

Hermetically Sealed Encasements for Historic  
Document Display and Preservation

by

Keith Vaughn Durand

Submitted to the Department of Mechanical Engineering  
in partial fulfillment of the requirements for the degree of

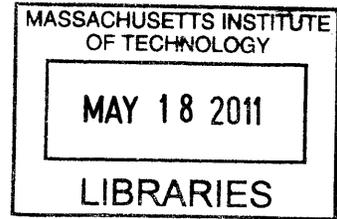
Doctor of Philosophy in Mechanical Engineering

at the

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

February 2011

© Massachusetts Institute of Technology 2011. All rights reserved.



**ARCHIVES**

Author .....

Department of Mechanical Engineering

January 14, 2011

Certified by..

Alexander H. Slocum  
Pappalardo Professor of Mechanical Engineering  
Thesis Supervisor

Accepted by .....

.....

David E. Hardt  
Chairman, Department Committee on Graduate Students



# Hermetically Sealed Encasements for Historic Document Display and Preservation

by

Keith Vaughn Durand

Submitted to the Department of Mechanical Engineering  
on January 14, 2011, in partial fulfillment of the  
requirements for the degree of  
Doctor of Philosophy in Mechanical Engineering

## Abstract

The goal of this thesis was to develop designs and methods for the preservation and display of historic documents. The results were applied via the design, manufacture, and installation of five hermetic display encasements for the original Massachusetts Constitution (1780), a Goddard broadside of the Declaration of Independence (1777), an original copy of the Bill of Rights (1789), the 1629 Charter of Massachusetts Bay, and the 1692 Charter of the Province of Massachusetts Bay. In addition to meeting the aesthetic requirements for a permanent exhibit, the encasements had to have leak rates that would maintain less than 0.5% oxygen content over 20 years and preserve 40% relative humidity at 70 °F. Furthermore, the encasement portion of the project had a 300,000 USD total budget, approximately one tenth the budget allocated for the "Charters of Freedom" project at the National Archives.

The encasements designed for this project incorporated a novel seal arrangement that "floats" the glass and allows for helium leak testing at any time, without disturbing the document. These design choices were motivated by developing a model for permeation of gases through a network of polymer seals. Preparing the preservation environment also required the design and fabrication of the "Moisturematic", a device that allows for controlled filling of a vessel with precisely humidified gas. Continuous monitoring of the atmosphere inside the encasements was made possible by the development of specialized instrumentation.

In January 2009, these historic documents were put on permanent display in the Treasures Gallery at The Massachusetts Archives. Initial leak testing indicates that all encasement have preservation quality environments exceeding 20 years.

In addition to design rules for developing high quality, relatively low cost environments for preservation, this thesis presents a roadmap for design of more generalized hermetic sealing and conditioned environments.

Thesis Supervisor: Alexander H. Slocum

Title: Pappalardo Professor of Mechanical Engineering



## Acknowledgments

I would like to thank my advisor, Prof. Alexander Slocum for his support and guidance throughout my graduate career. I would also like to thank Prof. Daniel Frey and Prof. Douglas Hart for serving on my thesis committee, and the graduate students of the Precision Engineering Research Group for friendship and scholarship.

There were many people instrumental to the success of the Massachusetts Archives encasement project. Richard Slocum worked tirelessly to keep the project on course and on budget. Josh Dittrich and Eric Correll logged many hours assisting with fabrication, assembly, and testing of the encasements. David Arsenault donated several Saturdays to complete the orbital welding of encasement components.

Throughout the project, the Massachusetts Archives staff have been enthusiastic and accomodating. Michael Comeau deserves special recognition for his vision for the Treasures Gallery at the newly renovated Commonwealth Museum. Without him, the encasement project would have never even started.

I would like to thank Richard Rhorer and Charles Tilford at the National Institute of Standards and Technology for sharing their experiences and insights gained from the Charters of Freedom project and the Waldseemüller Map encasement project.

David McCarthy from Varian coordinated the loan of a helium leak detector and provided instruction and support. Lesley Yu of National Instruments secured product donations and assisted with software development for encasement monitoring. Gary Sullivan of Swagelok provided technical advice and secured for us a significant discount. Walter Newman of the New England Document Conservation Center provided assistance with loading documents into the encasements– and told us we could touch the Bill of Rights.

I would also like to thank my family and friends for encouragement, advice, and copy editing. Vanessa Wood, Blake Stacey, and Brian Neltner deserve special recognition for their contributions.



# Contents

<b>1</b>	<b>Introduction</b>	<b>23</b>
1.1	Previous Encasement Designs . . . . .	26
1.1.1	The Magna Carta . . . . .	26
1.1.2	The Royal Mummy Collection and the Indian Constitution . .	28
1.1.3	The Charters of Freedom . . . . .	28
1.1.4	The Waldseemüller map . . . . .	29
1.2	The Massachusetts Archives Project: Advancing Science and Historical Preservation Techniques . . . . .	31
<b>2</b>	<b>Sealing Technologies</b>	<b>33</b>
2.1	Helium leak detection . . . . .	34
2.1.1	Physical leaks vs. permeation . . . . .	35
2.2	Metal seals . . . . .	37
2.3	Energized plastic seals . . . . .	39
2.4	Elastomer seals . . . . .	40
2.4.1	Designing for elastomer seals . . . . .	40
2.4.2	O-ring manufacturing . . . . .	47
2.5	Leakage analysis . . . . .	48
2.5.1	Single seal, single volume analysis . . . . .	49
2.5.2	Multiple seal, multiple volume analysis . . . . .	52
2.5.3	Leak budgeting . . . . .	53
2.6	Oxygen scavenging . . . . .	55
2.7	Massachusetts Archives encasement testing . . . . .	56

