone generator and wait until the screen turns green
- a. Screen should read: MAINS ON→ SYSTEM CHECK

6.5.3 Select desired language (English, German, Latin, French, Italian, Spanish)
- a. Continually press SET POINT until desired language is displayed

6.5.4 Wait 5 seconds to allow the ozone generator to set the language

6.5.5 Ensure SET POINT is set to LOCAL
- a. Screen should read 0% LOCAL, PSU OFF

6.5.6 Open oxygen cylinder

6.5.7 Set PR1 to 50psi by turning the regulator knob

6.5.8 Open valves (located under table)
- a. BV2
- b. BV1

6.5.9 Set pressure on the generator to 1.5bar using the PCV 201 dial
- a. Refer to PI 201 pressure gauge

6.5.10 Turn power supply on by switching PSU ON (located on ozone generator)
- a. Screen should read PSU ON

6.5.11 Set gas flow by adjusting the HCV 201 dial located on the front of the generator

6.5.12 Adjust pressure and gas flow to desired parameters

6.5.13 Increase %PSU by pressing the SET POINT button.

6.6 <u>Checking MnO_2 Effectiveness</u>

6.6.1 Ensure BV2 is open

6.6.2 Follow ozone generator startup procedure

6.6.3 Place tubing connected to the ambient ozone analyzer (Model IN2000L2-LC) perpendicular to flow to the exit port of the MnO_2 destruction column
- a. The analyzer should read 0.05ppm or less

6.6.4 Record concentration in the log book

6.6.5 Using temperature probe determine temperature of the gas at the exit port
- a. **Do not touch column – it may be hot**

6.6.6 Record information in the log book

6.6.7 If concentration exceeds 0.05ppm immediately switch ozone generator MAINS OFF. If the concentration is below 0.05ppm continue with the following steps

a. DO NOT OPEN COLUMN → MnO_2 requires special handling

6.6.7..1 Notify Ed Steiner (NCFST Director of Facilities and Pilot Plant) and Vaushi Rasanayagam (Air-Liquide)

6.6.8 Open BV7 and allow ozone to flow into the Buffer Tank
6.6.9 Close BV2
6.6.10 Pressurize tank to 5 psi
6.6.11 Open BV2
6.6.12 Close BV7

6.7 <u>Checking KI Destruct Tank Effectiveness</u>

6.7.1 Open BV6
6.7.2 Open BV4
6.7.3 Open BV10
6.7.4 Open BV8
6.7.5 Allow ozone to flow through the KI Destruction Tank
6.7.6 Place tubing connected to the ambient ozone analyzer (Model IN2000L2-LC) at the exit port of the KI destruction tank

a. The analyzer should read 0.05ppm

6.7.7 Close BV8
6.7.8 Close BV10
6.7.9 Close BV4
6.7.10 Close BV6
6.7.11 Record effluent ozone concentration in log book
6.7.12 If ozone limits exceed 0.05ppm follow the next steps otherwise skip to step 6.7.18
6.7.13 Remove KI Destruction Tank from the system
6.7.14 Dispose of the KI according to safety procedures
6.7.15 Refill with new KI solution
6.7.16 Reattach KI Destruction Tank to system
6.7.17 Follow the Flushing the Anaerobic Reaction Tank procedure

6.8 <u>Achieving Desired Concentration</u>

6.8.1 Open BV3
6.8.2 Open BV11
6.8.3 Open BV8
6.8.4 Open BV7
6.8.5 Close BV2

6.8.6 Allow ozone to flow through the H1 analyzer at 0.5L/min until desired concentration is achieved
 a. Adjust %PSU and flow rates as needed
 b. Monitor the flow rate on the analyzer to ensure it is exactly 0.5L/min. You may have to adjust NV2 and BV3
 c. Monitor G1 on the buffer tank to make sure it remains zero

6.8.7 Close valve BV8

6.8.8 Close valve BV11

6.8.9 Close valve BV3

6.8.10 Pressurize buffer tank to 5psi
 a. Refer to G1

6.8.11 Open valve BV2

6.8.12 Close valve BV7

6.9 <u>Stopping Gas Flow</u>

6.9.1 Reduce power supply to 0% LOCAL by pressing the down button for SET POINT

6.9.2 Turn off ozone generator power supply by switching to PSU OFF
 a. Screen should indicate: PSU OFF and PURGING time
 b. The screen background should be orange

6.9.3 Allow for the equipment to purge the system with the feedgas and remove the residual ozone in the generator
 a. This process runs for 90s

6.9.4 Once this is completed the screen will turn green and read: PSU OFF and POWER 00000kw

6.9.5 Set ozone generator pressure to zero
 a. Adjust by turning PCV 201 dial

6.9.6 Stop oxygen flow into the ozone generator
 a. Set PR1 to zero
 b. Close valve BV1

6.9.7 Close valve BV2

6.10 <u>Experimental Run</u>

6.10.1 Once desired concentration is achieved open valve BV6

6.10.2 Open valve BV4

6.10.3 Open valve BV10

6.10.4 Open valve BV8

6.10.5 Allow ozone to flow through the anaerobic tank

6.10.6 Close valve BV6

6.10.7 Pressurize anaerobic tank to 1.4psi (0.1bar)
 a. Refer to G2

6.10.8 Close valve BV8
6.10.9 Close valve BV10
6.10.10 Close valve BV4
6.10.11 Perform experimental run
6.10.12 Refer to Step 6.4 for flushing the Anaerobic Reaction Tank
6.10.13 Remove samples replace lid securely

6.11 Flushing the System

6.11.1 Open valves
 a. BV3
 b. BV8
6.11.2 Depressurize buffer tank by allowing the ozone to flow out of the tank
 a. G1 should read 0psi before continuing
6.11.3 Set PR2 to 25psi
6.11.4 Open valves
 a. BV11
 b.BV9
6.11.5 Allow nitrogen to flush through the system
6.11.6 Monitor the H1 in-line analyzer until the %wt reads 0%
6.11.7 Close valves
 a. BV8
 b. BV11
 c. BV3
6.11.8 Pressurize buffer tank to 10psi
6.11.9 Close BV9
6.11.10 Open valves (in the following order)
 a. BV6
 b. BV4
 c.BV10
 d.BV8
6.11.11 Allow nitrogen to flush through system
 a. Monitor ozone concentration released from the KI column using the ambient ozone analyzer ensuring that it does not exceed 0.05ppm
 b. If the concentration does not exceeds 0.05ppm continue with Step 6.11.17
6.11.12 If concentration exceeds 0.05ppm immediately close the following valves:
 a. BV8
 b. BV10
 c.BV4
 d.BV6
6.11.13 Remove both KI destruct columns from the system
6.11.14 Transfer the KI waste into the designated waste container

6.11.15 Fill the bottles with new KI solution and then continue with Step 6.11.18
6.11.16 Open BV3
6.11.17 Close valves (in the following order)
a.BV10
b.BV4
6.11.18 Allow remainder of nitrogen to flow out of tank
a. Make sure G1 reads 0psi
6.11.19 Close
a. BV8
b. BV3
6.11.20 Set PR2 to 10psi
a. Flow rate: 5 L/min
6.11.21 Open valve BV5 to remove humidity in the anaerobic reaction tank
a. Switch orange lever to open
6.11.22 Allow nitrogen to flush through the tank for at least 1.5min
6.11.23 Set PR2 to zero
6.11.24 Close valve BV5
a. Switch orange lever to shut position
6.11.25 Allow Anaerobic Reaction Tank pressure to reach 0psi
a. Refer to G2
6.11.26 Close valve BV6

6.12 If performing another experimental run return to Step 6.5.5. If the experimentation is done for the day, continue with step 6.13

6.13 Shut Down Procedure (once all experimental runs are completed)
6.13.1 Switch MAINS OFF
6.13.2 Turn off water supply
a. Close butterfly valve
6.13.3 Close gas cylinders by turning the valve
a. Nitrogen
b. Oxygen
6.13.4 Turn off analyzers
a. H1 (in-line)
a. Ambient analyzer
6.13.5 Turn off fume hood

7.0 References and Supporting Documents

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Operating Instructions. OZAT® Ozone Generator. Type CFS-1…3A.

Ozone Material Safety Data Sheet (MSDS).
<http://www.ozoneapplications.com/info/ozone_msds.htm>.

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MOFFETT CENTER	Page 1 of 7
Title: **Emergency Shutdown Procedures**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP002-V01	Effective Date: June 2008

1.0 Purpose and Scope

1.1 To safely shutdown the ozone system in a timely fashion if procedure limits or equipment fails.

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Ozone is toxic to humans and should be contained and destroyed effectively
3.2 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned
3.3 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere
3.4 Release of nitrogen into the atmosphere can reduce the level of oxygen causing potential suffocation.

4.0 Equipment and Supplies

- Oxygen tank
- Nitrogen tank
- Ozone Generator (Ozonia/CFS-1A)
- Buffer tank (10gal pressure vessel)
- Ozone Analyzer (IN USA/H1)
- Ambient Ozone Analyzer (IN USA/IN2000L2-LC)
- Anaerobic Reaction Tank (Schütt Labortechnik/stainless steel)
- MnO_2 Destruction tank
- Humidity tank, excess water tank, and destruction tank (Ace Glass/500ml Gas washing bottle)
- Stainless Steel piping (Valex 316L, Specification 301, ¼ in)
- Ball Valves (Swagelok/SS-42GS4-SC11)
- Needle Valves (Swagelok/SS-4MG-SL-SC11)
- Pressure Release Valve (Swagelok/SS-RL3S4/SC11)
- Pressure Gauge (Blue Ribbon Sales & Service Corp/BR4001-4LD, oxygen cleaned)
- PVDF Tubing (McMaster-Carr/5390K342, 3-A sanitary tubing)
- Butterfly Valve (McMaster-Carr/4682K74, ½ in connections)
- Check Valve (Swagelok/SS-4C-1-SC11)
- Rotameter (Mcmaster-Carr/8051K17)
- Nylon Tubing (McMaster-Carr/5112K653, 3/8in OD)
- Oxygen Detector (McMaster-Carr/18995T14)
- Cotton/Polyester Blend Knit Gloves (McMaster-Carr/6078T53)
- Vaneometer

5.0 Responsible Personnel

- Kerri Cooper: Ozone System Engineer (Student) 803-669-3737
- Ed Steiner: Director of Facilities and Pilot Plant 708-308-8911
- Dr. Peter Slade: Project Instructor 708-563-8172

- Claudia Rodriguez: Co-Project Instructor 708-563-2052

6.0 Operational Procedure

6.1 Fume Hood Failure (due to mechanical or power failure)

6.1.1 Refer to vaneometer to determine if air is flowing in the direction of fume hood draw and use sound for indication of failure

6.1.2 Check efficacy of the KI destruction tank and the MnO_2 destruct column

a. If **ozone's** limits are less than **0.1ppm** and **oxygen** detector is within the range of **20-21%** continue experiment but monitor levels

b. If limits are not within range continue to step 6.1.3

6.1.3 Do emergency shutdown of the ozone generator by switching MAINS to OFF

a. This will skip the generator purge step (do not allow system to purge)

6.1.4 Close valves BV2, BV3, and BV6

6.1.5 Close N_2 and O_2 cylinders if they are open

6.1.6 Set PR1-2 to zero

6.1.7 Evacuate room and close door

6.1.8 Post note on door warning individuals not to enter room

6.1.9 Contact responsible personnel

6.2 Oxygen Leak or High Concentrations of 23% or greater (refer to oxygen detector)

6.2.1 Ensure fume hood is running

a. Refer to vaneometer

6.2.2 Perform emergency shutdown of ozone generator by switching MAINS to OFF

6.2.3 Set PR1 to zero

6.2.4 Close O_2 and N_2 cylinders

6.2.5 Close valves BV2, BV3, BV10 and BV6

6.2.6 Evacuate room

6.2.7 Post note on door warning individuals not to enter room

6.2.8 Contact responsible personnel

6.3 Ozone Leak or Release of High Concentrations

6.3.1 Ensure fume hood is running

a. Refer to vaneometer

6.3.2 Refer to O_3 analyzer Model: IN2000L2-LC

a. If concentration is less than 0.1ppm perform leak test and check KI destruction tank and MnO_2 exhaust

a. If concentration is 0.1ppm or greater continue with emergency shutdown procedure

6.3.3 Perform emergency shutdown of ozone generator by switching MAINS to OFF

6.3.4 Close valves BV2, BV3,BV10 and BV6

6.3.5 Close O_2 and N_2 cylinders

6.3.6 Evacuate room and close door

6.3.7 Post note on door warning individuals not to enter room
6.3.8 Contact responsible personnel

6.4 Nitrogen Release into Atmosphere (refer to O_2 analyzer)
6.4.1 Ensure fume hood is running
6.4.2 Refer to oxygen analyzer to determine percentage of O_2 in atmosphere
a. Oxygen level should not fall below 19.5%
6.4.3 Perform emergency shutdown of ozone generator by switching MAINS to OFF
6.4.4 Close O_2 and N_2 cylinder
6.4.5 Close valves BV2, BV3, BV10 and BV6
6.4.6 Evacuate room and close door
6.4.7 Post note on door warning individuals not to enter room
6.4.8 Contact responsible personnel

6.5. Determining percent oxygen in the atmosphere after a nitrogen release

$$\%O_2 = \frac{V_O}{V_r} * 100 \quad \text{where,}$$

V_r = room volume
V_g = max gas release (cylinder volume)
$V_o = 0.2095*(V_r - V_g)$

6.5.1 Worst Case Scenario for the Release of the Maximum Volume of the N_2 Cylinder into Laboratory 105 – NCFST

Room volume = 2457ft^3 Volume of N_2 cylinder = 300ft^3

$$\%O_2 = \frac{V_O}{V_r} * 100$$

$$\%O_2 = \frac{0.2095 * (2457 ft^3 - 300 ft^3)}{2457 ft^3}$$

$$\%O_2 = 18.4\%$$

7.0 References and Supporting Documents

Oxygen Material Safety Data Sheet (MSDS).