2.8	Conclusion . . . . .	57
<b>3</b>	<b>Mechanical Design</b>	<b>59</b>
3.1	A few words on design process . . . . .	59
3.2	Anatomy of an encasement . . . . .	61
3.3	Tub, clamp plate, and glass . . . . .	61
3.4	Plumbing design . . . . .	64
3.5	Platen design . . . . .	68
3.5.1	Digitization of document outlines . . . . .	69
3.5.2	Platen design details . . . . .	70
3.5.3	Document clips . . . . .	70
3.5.4	Platen mounting . . . . .	73
3.6	Second-generation encasement design . . . . .	77
<b>4</b>	<b>Fabrication Tooling and Techniques</b>	<b>81</b>
4.1	Tubing run fabrication . . . . .	81
4.1.1	Tube bending . . . . .	82
4.1.2	Tube trimming . . . . .	86
4.1.3	Tube welding . . . . .	86
4.2	Finish polishing . . . . .	89
4.2.1	Fitting block preparation . . . . .	90
4.2.2	Encasement polishing . . . . .	91
4.2.3	O <sub>2</sub> getter bottle polishing . . . . .	94
<b>5</b>	<b>Instrumentation</b>	<b>97</b>
5.1	Massachusetts Archives instrumentation design . . . . .	97
5.1.1	Overall layout . . . . .	97
5.1.2	Pressure sensing . . . . .	98
5.1.3	Humidity sensing . . . . .	99
5.1.4	Acquisition hardware . . . . .	113
5.1.5	Software . . . . .	120

5.1.6	System preparation . . . . .	122
5.1.7	Conclusion . . . . .	128
<b>6</b>	<b>Humidity Generation</b>	<b>131</b>
6.1	Humidity generation methods . . . . .	132
6.1.1	Dilution method . . . . .	132
6.1.2	Two temperature method . . . . .	133
6.1.3	Two pressure/two temperature method . . . . .	133
6.2	Commercial humidity generation equipment . . . . .	136
6.3	Purge parameters . . . . .	137
6.4	The Moisturematic . . . . .	138
6.4.1	Design Approach . . . . .	138
6.4.2	Saturator Design . . . . .	140
6.4.3	Schematic and description of components . . . . .	145
6.4.4	Integration . . . . .	148
6.4.5	Safety . . . . .	152
6.4.6	Operational Envelope and Accuracy . . . . .	152
6.4.7	Operation . . . . .	155
6.4.8	Future work . . . . .	155
<b>A</b>	<b>Procedures</b>	<b>159</b>
A.1	Cleaning . . . . .	159
A.1.1	Glass . . . . .	159
A.1.2	Small metal parts . . . . .	159
A.1.3	Tub and clamp . . . . .	160
A.1.4	Platens . . . . .	160
A.1.5	Melinex document clips . . . . .	160
A.1.6	O-rings . . . . .	160
A.2	Encasement Assembly . . . . .	161
A.2.1	Fasteners and fittings . . . . .	161
A.2.2	Base assembly . . . . .	162

A.2.3	Helium leak checking the base assembly . . . . .	164
A.2.4	Topside assembly . . . . .	167
A.2.5	Glass and clamp preparation . . . . .	169
A.2.6	Platen preparation . . . . .	169
A.2.7	Final assembly . . . . .	172
A.3	Moisturematic operation . . . . .	175
A.4	Helium leak testing . . . . .	178
<b>B</b>	<b>Humidity Sensor Code</b>	<b>181</b>
<b>C</b>	<b>Supplier Information</b>	<b>187</b>

# List of Figures

1-1	Photographs of the Treasures Gallery at the Massachusetts Archives showing the 5 cases designed, manufactured, and assembled for this thesis. <i>Photographs courtesy of the Massachusetts Archives.</i> . . . . .	24
1-2	Photographs of a Goddard broadside of the Declaration of Independence (1777), the original Massachusetts Constitution (1780), an original copy of the Bill of Rights (1789), the 1629 Charter of Massachusetts Bay, and the 1692 Charter of the Province of Massachusetts Bay, showing their excellent condition. <i>Photographs courtesy of the Massachusetts Archives.</i> . . . . .	25
1-3	Photograph of the current encasement for the Magna Carta, completed in the 1980s. . . . .	27
1-4	Base of the Waldseemüller map encasement in the NIST shop. This encasement was under construction concurrently with The Massachusetts Archives encasements. . . . .	30
2-1	Schematic of helium leak detection for an encasement. . . . .	35
2-2	Time response of helium permeation through a -334 (2.6" ID × 0.210" CS) Viton A o-ring using 35% Helium/63% Argon/2% CO <sub>2</sub> tracer gas. Values of $1 \times 10^{-12}$ atm – cc/sec indicate the lower detection limit of the Varian VS MD30 helium leak detector. Helium was not detected until 12 minutes after the flow of tracer gas was started. . . . .	37
2-3	Helicoflex and C-Flex™ metal seals. <i>Garlock Helicoflex</i> . . . . .	38
2-4	An energized plastic “OmniSeal.” <i>St. Gobain Performance Plastics</i> . . . . .	39

2-5	Cross section of encasement with floating glass. . . . .	43
2-6	Dovetail o-ring gland machining. <i>Harvey Tool Company, LLC</i> . . . .	44
2-7	O-ring permeation study assumptions. . . . .	46
2-8	A defective splice in an extruded and centerless-ground o-ring. . . . .	48
2-9	Schematic of seal network model. . . . .	54
3-1	Encasement exploded view. . . . .	62
3-2	Machining the tub at The Bechdon Company. . . . .	63
3-3	Custom fitting block for connecting stainless tubing to the encasement tub. . . . .	65
3-4	Top panel: Plumbing for the encasement interior volume. Bottom panel: Plumbing for the inner sensing groove. The large cylinder con- tains oxygen absorber. . . . .	67
3-5	Testing clearances for a proposed design of a wheeled cart. The cart design required some minor changes to enable access to all fittings. . .	68
3-6	Platen outlines of the three “portrait” orientation documents. . . . .	71
3-7	Platen hole pattern to promote gas contact with document. . . . .	72
3-8	Die cutting pattern for the document clips. Material is 1000 gauge Melinex 515. . . . .	73
3-9	Steel rule die for cutting document clips. . . . .	74
3-10	Document clips in uninstalled and installed configurations. . . . .	74
3-11	Document clip bending fixtures. . . . .	75
3-12	Kinematic coupling exploded view. . . . .	76
3-13	Kinematic couplings on document platen. . . . .	76
3-14	Second-generation encasement cross section. . . . .	78
3-15	Revised sensing groove arrangement to allow for improved helium leak testing ability. . . . .	79
3-16	Revised manifold to simplify manufacturing and reduce parts count. . . . .	79
3-17	Positive-locking platen clip, actuated by flip-out handles . . . . .	80
4-1	Minimum end tangents of a Swagelok MS-HTB-4 bender. . . . .	83

4-2	Minimum tangents for tube manufacturing and path length determination for an offset bend. . . . .	83
4-3	Photographs showing the offset bend fixture (top), which is used to hold a tube in the correct shape while trimming to length with a mill (bottom). . . . .	84
4-4	Bending sequence for an offset bend. . . . .	85
4-5	90° bend fixture allows individual legs to be measured to determine how much material to remove. . . . .	87
4-6	Welding fixture to align components and provide connections for shielding the inside of the tube with inert gas. . . . .	88
4-7	Custom purge adaptors for welding Swagelok Micro-Fit tees. . . . .	90
4-8	Polished block with defect in sealing surface . . . . .	92
4-9	The o-ring groove is polished using foam poster board as a soft sanding block. . . . .	93
4-10	The encasement is inspected with low ambient light and a flashlight. . . . .	94
4-11	Getter bottle polishing. . . . .	95
5-1	Lantronix Ethernet bridges . . . . .	99
5-2	Pressure sensor electrical connections. . . . .	100
5-3	Picture of finished hermetically sealed humidity sensor. . . . .	102
5-4	Assembly drawing and BOM for the hermetically sealed humidity sensor design. . . . .	103
5-5	Drawing of the adapter used to connect the HVCR fitting to a high vacuum feedthrough. . . . .	103
5-6	EAGLE circuit board artwork and mechanical drawing. The traces (in red) are on the component side of the board. . . . .	104
5-7	DLP-232PC used for interfacing the Sensirion SHT75 humidity sensor to USB . . . . .	107
5-8	Pullup resistor installed between SHT75 VDD and DATA pins. . . . .	108

5-9	BOM for humidity sensor interface box and picture of finished assembly (cover removed). . . . .	110
5-10	Development tool for humidity data acquisition with alternate connec- tions to humidity sensor. . . . .	111
5-11	Top view of assembled instrumentation box . . . . .	114
5-12	End view of assembled instrumentation box . . . . .	114
5-13	Instrument enclosure drawing. . . . .	117
5-14	Enclosure endcaps drawing. . . . .	118
5-15	Enclosure bracket drawing. Note that two parts are shown (with same outline, but different holes); two of each are required for one assembly.	119
5-16	Completed instrument installation. . . . .	121
5-17	Data acquisition front panel. . . . .	123
5-18	Data acquisition block diagram (single encasement shown). . . . .	124
5-19	Modified D2XX driver subVI example. . . . .	125
5-20	Screen capture from the FTDI programming utility, FT_PROG. . . .	127
6-1	Completed Moisturematic (front and rear views) . . . . .	139
6-2	Gas diffuser, modified with bonded-in stainless steel fitting. . . . .	142
6-3	Saturator assembly drawing. . . . .	143
6-4	Conceptual extended surface saturator . . . . .	145
6-5	Schematic of Moisturematic . . . . .	146
6-6	Moisturematic control panel . . . . .	150
6-7	Moisturematic electronics. . . . .	151
6-8	Error contributions from various sources. . . . .	156
A-1	VCR fittings with marks indicating tightening 1/8 of a turn beyond initial seal contact. . . . .	163
A-2	The first assembly step is to install the valves and brackets, but the screws should not be tightened at this time. . . . .	164
A-3	Plumbing and instrumentation in the center popup. . . . .	165
A-4	Test plug for backside plumbing . . . . .	165

A-5 Encasement upside down on the work bench. The large hose is for helium leak testing the assembled manifold. . . . .	166
A-6 The helium dispensing probe with needle and valve. . . . .	167
A-7 A scratch in a sealing surface that required polishing to seal. . . . .	168
A-8 Platen post washer centering tool. . . . .	168
A-9 Argon deflectors and platen posts are installed. . . . .	169
A-10 The glass is lowered onto the clamp plate, after the o-rings are installed in the clamp plate. . . . .	170
A-11 Cleaning glass just before final assembly. . . . .	171
A-12 The platen clips are adjusted. . . . .	172
A-13 Installation of document clips onto the Bill of Rights. . . . .	173
A-14 The platen is installed in the tub. . . . .	174
A-15 The glass and clamp assembly is installed on the tub. . . . .	174
A-16 Tee fitting for evacuating sensing grooves simultaneously, used for fast reduction of helium background levels. . . . .	179



# List of Tables

2.1	O-ring parameters for “floating glass” design. Red values are inputs, and black values are outputs. . . . .	42
2.2	Geometry factors as a function of o-ring squeeze. . . . .	45
2.3	Leak detection/electrical analogy. . . . .	49
2.4	Predicted time until O <sub>2</sub> concentration inside encasement reaches limit of 0.5 % for various encasement sizes using two Viton seals. . . . .	54
2.5	Massachusetts Archives encasement leak testing results. . . . .	57
5.1	Connections between DLP-232PC and Sensirion SHT75. . . . .	107
5.2	BOM for soldered components (1st PCB operation) . . . . .	115
5.3	BOM for bolt-on parts (2nd PCB operation) . . . . .	116
5.4	BOM for instrument enclosure . . . . .	117
5.5	BOM for instrument installation supplies. . . . .	120
5.6	Network configuration for host and UBoxes. . . . .	126
6.1	Parameters suitable for generating humidified gas with a relative humidity of 40 %. . . . .	135
6.2	Commercially available humidity generators . . . . .	136
6.3	Moisturematic electronics BOM. . . . .	151
6.4	Effect of estimated errors on humidity of output gas. . . . .	155
A.1	Fastener torque specifications. . . . .	161
A.2	Rotameter correlation data for argon and helium, using approximate correction factors. . . . .	175