Ozone Material Safety Data Sheet (MSDS). <http://www.ozoneapplications.com/info/ozone_msds.htm>.

Ozone Safe Working Practices. Work SafeBC. <http://www.worksafebc.com/publications/health_and_safety/by_topic/assets/pdf/ozone_bk47.pdf>.

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MOFFETT CENTER	Page 1 of 5
Title: **Disconnecting the Oxygen and Nitrogen Cylinders**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP003-V01	Effective Date: June 2008

1.0 Purpose and Scope

1.1 To safely disconnect the oxygen and nitrogen cylinders from the system

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Incorrect removal could cause release of ozone, oxygen, or nitrogen into the atmosphere
3.2 Ozone is toxic to humans and should be contained and destroyed effectively
3.3 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned
3.4 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere
3.5 Inappropriate disconnection of the cylinders can cause over pressurization of the system causing gas leaks or system failure

4.0 Equipment and Supplies

- Oxygen tank
- Nitrogen tank
- Ozone Generator (Ozonia/CFS-1A)
- Ambient Ozone Analyzer (IN USA/IN2000L2-LC)
- Stainless Steel piping (Valex 316L, Specification 301, ¼ in)
- Ball Valves (Swagelok/SS-42GS4-SC11)
- Needle Valves (Swagelok/SS-4MG-SL-SC11)
- Pressure Release Valve (Swagelok/SS-RL3S4/SC11)
- Butterfly Valve (McMaster-Carr/4682K74, ½ in connections)
- Rotameter (Mcmaster-Carr/8051K17)
- Nylon Tubing (McMaster-Carr/5112K653, 3/8in OD)
- Oxygen Detector (McMaster-Carr/18995T14)
- Cotton/Polyester Blend Knit Gloves (McMaster-Carr/6078T53)
- Vaneometer
-

5.0 Operational Procedure

5.1 Turn on Fume Hood

5.1.1 Refer to vaneometer to ensure that air is flowing upward into the fume hood

5.2 Oxygen Cylinder Disconnection

- 5.2.1 Ensure the ozone generator is not running
- 5.2.2 Check valves to ensure they are closed
  - a. BV1 – 11
- 5.2.3 Close O_2 cylinder valve
- 5.2.4 Ensure PR1 is set to zero
- 5.2.5 Disconnect O_2 cylinder from system
  - a. Monitor O_2 detector to ensure high concentrations of oxygen is not being released
- 5.2.6 Reconnect new cylinder to system
- 5.2.7 Check connections for any leaks using the leak detector

5.3 Nitrogen Cylinder Disconnection

- 5.3.1 Ensure all valves are closed
  - a. BV1 – 11
- 5.3.2 Close N_2 cylinder valve
- 5.3.3 Ensure PR2 is set to zero
- 5.3.4 Disconnect the N_2 cylinder from the system
- 5.3.5 Reconnect new cylinder to system
- 5.3.6 Check connections for any leaks using the leak detector

***Note: this procedure should not be done while any pieces of the equipment are running or in use. Cylinders should be disconnected before or after experimental runs.**

6.0 References and Supporting Documents

Ozone Material Safety Data Sheet (MSDS). <http://www.ozoneapplications.com/info/ozone_msds.htm>.

Ozone Safe Working Practices. Work SafeBC. <http://www.worksafebc.com/publications/health_and_safety/by_topic/assets/pdf/ozone_bk47.pdf>.

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MOFFETT CENTER	Page 1 of 6
Title: **Ozone System General Safety Procedures**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP004-V01	Effective Date: June 2008

1.0 Purpose and Scope

1.1 To safely operate the ozone design system in accordance with the safety regulations.

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Ozone is toxic to humans and should be contained and destroyed effectively

3.2 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned

3.3 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere

4.0 Required Materials for Safely Operating the System

- Nitrile Gloves
- Laboratory Coats
- Fume Hood with vinyl curtain
- Safety Glasses
- Oxygen cleaned parts and components
- Vaneometer

5.0 Operational Procedure

5.1 Turn on Fume Hood

5.1.1 Refer to vaneometer to ensure that air is flowing upward into the fume hood

5.1.2 Noise from the fume hood also indicates it is in operation

***Fume Hood should be running at all times while operating the system**

5.2 Operational Safety and Health Administration (OSHA): Ozone exposure limits

Conditions	Ozone Concentrations
Detectable odor	0.01 to 0.05 ppm
OSHA 8hr limit	0.1 ppm
Short term exposure	0.3 ppm (15min)
Severe coughing, shortness of breath, or pain on deep inspiration	≥ 0.4 ppm (2hrs)
Immediately Dangerous to Life (IDLH)	5.0 ppm
Lethal Concentration	50 ppm (30min)

5.3 <u>System Leak Test (performed with nitrogen only)</u>

5.3.1 Set PR2 TO 25 psig
  a. Flow: 10 L/min
5.3.2 Open BV9
5.3.3 Pressurize buffer tank to 10.5psi
5.3.4 Close BV9
  a. Allow for pressure to equilibrate
5.3.5 Open valves
  a. BV2
  b. BV3
  c. BV11
  d. BV8
5.3.6 Allow nitrogen to flush through the open lines
5.3.7 Using the leak detector solution apply to connections
  a. If solution bubbles the connections need to be tightened
5.3.8 Close BV8
5.3.9 Close valves
  a. BV11
  b. BV3
  c.BV2
5.3.10 Open BV9
5.3.11 Pressurize buffer tank to 10.0 psi
5.3.12 Close BV9
5.3.13 Open valves
  a. BV6
  b. BV4
  c. BV10
  d. BV8
5.3.14 Allow nitrogen to flush through the open lines
5.3.15 Using the leak detector solution apply to connections
  a. If solution bubbles the connections need to be tightened
5.3.16 Close valves
  a. BV8
  a. BV10
  a. BV4
5.3.17 Open BV5
  a. Ensure BV6 is open before performing this action
5.3.18 Allow nitrogen to flush through the open lines
5.3.19 Using the leak detector solution apply to connections
  a. If solution bubbles the connections need to be tightened

5.3.20 Close BV5
5.3.21 Close BV6

6.0 References and Supporting Documents

Nitrogen Material Safety Data Sheet (MSDS)

Occupational Safety & Health Administration (OSHA). Ozone. May 2007. <http://www.osha.gov/dts/chemicalsampling/data/CH_259300.html#exposure>.