# List of Symbols

## Chapter 2

$A$	area of permeable membrane [cm <sup>2</sup> ]
$D$	thickness of permeable membrane [cm]
$G$	geometry factor
$k$	seal permeability [cc/sec – cm]
$K$	seal permeation [cc/sec – cm]
$k_{PV}$	pressure-volume product [atm – cc]
$L$	seal length [cm]
$l, w, h$	encasement dimensions [cm]
$m_{He}, m_{O_2}$	molecular mass of helium, oxygen [u]
$P$	pressure [atm]
$P_{He}, P_{O_2}$	partial pressure of helium, oxygen [atm]
$P_{in}$	partial pressure of oxygen inside sealed volume [atm]
$P_{in,max}$	maximum O <sub>2</sub> concentration in encasement (0.005 [atm])
$P_{out}$	partial pressure of oxygen outside sealed volume [atm]
$Q$	leak rate [atm – cc/sec]

$Q_{He}$	physical leak rate of helium [atm – cc/sec]
$Q_{O_2}$	total oxygen leak rate [atm – cc/sec]
$Q_{O_2,perm}$	permeation leak rate of oxygen [atm – cc/sec]
$Q_{O_2,phys}$	physical leak rate of oxygen [atm – cc/sec]
$R$	seal “resistance” [sec/cc]
$R_{eq}$	seal network equivalent resistance[sec/cc]
$R_{phys}$	seal physical leak resistance component [sec/cc]
$R_{perm}$	seal permeation resistance component [sec/cc]
$S$	seal squeeze [%]
$SR$	sachet rating (RP System®) [cc]
$t$	time [s]
$t_{sat}$	time until oxygen scavenger saturates [s]
$T_{STP}$	temperature of STP (often 0 °C) [K]
$T_{ambient}$	ambient temperature [K]
$V$	volume [cc]

## Chapter 6

$e_w$	vapor pressure of water [kPa]
$f$	enhancement factor
$m_w$	mass of water used in one purge cycle [g]
$P$	pressure[kPa]
$P_c$	encasement absolute pressure[kPa]

$P_s$	saturator absolute pressure[kPa]
$P_{O_2}$	partial pressure of oxygen in encasement [kPa]
$RH$	relative humidity [%]
$\rho_v(T_s)$	density of saturated water vapor [ $g/L$ ]
$t$	length of purge [min]
$T$	temperature [C]
$T_c$	encasement temperature [C]
$T_s$	saturator temperature [C]
$V$	encasement volume [L]
$V_t$	total volume of dry gas dispensed [L]
$\dot{V}_d$	flow rate of dry gas [L/min]
$\dot{V}_w$	flow rate of wet gas [L/min]



# Chapter 1

## Introduction

Long term display and preservation of historic documents is a difficult engineering challenge. The encasements that hold and protect documents must balance the preservation demands of conservators, the aesthetic requirements of curators, and the technical constraints imposed by physical phenomena such as diffusion of oxygen.

In 2007, The Massachusetts Archives sponsored research at MIT to design five encasements for the original Massachusetts Constitution (1780), a Goddard broadside of the Declaration of Independence (1777), an original copy of the Bill of Rights (1789), the 1629 Charter of Massachusetts Bay, and the 1692 Charter of the Province of Massachusetts Bay. This thesis documents the design, fabrication, and implementation of these encasements, which are presently being used to display the documents in a new permanent exhibit at the Commonwealth Museum (See Figure 1-1). The encasements I designed for this project successfully came in below budget while meeting and even exceeding the requirements for conservation and display of such historically significant documents.

As shown in Figure 1-2, the five documents are in remarkably good condition for their age. In recent years, they have been stored in a temperature- and humidity-controlled vault, with very little exposure to light. The MIT-designed encasements will continue to protect the documents while allowing them to be displayed by mimicking the conditions that have preserved the documents in the vault. The principle project requirements were to:



Figure 1-1: Photographs of the Treasures Gallery at the Massachusetts Archives showing the 5 cases designed, manufactured, and assembled for this thesis. *Photographs courtesy of the Massachusetts Archives.*

- Preserve specified environmental conditions. The target leak rates were to allow no more than 0.5% oxygen content over 20 years without refilling the encasement. Furthermore the environment was to provide 40% relative humidity (specified at 70°F). Conservators know that a combination of oxygen and light is responsible for fading of inks on historic documents [1, 2]. Removing the oxygen from the atmosphere surrounding the document eliminates photo-oxidation. Light can also be harmful in other ways, and generally steps are taken to reduce the document's exposure to ultraviolet light. Additionally, documents are believed to be damaged by rapid or extreme changes in temperature or humidity. Anorexic environments have the additional benefit of stopping growth of most fungi and aerobic bacteria, and will exterminate most insects. Anaerobic bacteria growth is reduced to near zero at relative humidities in the 30 – 40% range, which is considered to be near optimal for the long term storage of parchment or paper artifacts.
- Offer an aesthetic and viewer-friendly museum experience. All instrumentation related to the encasements must fit on the underside of the encasement and not be visible, as shown in Figure 1-1.
- Provide a low cost method of protecting documents: The Massachusetts Archives

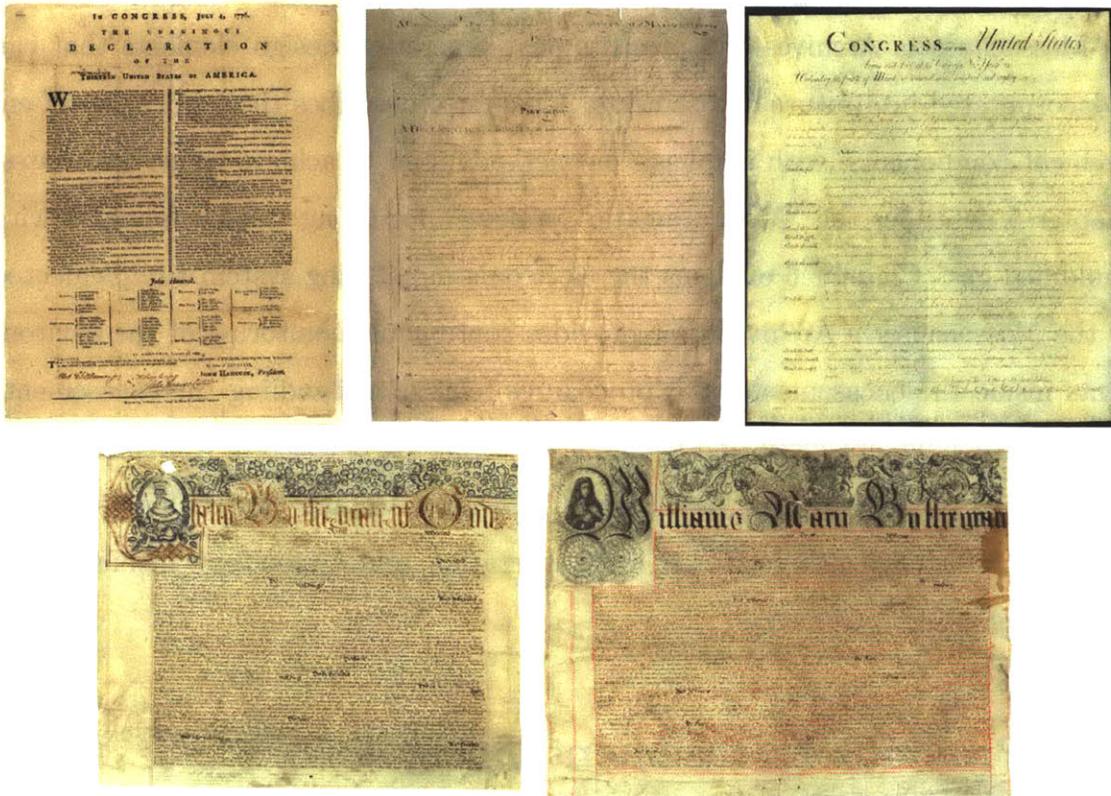


Figure 1-2: Photographs of a Goddard broadside of the Declaration of Independence (1777), the original Massachusetts Constitution (1780), an original copy of the Bill of Rights (1789), the 1629 Charter of Massachusetts Bay, and the 1692 Charter of the Province of Massachusetts Bay, showing their excellent condition. *Photographs courtesy of the Massachusetts Archives.*

allocated approximately 300,000 USD for design, manufacturing, and assembly for the five encasements. To accommodate the documents, two different size encasements were prepared. The previous major encasement project that required similar environment control had a 5 million USD budget for nine encasements.

This thesis details the challenges in designing low cost hermetic encasements and the solutions I developed to provide state-of-the-art atmospheric control. Chapter 1 summarizes previous approaches and explains the benefits of the approach and innovations I developed for the Massachusetts Archives project. Chapter 2 provides a scientific analysis of the sealing technologies, including a formalism for diffusion and permeation. This chapter also describes verification of the seals through he-

lium leak detection. Chapter 3 details the mechanical design of the encasements for the Massachusetts Archives and presents next-generation design elements that can be incorporated into future encasements. Chapter 4 describes the fabrication of encasement components, with emphasis on methods used to achieve target leak rates. Chapter 5 describes the “Moisturematic”, a device for filling the encasements with humidified gas. Chapter 6 explains the electronic monitoring system I implemented for the Massachusetts Archives that provides continuous monitoring of the environments inside the encasements. All procedures related to handling the encasements including cleaning, assembly, and helium leak testing are detailed in Appendix A and accompanied by photographs, providing an “Owner’s Manual” for the encasements. Appendix B includes code related to instrumentation, and Appendix C includes a list of suppliers.

## **1.1 Previous Encasement Designs**

This section surveys previous encasement techniques and designs over the past two decades. In almost all cases, the encasement takes the form of a sealed box that has a glass or plastic front, and is filled with a humidified inert gas with the goal of achieving a carefully controlled environment that can balance the needs of conservation with the wishes of the public to see these historic documents and artifacts.

### **1.1.1 The Magna Carta**

An encasement for the only copy of the Magna Carta in the United States was designed by Donald Etherington and Nathan Stolow. A similar encasement was designed for the Gettysburg Address. Both systems used silica gel to buffer relative humidity levels, a topic which Stolow has written about extensively [3], and which is the subject of a heated debate shown on PBS about the Charters of Freedom encasement project [4]. The current conservation trend is to use only the document and backing papers for buffering humidity [5].