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MOFFETT CENTER	Page 1 of 7
Title: **Setting Needle Valves for the Ozone System**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP005-V01	Effective Date: June 2008

1.0 Purpose and Scope

1.1 To safely startup and shutdown the ozone system for delivery and destruction to the anaerobic reaction tank

2.0 Responsibility

2.1 The scientific personnel that carries out this procedure

3.0 Hazards and Safety Considerations

3.1 Ozone is toxic to humans and should be contained and destroyed effectively

3.2 Oxygen can be combustible if in contact with organic materials, all parts are oxygen cleaned

3.3 Oxygen analyzer should be used to ensure less than 23% is being released into the atmosphere

3.4 Inappropriate valve alignment can cause over pressurization which could result in an ozone, oxygen, or nitrogen leak into the atmosphere

4.0 Equipment and Supplies

- Oxygen tank
- Nitrogen tank
- Ozone Generator (Ozonia/CFS-1A)
- Buffer tank (10gal pressure vessel)
- Ozone Analyzer (IN USA/H1)
- Ambient Ozone Analyzer (IN USA/IN2000L2-LC)
- Anaerobic Reaction Tank (Schütt Labortechnik/stainless steel)
- MnO_2 Destruction tank
- Humidity tank, excess water tank, and destruction tank (Ace Glass/500ml Gas washing bottle)
- Stainless Steel piping (Valex 316L, Specification 301, ¼ in)
- Ball Valves (Swagelok/SS-42GS4-SC11)
- Needle Valves (Swagelok/SS-4MG-SL-SC11)
- Pressure Release Valve (Swagelok/SS-RL3S4/SC11)
- Pressure Gauge (Blue Ribbon Sales & Service Corp/BR4001-4LD, oxygen cleaned)
- PVDF Tubing (McMaster-Carr/5390K342, 3-A sanitary tubing)
- Butterfly Valve (McMaster-Carr/4682K74, ½ in connections)
- Check Valve (Swagelok/SS-4C-1-SC11)
- Rotameter (Mcmaster-Carr/8051K17)
- Nylon Tubing (McMaster-Carr/5112K653, 3/8in OD)
- Oxygen Detector (McMaster-Carr/18995T14)
- Vaneometer

5.0 Operational Procedure

5.1 Turn on Fume Hood

5.1.1 Refer to vaneometer to ensure that air is flowing upward into the fume hood

5.2 Refer to SOP: MC-SA-105-SOP001-V01 for starting up the ozone generator

5.3 Setting Valve Flow (these valves should not have to be adjusted once the design is setup unless flow begins to vary)

5.3.1 For NV1

a. Open valve BV1
b. Open valve BV2
c. Set NV1 by referring to the flow meter on the ozone generator. Flow should be 21 L/min
d. Close valve BV1
e. Close BV2

5.3.2 For NV2 and NV3

a. Open BV7
b. Open BV1
c. Pressurize buffer tank to 5 psi
d. Close BV7
e. Open BV2
f. Open valve BV3
g. Open valve BV11
h. Open valve BV8
i. Adjust NV2 to 0.5 L/min by referring to the ozone analyzer's flow meter
j. Close BV8
k. Close BV11
l. Close BV3
m. Open BV7
n. Pressurize buffer tank to 5psi
o. Close BV7
p. Open BV6
q. Open BV4
r. Open BV10
s. Adjust NV3 by referring to the flow meter located upstream of the valve

5.3.2..1 Set flow to 15 (3.51 L/min)

5.3.2..2 The water contained in the humidity tank should not splash excessively. If this occurs reduce the flow.

Table 1: NV3 Flow Meter Calibrations for Air

Scale Reading	Flow (L/min)
65	13.15
60	12.49
55	11.82
50	10.98
45	10.06
40	9.14
35	8.13
30	7.09
25	5.92
20	4.74
15	3.51
10	2.20
5	.96

t. Open BV2
u. Close BV8
v. Close BV10
w. Close BV4
x. Close BV6
y. Turn off ozone generator by referring to SOP: MC-SA-105-SOP001-V01 for stopping gas flow

5.3.3 Adjusting NV4 and NV5

a. Open BV6
b. Open BV5
c. Set PR2 to 10 psi
d. Adjust NV4 to 5 L/min by referring to the FM5
e. Close BV5
f. Close BV6
g. Set PR2 to 25 psi
h. Open BV9
i. Adjust NV5 to 9.5 L/min by referring to the FM4
j. Set PR2 to zero
k. Close BV9

MOFFETT CENTER	Page 4 of 7
Title: **Setting Needle Valves for the Ozone System**	Author: Kerri Cooper
Document No.: MC-SA-105-SOP005-V01	Effective Date: June 2008

6.0 References and Supporting Documents

Manganese Dioxide (MnO_2) Material Safety Data Sheet (MSDS).

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Ozone Safe Working Practices. Work SafeBC. <http://www.worksafebc.com/publications/health_and_safety/by_topic/assets/pdf/ozone_bk47.pdf>.

Ozone Design System P&ID

Potassium Iodide (KI) Material Safety Data Sheet (MSDS).

Steiner, Ed. National Center for Food Safety and Technology: Director of Facilities and Pilot Plant.

The Hazards of ozone & Ozone Gas. <http://www.inspect-ny.com/sickhouse/OzoneHazards.htm>.

Wikepedia. Ozone. 2007. <http://en.wikipedia.org/wiki/Ozone>.

Kerri C. Cooper 4/3/09

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Description of the method

The purpose of the HAZOP (hazard operability study) procedure is to analyse the operation integrity of a system. The HAZOP method is a technique for identifying potential problems and for suggesting risk mitigation measures by systematically identifying and determining the effects of the deviations liable to occur during routine operations.

The HAZOP method is based on the following steps:

- Describe the system, its functions and features (may be done by means of a preparatory Functional Analysis);
- List all the "nodes" of the system. Nodes are basic parts of the P&ID that are homogeneous in their function from the process point of view;
- List all the significant deviations of the parameters of each node (pressure, flow-rate, temperature…) by means of keywords (too high, to low…);
- Find out the possible reasons of deviations;
- Find out the potential effects of the deviations while taking into account the different operation steps of the system;
- List the means to reduce the risks. (lower the probability of failure, lower the severity level...). Identify the possible mean to detect the deviation risk.
- Assess the severity and the criticality of the residual risks while taking into account the risk mitigation measures previously set up.
- List some guidelines to be implemented to reduce the residual risk when it is too serious.