A picture of the Magna Carta encasement is shown in Figure 1-3. Several design

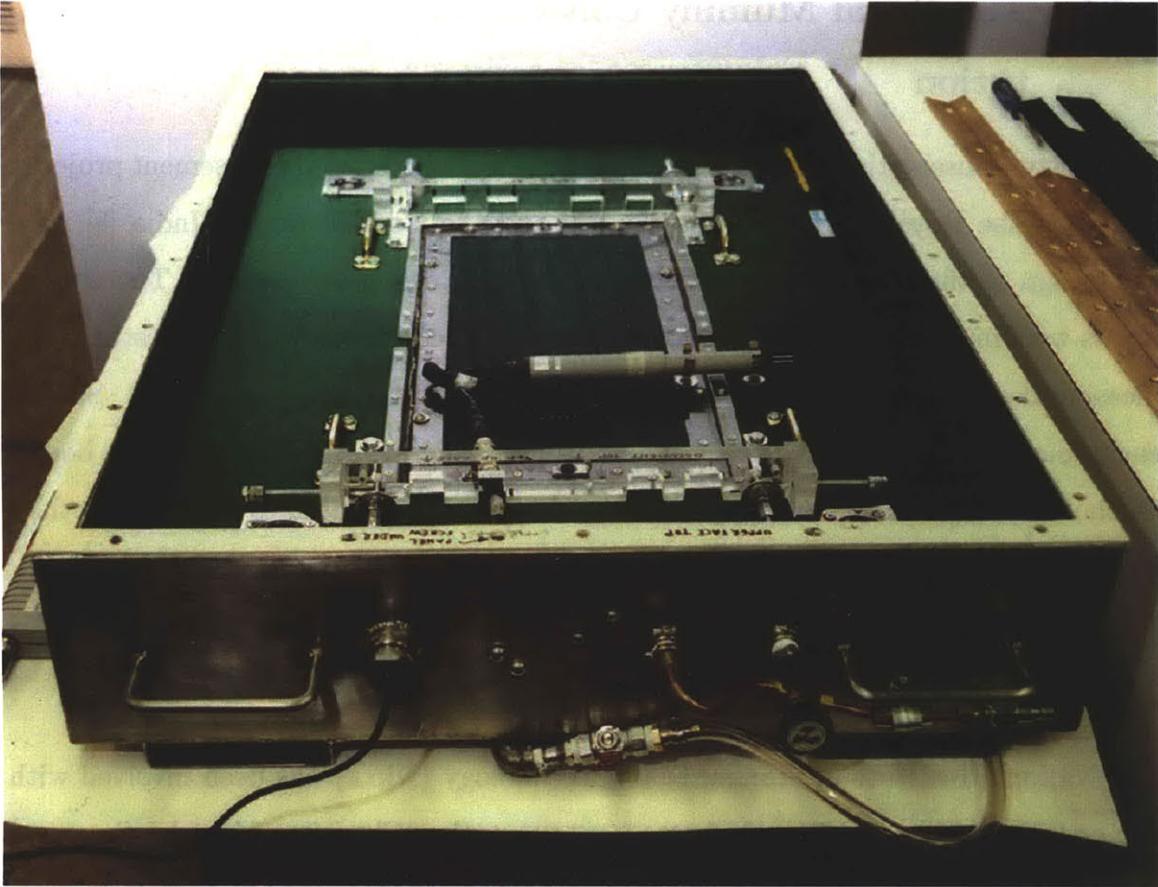


Figure 1-3: Photograph of the current encasement for the Magna Carta, completed in the 1980s.

flaws are apparent, including too few bolts spaced too far apart and a flat gasket, which results in low and non-uniform contact pressures. The mechanical drawing [6] references an acrylic front, which oxygen permeates readily. The NPT and compression fittings are inappropriate for what is essentially a high vacuum system—a single NPT fitting can consume the entire oxygen leak budget for a 20 year encasement. These shortcomings translate to an encasement that would require regular purging to maintain acceptable levels of oxygen. Despite these shortcomings, this work established the precedent for applying new engineering concepts to historical preservation.

### **1.1.2 The Royal Mummy Collection and the Indian Constitution**

The Getty Conservation Institute has been involved with several encasement projects, including the Royal Mummy Collection [7] and the Constitution of India [8]. For the design of the mummy encasements, minimizing cost was the goal. The encasements were made from aluminum extrusion with bolted corner joints, an aesthetically pleasing design that allows viewing from five sides, but is difficult to seal. Similar encasements were designed for the Indian Constitution, which consists of two books. Of the two encasements, one had an oxygen leak rate over 50 times that of the other, illustrating just how sensitive sealing is to variations in technique.

### **1.1.3 The Charters of Freedom**

The National Institute of Standards and Technology (NIST) has been involved with two ambitious encasement projects in recent years, the “Charters of Freedom” project and the design of an encasement for the Waldseemüller map. The “Charters of Freedom” project replaced failing encasements from the 1950s, which were also designed by NIST (then the National Bureau of Standards). The re-encasement project, finished in 2003, took over 4 years and cost nearly 5 million dollars. While the project set a new standard for encasements, the design had some shortcomings. In particular, the high cost makes the NIST approach out of reach of most museums and historical societies. The NIST design used a custom-designed tin-plated Inconel<sup>®</sup> seal. Metal seals accomplish a tight seal by deforming the surface of the seal gland. As a result, the gland must be polished if it is to be resealed after opening. The number of times a gland can be polished is limited, and the seal itself is not reusable.

Unfortunately, long term leak data is unavailable for these encasements. The NIST design precluded a helium leak test beyond the initial test for acceptance, and the oxygen content of the encasements has never been measured. The initial helium leak test did indeed exceed expectations—the seal was described as a “1000 year seal.” However, as described in Chapter 2, helium leak testing must be performed between

the inner and outer seals of an encasement. The outer seal in the NIST design was simply an indium wire, which due to creep and corrosion can only seal for a short time. An engineer involved with the project noted that if they had the opportunity to go back in time, they would have used two seals [9].

In summary, while the phenomenally low initial leak rate measured by NIST makes this design the gold standard for encasements, the high cost of producing such an encasement and the inability to easily open and reseal the encasement made this design unsuitable for the Massachusetts Archives.

#### **1.1.4 The Waldseemüller map**

At the same time as I was working on the design for the Massachusetts Archives project, NIST designed and built an encasement for the Waldseemüller map (1507) (See Figure 1-4). Often called “America’s birth certificate”, this document is currently on display at the Library of Congress. In a departure from the “Charters of Freedom” project, NIST chose not to use metal seals. This choice was in part dictated by the large size of the map (the encasement itself measures approximately 5 by 8 feet) due to differences in thermal expansion between the glass and the encasement. The large size also means that barometric pressure changes put enormous stress on the glass viewing window as well as the encasement itself. To compensate for barometric pressure changes, the back of this encasement was designed to flex, reducing the pressure differential.

There are two minor shortcomings of the Waldseemüller map encasement design. First, it has no positive stop on the plate that clamps the glass to the seals, a design that allows adjustment of seal crush to obtain a tighter seal, but resulted in at least one piece of broken glass (thankfully not with the document inside!). Second, the seal architecture made it difficult to surround the outside seal with helium, making leak testing especially difficult, given the slow response time due to the large encasement and the difficulty in differentiating permeation and physical leaks over long time scales.

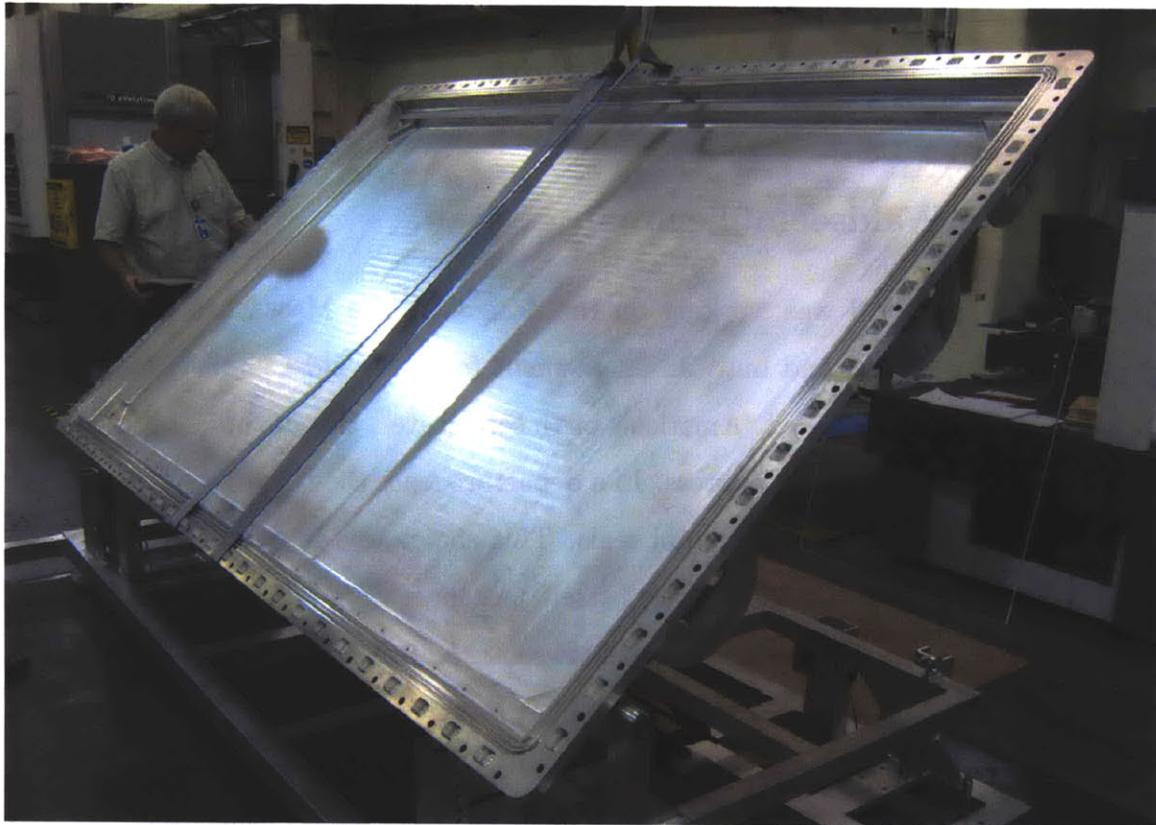


Figure 1-4: Base of the Waldseemüller map encasement in the NIST shop. This encasement was under construction concurrently with The Massachusetts Archives encasements.

## 1.2 The Massachusetts Archives Project: Advancing Science and Historical Preservation Techniques

When designing the encasements for The Massachusetts Archives, I studied these previous encasement examples and learned from their shortcomings. I made sure that design decisions were motivated by first principles, best practices, and understanding of phenomena such as oxygen permeation and limitations of leak checking techniques. The justifications for various design decisions are detailed in the subsequent chapters.

Ph.D. theses often require the doctoral student to develop a proof of concept, which might require further refinement by industry or academia. Instead, this thesis project presented both design-for-robustness and systems integration challenges. Hardware had to be delivered before the museum opened for business, and the hardware must remain functional for decades to come. I was responsible for overseeing the mechanical design, manufacturing, on-site installation, and testing of five encasements. Documents were loaded into the encasements in January 2009 and have been on display in the Treasures Gallery at the Massachusetts Archives since the museum officially reopened in April 2009. In addition to staying under budget, we successfully achieved acceptance criteria. The encasements were filled with humidified argon gas, and the initial leak rates on all five encasements indicate that the preservation environment can be sustained for at least 20 years. Instrumentation shows pressures and humidity levels are holding steady. Furthermore, commercially available Viton<sup>®</sup> o-rings were used, so that in the future the encasements can be easily serviced.

However, in addition to achieving the established technical specifications to preserve an important part of American history, this project enabled us to make several scientific and engineering advancements. I developed:

- 1) A framework for understanding permeation of gases through polymer seals. This formalism enables one to account for crush or squeeze that a polymer seal experiences. This framework, presented in Chapter 2, can be applied to a variety of

systems and gases mixtures other than oxygen permeation in encasements for historical documents.