Comment [FB1]: Page : 5
Le fait on vraiment, avant la réduction du risque ?

Kim C-Coggs 4/3/09

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AIR LIQUIDE ÉQUIPE CONTRÔLE DES PROCÉDÉS	What is an HAZOP ?	Date : 1/02/04 Author : Emmanuel Lardeux R&D : CRCD – Process Control

An example step by step

The results of an analysis can be summarized in a table, where steps are listed in columns. Deviations of a node are investigated in rows.

Node	Function	Deviation	Causes	Effects	Means of reduction	L	S	C	Action	Comment	Ref

Table 1: HAZOP table

The columns contain the following information:

Node .... Description of a functional element of system.

Function .... Purpose of the node. What is the node for.

Deviation .... Possible deviation of a parameter of the node. The parameters may be "Pressure", "Flow-rate", "Temperature", "Level", "Composition"… Deviation is guess by systematic application of key words: "More", "Less", "No", "Too high"… One line of the table stands for one deviation.

Causes .... Are indicated the possible causes of the deviation.

Effect .... Possible effect caused by the deviation on the system and the process. Currently set up means of reduction are not taken into account yet at this stage of the analysis.

Means of reduction .... Are indicated the risk reduction means. One may act over the causes to reduce the happening probability of the risk, or one may act over the consequences to minimize its severity.

L (Likelihood) .... Is assessed the happening frequency of the deviation while taking into account the currently set up risk reduction measures.

S (Severity) .... Is assessed the severity level of the effect, taking into account the risk reduction means. Severity classes are presented below.

C (Criticality) .... Criticality is a combination of the likelihood and the severity of a risk.

A (Action) .... Guidelines to be implemented to reduce the residual risks. Guidelines must be implemented when the criticality of the risk is high.

Comments .... Comments.

Reference (Ref) .... Identification of the row.

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Risk levels

Likelihood levels

Level	Wording	Expected rate of occurrence
1	Unlikely	Has never happened before, or has happened before and has been completely cleared
2	Possible	Happened before, have been cleared on a different system or environment
3	Frequent	Happens regularly. There is no way to tackle the risk nor Air Liquide experience on this matter.

Table 2: Probability classes

Severity Level

The severity level is categorized with respect to the nature of the effect:

- **Safety**: Person safety working on the system or near-by, impact on the environment
- **Availability**: system availability, ability of the system when it is meant to produce

	Severity		
Impact	1	2	3
Safety	Minor injury First Aid only	Serious injury Medical treatment	Disabling injury or fatalities
Availability	Minor loss or facility damage No impact to the client Low cost	Return on investment is badly hit Impact to the client	Sustainability of the project is affected. Client or AL plant partially or totally wiped out

Table 3: Severity classes

Level of Criticality

Criticality is a combination of the likelihood and the severity of a risk, according to the table below.

Severity Likelihood	1	2	3
1	I	I	II
2	I	II	III
3	I	III	III

Table 4: Risk assessment categories

Khi C. Ooge 4/3/09

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Direct assessment of the criticality

In some studies, the level of criticality may be assessed directly according to the criteria below:

- Criticality I : Risk mitigation measures are effective so that the residual risk is minor. It is said that risk is under control. There is no need for extra risk mitigation measure
- Criticality II : Residual risk is not satisfactory. Extra risk mitigation measures (preventive measures, detection, protection..) are required.
- Criticality III : Residual risk cannot be beard. Risk mitigation measures are definitely required. When no solution is identified or when measures failed to be completed, the project must stop.

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AIR LIQUIDE

O3 Delivery system

Hazard and Operability Study (HAZOP)

3-April-2008

HAZOP – Hazard and Operability study

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- The purpose of an HAZOP study is to analyze the operation integrity of a system. An HAZOP identifies the effects of deviations that might occur during normal operation of the system.
- Objectives of HAZOP study:
  - Identify the risks of malfunction of an installation (reliability, safety or the environment)
  - Check the installed means of detection and protection
  - Recommend changes in the design and/or fitting of protection systems and means of detection to reduce the level of risk
- "Inductive reasoning" type analysis (Causes → Results)

HAZOP – Hazard and Operability study

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- Typical steps of an HAZOP
  - Describe the system, its functions and features
  - Define all the "**nodes**" of the system. Nodes are basic part of the system that are homogeneous in their function from a process point of view. It is chosen in such a way as to correspond to an intention (pressure reduction, heating, storage, etc...)
  - List **all** the possible **deviations** of the parameters of each node
  - Determine the possible **causes** of deviations
  - Determine the **effects** of deviations
  - List the means to **reduce the risk**
  - Assess the **severity** and **criticality**
  - List some guidelines to implement the risks

HAZOP – Hazard and Operability study

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- HAZOP Method detailed in Risk Management Procedure PMR-G-04
- Tool used for HAZOP

  - Standard software package used by Air Liquide America
  - Uses a systematic approach
  - Formalizes HAZOP study

Method

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- The concept involves investigating how the plant might deviate

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KC Date: 4/3/09

Project No. DRTC-2008-HAZOP-02

NCFST Ozone Delivery System

Leader: Ted Rekart

Scribe: Ted Rekart

Meeting Location: NCFST Moffet Center, Chicago, IL

Meeting Days: 2

Unit: Ozone Delivery System

June 2008

Air Liquide Ozone Delivery System for Destruction of Microorganisms. Collaborative effort between Air Liquide DRTC and NCFST

Kri C. Cog

Table 1 Drawings Used in the Analysis

Type	Number	Title	Revision	Revision Date	Document Location
Procedure	MC-SA-105-SOP002-V01	Emergency Shutdown Procedures	V-01		
Procedure	MC-SA-105-SOP003-V01	Disconnecting the Oxygen and Nitrogen Cylinders	V-01		
Procedure	MC-SA-105-SOP004-V01	Ozone System General Safety Procedures	V-01		
Procedure	MC-SA-105-SOP005-V01	Setting Needle Valves for the Ozone System	V-01		
Procedure	MC-SA-106-SOP001-V01	Ozone System Startup and shutdown Procedure	V-01		
Drawing	P&ID	H1 Series Ozone Analyzer	-	6/20/2008	
Drawing	P&ID	Ozonia Ozat Model CFS-1A Ozone Generator	-	6/20/2008	
Drawing	PFD/P&ID	Ozone Design system	1	6/9/2008	

Table 2 Team Members

First Name	Last Name	Role	E-Mail	Phone	Job Title	Company
Bharat	Aluri		Baluri@iit.edu		Student	NCFST
Kerri	Cooper	Operator	kcooper4@iit.edu	803-669-3727	Student	NCFST
Todd	Diel		diel@iit.edu	703-563-8190	Safety Officer	NCFST
Steven	Fisher	Ozone Operations Expert	Steven.Fisher@AirLiquide.com	(708) 579-7709	Project Engineer	AL Consultant
Vishwesh	Kelkar		vkelkar@iit.edu		Student	NCFST
Vasuhi	Rasanayagam	AL Project Leader	vasuhi.rasanayagam@airliquide.com	302-286-5439	Scientist	American Air Liquide - DRTC
Ted	Rekart	RMR/HAZOP Leader	Theodore.Rekart@airliquide.com	302-286-5514	Safety Officer	American Air Liquide - DRTC
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Peter	Slade	PI	slade@it.edu	708-563-8172	Director of Education & Outreach	NCFST
Ed	Steiner	Lead Engineer	steiner@iit.edu	708-563-8273	Director of Facilities & Pilot Plant	NCFST
Lei	Wang	Operator	Lwang59@iit.edu		Student	

Table 3 Action Items

Type	No.	Action	Status	Responsibility	Date Complete	References
Recommendation	1	Add relief valve between NV1 and BV1 to outside building	Completed	Cooper/Slade	6/12/2008	1.1 High Oxygen Flow — Ozone Generator & Oxygen supply 1.4 High ozone generator temperature — Ozone Generator & Oxygen supply
Recommendation	2	Ensure standard operating procedure sets secondary side of regaulator to zero before opening oxygen valve.	Completed	Kerri Cooper, Lei Wang	6/17/2008	1.1 High Oxygen Flow — Ozone Generator & Oxygen supply 1.2 Low oxygen flow — Ozone Generator & Oxygen supply
Recommendation	3	Verify standard operating procedure precaition on adequate oxygen supply before startup	Completed	Cooper/Wang	6/17/2008	1.2 Low oxygen flow — Ozone Generator & Oxygen supply
Recommendation	4	Ensure standard operating procedure addresses venting and purging the buffer tank when experiment is completed and before any maintenance.	Completed	Coopr/Wang	6/10/2008	1.3 Reverse oxygen flow — Ozone Generator & Oxygen supply
Recommendation	5	Add flow meter to cooling water flow to ozone generator and verify that the standard operating procedure addresses verifying cooling water flow prior to ozone generator startup	Completed	Coopr/wang	6/10/2008	1.4 High ozone generator temperature — Ozone Generator & Oxygen supply
Recommendation	6	Verify standard operating procedure valve alignments	Completed	Cooper/Wang	6/10/2008	1.6 High ozone pressure at Generator Outlet — Ozone Generator & Oxygen supply
Recommendation	7	Verify standard operating procedure addresses nitrogen purging of anaerobic reaction tank with BV4 closed prior to inserting samples	Completed	Cooper/wang	6/10/2008	2.4 Misdirected ozone flow — Ozone Humidification Line
Recommendation	8	Outlet routinely checked for ambient ozone concentration	Completed	Cooper/wang	6/10/2008	3.1 High ozone concentration exiting column — MnO2 Ozone Destruction Column
Recommendation	9	Add MnO2 Destruct column thermocouple, determine operating limits and standard operating procedure response	Completed	Cooper/wang	6/10/2008	3.3 High destruct column temperature — MnO2 Ozone Destruction Column
Recommendation	10	Ensure standard operating procedure sets flow to ozone analyzer to recommended flow setting	Completed	Cooper/Wang	6/17/2008	4.1 High ozone flow — Ozone Analyzer

Type	No.	Action	Status	Responsibility	Date Complete	References
Recommendation	11	Add internal process flow diagram for ozone analyzer	Completed	Cooper/Wang	6/19/2008	4.8 Low ozone pressure — Ozone Analyzer
Recommendation	12	Add internal process flow diagram for ozone generator	Completed	Cooper Wang	6/13/2008	1.1 High Oxygen Flow — Ozone Generator & Oxygen supply
Recommendation	13	Standard operating procedure should contain steps to verify nitrogen flow	Completed	Cooper/wang	6/13/2008	5.2 Low/no nitrogen flow — Nitrogen supply
Recommendation	14	Add check valve at exit of ozone generator before BV7 & BV2	Completed	Cooper/Steiner	6/10/2008	5.4 Misdirected nitrogen flow — Nitrogen supply
Recommendation	15	Add to standard operating procedure procedure to calculate scrubber capacity and monitor ozone effluent gas stream accordingly	Completed	Cooper/Wang	6/17/2008	6.1 High ozone concentration exiting KI destruction tank — Anaerobic Reaction Tank
Recommendation	16	Evaluate hood set up and install velocimeter	Completed	Cooper/Steiner	6/16/2008	7.1 High flow — Fume Hood
Recommendation	17	Standard operating procedure needs to have emegency response to turn off all gas supplies	Completed	Cooper/Wang	6/16/2008	7.2 Low/no flow — Fume Hood
Recommendation	18	Verify nitrogen supply and room flooding calculations	Completed	Cooper/Steiner	6/17/2008	7.2 Low/no flow — Fume Hood
Recommendation	19	Standard operating procedure and P&ID's need revised	Completed	Cooper/wang/Slade	6/18/2008	

Table 4 List of Sections

No.	Type	Name	Description	Status	Drawings
1	Line/Pipe	Ozone Generator & Oxygen supply	Oxygen Cylinder, supply tubing and components including Ozone Generator		PFD/P&ID
2	Line/Pipe	Ozone Humidification Line	Line from Ozone Generator to the inlet of the Anaerobic Reaction Tank, including the Buffer Tank, Humidification tank and Excess Water Tank		PFD/P&ID
3	Tank/Vessel	MnO2 Ozone Destruction Column	Ozone destruction column (MnO2) and bypass lines to the column and valves BV2 & BV3		PFD/P&ID
4	Other	Ozone Analyzer	In-line Ozone Analyzer including valve NV2		PFD/P&ID
5	Line/Pipe	Nitrogen supply	Purge Nitrogen supplies for the anaerobic reaction tank and the ozone buffer tank through humidification train to the anaerobic reaction tank		PFD/P&ID
6	Tank/Vessel	Anaerobic Reaction Tank	Anaerobic reaction tank and KI destruction tank		PFD/P&ID
7	Other	Fume Hood	Fume hood containing experimental setup		

Table 5 for use in PHA Risk Matrix Used in Analysis

	0 No bodily injury, no damage to environment, no damage to eqiupment or production.	1 Minor injury, moderate damage with no durable effect on environment, or damage to small and medium-sized eqiupment, or a brief loss of production (several hours)	2 Serious injury, serious damage to environment but may be corrected, or damage to large equipment or lost of production (several days)	3 Potential victim, serious and durable damage to environment, or damage to very large items of equipment or extended loss of production (several weeks to months)	4 Major accident with potentiality of several victims, ecological catastrophe or massive destruction of facilities or total lost of production (permanent shutdown)
4 Frequent Event occur more than once every 10 years.	04 Low risk	14 Studies to minimize risk must be conducted	24 Unacceptable risk	34 Unacceptable risk	44 Unacceptable risk
3 Possible Event occurs between every 10 to 1000 years	03 Low risk	13 Low risk	23 Studies to minimize risk must be conducted	33 Unacceptable risk	43 Unacceptable risk
2 Rare Has happended to structures, static equipment, or redundant equipment	02 Low risk	12 Low risk	22 Low risk	32 Studies to minimize risk must be conductied	42 Unacceptable risk
1 Very rare The event requires the occurrence of two rare events	01 Low risk	11 Low risk	21 Low risk	31 Low risk	41 Studies to minimize risk must be conducted
0 Improbable The event has never occurred	00 Low risk	10 Low risk	20 Low risk	30 Low risk	40 Low risk

Company: DRTC-NCFST	**Plant:**	**Site:**	**Unit:** Ozone Delivery System	**System:**

Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)

No.: 1 — Oxygen Cylinder, supply tubing and components including Ozone Generator

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
1.1	High Oxygen Flow	Oxygen pressure regulator failure Valve misalignmemt	Loss of containment	2	3	23	2	22	Oxygen regulator Needle valve Hood & Ventilation
			Potential for internal damage to in-line component	2	3	23	2	22	
			High ozone pressure at Generator Outlet (see 1.6)	2	3	23	2	22	
			High ozone flow - Ozone Humidification Line (see 2.1)						
			High ozone pressure - Anaerobic Reaction Tank (see 6.5)						
1.2	Low oxygen flow	Closed valve High pressure downstream Low pressure upstream Oxygen supply depletion	High temp in Ozone generator	1	4	14	3	13	Automatic high temp shutdown of ozone generator
			High ozone generator temperature (see 1.4)						
			Low ozone pressure at generator outlet (see 1.7)						
			Low/no ozone flow - Ozone Humidification Line (see 2.2)						
			Low/no ozone flow - Ozone Analyzer (see 4.2)						
1.3	Reverse oxygen flow		Line Pressurized	0	4	04	4	04	
1.4	High ozone generator temperature	High ambient temperature Loss of cooling water No oxygen flow (see 1.2)	High temperature in ozone generator	1	4	14	3	13	Automatic high temp shutdown of ozone generator
			Low ozone pressure at generator						

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
No.: 1	Oxygen Cylinder, supply tubing and components including Ozone Generator								
			outlet (see 1.7)						
1.5	Low ozone generator temperature	Low cooling water temperature	Condensation of water causing electrical arcing	2	2	22	2	22	Only tap water used for cooling
1.6	High ozone pressure at Generator Outlet	High Oxygen Flow (see 1.1)							
		Valve misalignment							
1.7	Low ozone pressure at generator outlet	Low oxygen flow (see 1.2)							
		High ozone generator temperature (see 1.4)							

Company: DRTC-NCFST	**Plant:**	**Site:**	**Unit:** Ozone Delivery System	**System:**
Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)				

No.: 2 Line from Ozone Generator to the inlet of the Anaerobic Reaction Tank, including the Buffer Tank, Humidification tank and Excess Water Tank

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
2.1	High ozone flow	humidity tank and excess water release tank glass stoppers loose High Oxygen Flow - Ozone Generator & Oxygen supply (see 1.1)	system depressurization	0	3	03	3	03	Experiment is in a ventilated hood
			release of ozone	2	3	23	2	22	
			High ozone pressure (see 2.5)						
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
2.2	Low/no ozone flow	Valve misalignment Low oxygen flow - Ozone Generator & Oxygen supply (see 1.2)	Loss of production and efficiency	1	2	12	1	11	Trained personnel & standard operating procedure
			Low/no ozone flow - Ozone Analyzer (see 4.2)						
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						
2.3	Reverse ozone flow	Humidity tank and excess water release tank glass stoppers loose	system depressurization	0	3	03	3	03	Hood and ventilation Isolation valve BV4
			release of ozone	2	3	23	2	22	
2.4	Misdirected ozone flow	valve misalignment	No safety consequences						
2.5	High ozone pressure	High ozone flow (see 2.1) High destruct column pressure - MnO2 Ozone Destruction Column (see 3.5) High ozone pressure - Anaerobic Reaction Tank (see 6.5)	Buffer tank relief valve releases ozone	2	3	23	2	22	Hood and ventilation
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
2.6	Low ozone pressure	Low ambient temperature Low temperature upstream	No additional safety related consequences						

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Company: DRTC-NCFST	Plant:	Site:	Unit: Ozone Delivery System	System:
Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)				

No.: 3	Ozone destruction column (MnO2) and bypass lines to the column and valves BV2 & BV3								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
3.1	High ozone concentration exiting column	Wet column causing channelling Water/water vapor in column (see 3.7)	Reduced ozone destruction	2	2	22	1	21	Check valve CV1 prevents wet gas backflowing to destruction column Dry gas feed used for ozone generation
			Water/water vapor in column (see 3.7)						
3.2	Low ozone concentration	No safety related issues							
3.3	High destruct column temperature	Normal operating situation as ozone destruction is exothermic	Fire and burn hazard	2	3	23	2	22	Themocouple on column exit gas to monitor temperature
3.4	Low column temperature	No safety related consequences	Freezing of water						
			Low pressure						
3.5	High destruct column pressure	Column inlet or exit screens plugged Water/water vapor in column (see 3.7)	High ozone pressure - Ozone Humidification Line (see 2.5)						
3.6	Low column pressure	No safety related consequences							
3.7	Water/water vapor in column	High ozone concentration exiting column (see 3.1)	High ozone concentration exiting column (see 3.1)						
			High destruct column pressure (see 3.5)						

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Company: DRTC-NCFST	Plant:	Site:	Unit: Ozone Delivery System	System:
Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)				

No.: 4 In-line Ozone Analyzer including valve NV2

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
4.1	High ozone flow	Valve misalignment High ozone pressure (see 4.7)	Inaccurate ozone analyzer readings	0	4	04	3	03	Trained Operator & SOP
			overpressure of ozone analyze with possible ozone release	2	2	22	1	21	
			High ozone pressure (see 4.7)						
4.2	Low/no ozone flow	Valve misalignment	No safety related consequences						
		Low oxygen flow - Ozone Generator & Oxygen supply (see 1.2)							
		Low/no ozone flow - Ozone Humidification Line (see 2.2)							
4.3	Reverse ozone flow	Valve misalignment	No safety related consequence						
4.4	Misdirected ozone flow	No credible cause							
4.5	High ozone temperature	No credible causes not already analyzed							
4.6	Low ozone temperature	No credible cause							
4.7	High ozone pressure	High ozone flow (see 4.1)	High ozone flow (see 4.1)						
4.8	Low ozone pressure	No credible safety cause							

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Company: DRTC-NCFST	**Plant:**	**Site:**	**Unit:** Ozone Delivery System	**System:**
Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)				

No.: 5 — Purge Nitrogen supplies for the anaerobic reaction tank and the ozone buffer tank through humidification train to the anaerobic reaction tank

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
5.1	High nitrogen flow	Valve misalignment (pressure regulator second stage set two high High gas flow - Anaerobic Reaction Tank (see 6.7)	lift relief in anaerobic reaction tank with possible ozone release	2	3	23	2	22	Hood and ventilation Training & standard operating procedure
			High nitrogen pressure (see 5.7)						
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						
5.2	Low/no nitrogen flow	Valve misalignment or regulator secondary stage not set properly Empty supply cylinder Low nitrogen pressure (see 5.8) Low/no gas flow - Anaerobic Reaction Tank (see 6.8)	Partial or no purge of ozone with potential ozone release	2	3	23	2	22	Hood and ventilation
			Low nitrogen pressure (see 5.8)						
			Low/no gas flow - Anaerobic Reaction Tank (see 6.8)						
5.3	Reverse nitrogen flow	No credible cause							
5.4	Misdirected nitrogen flow	Operator error -- valve misalignment	Nitrogen backflow in to the ozone generator	1	3	13	2	12	Trained Operator & standard operating procedure Valve to be added to outlet of ozone generator
5.5	High nitrogen temperature	No credible cause							
5.6	Low nitrogen temperature	No credible cause							
5.7	High nitrogen pressure	High nitrogen flow (see 5.1)	Potential ozone release through the relief valve on buffer tank	2	3	23	2	22	Hood and ventilation
			Potential ozone release through the relief valve on anaerobic reaction tank	2	3	23	2	22	

No.: 5	Purge Nitrogen supplies for the anaerobic reaction tank and the ozone buffer tank through humidification train to the anaerobic reaction tank								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
			Lifting of glass stopper on humidity tank and excess water relaease tank releasing ozone	2	3	23	2	22	
			High gas flow - Anaerobic Reaction Tank (see 6.7)						
5.8	Low nitrogen pressure	Low/no nitrogen flow (see 5.2)	Low/no nitrogen flow (see 5.2)						
5.9	Loss of Nitrogen containment	Cylinder or cylinder valve failure	Oxygen deficient atmosphere	3	2	32	1	31	Ambient oxygen monitor

Company: DRTC-NCFST	Plant:	Site:	Unit: Ozone Delivery System	System:

Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)

No.: 6 — Anaerobic reaction tank and KI destruction tank

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
6.1	High ozone concentration exiting KI destruction tank	Depleted KI	ozone release	2	3	23	2	22	Hood and ventilation Ambient ozone analyzer Nitrogen flooding calculation shows 17% oxygen is possible in static room from one cylinder of nitrogen
6.2	Low ozone concentration	Valve misalignment	No significant safety consequences						
6.3	High ozone temperature	No credible cause	no significant safety consequences						
6.4	Misdirected ozone flow	Standard operating procedure not followed with nitrogen tank changeout not in proper sequence	Ozone release	2	3	23	2	22	Standard operating procedure to purge anaerobic reaction tank with BV4 closed Ventilation system and hood
6.5	High ozone pressure	High Oxygen Flow - Ozone Generator & Oxygen supply (see 1.1)	Potential release through the relief valve	2	3	23	2	22	Hood & ventilation
			High ozone pressure - Ozone Humidification Line (see 2.5)						
6.6	Low ozone pressure	No credible safety related issues							
6.7	High gas flow	High nitrogen flow - Nitrogen supply (see 5.1) High nitrogen pressure - Nitrogen supply (see 5.7) High ozone flow - Ozone Humidification Line (see 2.1) High ozone pressure - Ozone Humidification Line (see 2.5)	High nitrogen flow - Nitrogen supply (see 5.1)						
6.8	Low/no gas flow	Low/no nitrogen flow - Nitrogen supply (see 5.2) High nitrogen flow - Nitrogen supply (see 5.1) Low/no ozone flow - Ozone	Low/no nitrogen flow - Nitrogen supply (see 5.2)						

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No.: 6	Anaerobic reaction tank and KI destruction tank								
Item	**Deviation**	**Causes**	**Consequences**	**S**	**UL**	**UR**	**ML**	**MR**	**Safeguards**
		Humidification Line (see 2.2) Low ozone pressure - Ozone Humidification Line (see 2.6)							

Company: DRTC-NCFST	Plant:	Site:	Unit: Ozone Delivery System	System:
Team Members: Kerri Cooper (Operator), Todd Diel (), Steven Fisher (Ozone Operations Expert), Vasuhi Rasanayagam (AL Project Leader), Ted Rekart (RMR/HAZOP Leader), Claudia Rodriguez (Co-PI), Peter Slade (PI), Ed Steiner (Lead Engineer), Lei Wang (Operator)				

No.: 7 — Fume hood containing experimental setup

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
7.1	High flow	Improper setup of hood openings	Backlfow and eddy currents into the room	2	3	23	2	22	Trained Operator and SOP
7.2	Low/no flow	Power failure	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI Ambient oxygen monitor
			High oxygen exposure to room occupants	2	3	23	2	22	
			nitrogen flooding of work space	3	3	33	1	31	
		Operator error, not turning in the hood blower	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI
		Mechanical failure of hood blower	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI Ambient oxygen monitor Hood velocimeter Limited nitrogen supply relative to room volume
			High oxygen exposure to room occupants	2	3	23	2	22	
			nitrogen flooding of work space	3	3	33	1	31	
		Operator error, PVC curtain misplacement	Potential ozone exposure to the occupants of the room	2	3	23	2	22	Ozone destruction tank MnO2 Ozone destruction tank KI Ambient oxygen monitor Hood velocimeter Limited nitrogen supply relative to room volume

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No.: 7 — Fume hood containing experimental setup

Item	Deviation	Causes	Consequences	S	UL	UR	ML	MR	Safeguards
			High oxygen exposure to room occupants	2	3	23	2	22	
			nitrogen flooding of work space	3	3	33	1	31	
7.3	Reverse flow	No credible cause							
7.4	High temperature	No credible cause							
7.5	Low temperature	No credible cause							