2) A novel device (the Moisturematic) for filling enclosed spaces with precisely humidified gas. Safety features are incorporated to prevent over-pressure situations. These safety features are especially critical when a historic document is in the enclosed space.

3) Tools and techniques that advance the state of the art in encasement design. These improvements include floating the glass between seals, leak testing that can be performed at any point in the future to assure that leak rates remain within specifications, and specialized fabrication techniques that enable quality components to be produced at low cost.

I hope that this thesis will serve two distinct purposes. On one hand, it can serve as a manual to museum and archive staff seeking to better understand the engineering challenges associate with conservation and that clever mechanical solutions can enable high quality preservation environments at more accessible costs than with other designs. Second, this thesis can provide mechanical engineers with a deeper understanding of sealing and humidification that can be applied to other challenges such as dry laboratory design and toxic waste containment.

## Chapter 2

# Sealing Technologies

All seals leak. There are many ways to detect a leak. Perhaps oil can be seen dripping, or a hissing sound can be heard. Smaller leaks are more difficult to detect. Maybe a bike tire goes flat overnight—likely, the leak could be found by dunking the tire in water or “snooping” with soapy water.

In many dynamic applications, some amount of leakage is required to prevent failure of the seal. If you’ve ever noticed a streak of oil on the underside of an older car’s hood, this may very well be from the shaft seal of an A/C compressor simply doing its job. Small leaks are hard to detect. In such a case, a halogen leak detector could be used to quantify the amount of refrigerant escaping, and then a determination can be made of whether repair is required.

Encasement work requires the ability to detect even smaller leaks, a job that requires specialized equipment such as a helium leak detector. Of course, for an encasement to function properly, seals that can maintain these low leak rates are required.

This chapter will begin with an introduction to helium leak detection, as well as seal material and geometry and their characteristics. A mathematical framework for analyzing a seal network of arbitrary complexity will be developed, with particular application to elastomer seals. Finally, conservation applications of chemical oxygen scavengers will be discussed.

## 2.1 Helium leak detection

Helium leak detectors use a mass spectrometer to detect minuscule amounts of helium. Helium is used because the molecule is very small, is not abundant in air, and is difficult to pump; these properties help make helium leak detection very sensitive to small leaks. A typical modern helium leak detector might measure leaks from  $10^{-4}$  to  $10^{-11}$  atm – cc/sec. To get an idea of the needed range for encasement work, let's suppose an encasement with a 40 L volume contains oxygen at a partial pressure of 0.005 atm. If it takes 20 years to reach this point, the oxygen leak rate

$$\begin{aligned} Q_{O_2} &= P_{in,max} \cdot V/t \\ &= 0.005 \text{ atm} \cdot 40000 \text{ cc} / 6.3 \times 10^8 \text{ s} \\ &= 3.2 \times 10^{-7} \text{ atm} - \text{cc/sec.} \end{aligned}$$

This corresponds to an allowable helium leak rate of about  $4.3 \times 10^{-6}$  atm – cc/sec, which can be computed from Equation 2.1. This value falls nicely into the usable range of the instrument, with several decades of sensitivity to spare.

The ability to test a system for leaks with such an instrument often requires the designer to plan in advance. Helium leak detectors require high vacuum to function. This means that the system being tested must be able to tolerate high vacuum on one side of the device under test, while there must also be a provision to introduce helium to the opposite side of the device under test. The device under test might be a seal, valve, fitting, weld, electrical feedthrough, bellows, etc. An encasement, however, cannot be evacuated, as the vacuum would damage a document. Even if this were not a consideration, a typical encasement is too large and too lightly constructed to withstand an atmosphere of differential pressure. Instead, vacuum must be applied to a volume created between two seals, as shown in Figure 2-1. Helium can then be introduced from outside and inside, allowing the integrity of both seals to be determined. This configuration is the simplest arrangement that allows an

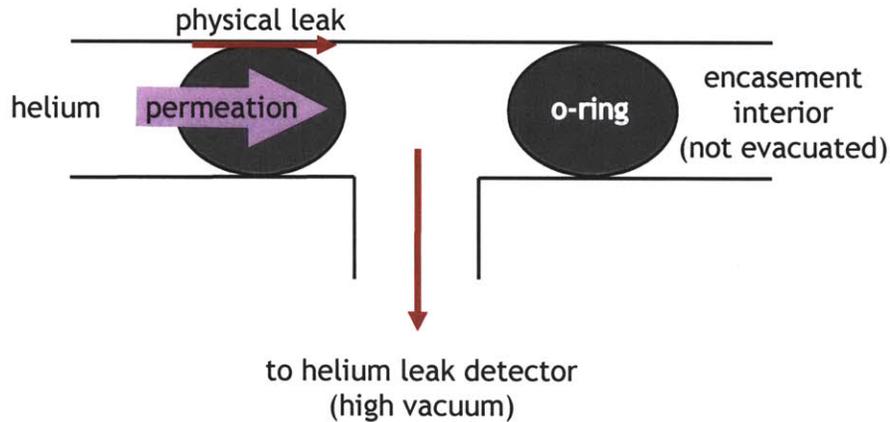


Figure 2-1: Schematic of helium leak detection for an encasement.

encasement to be helium leak checked; other design constraints may dictate a more complex arrangement. Section 2.5 presents a methodology for analyzing networks of seals. We must begin our analysis, however, with a discussion of seal materials and their properties and develop an understanding of leakage through a single seal.

### 2.1.1 Physical leaks vs. permeation

A helium leak detector cannot differentiate between helium that diffused through a seal (permeation) and helium that bypassed a seal (physical leak). Physical leaks are caused by mechanical defects or contamination, such as a scratch in a sealing surface, a defective weld, or lint caught under a seal. Permeation is an unavoidable consequence of using polymer seals. The reason these two must be distinguished is that engineers are rarely interested in how fast helium will leak into or out of a system, but instead are interested in the leak rate of other gases.

Physical leaks can be measured with helium as the tracer gas; the leak rate can then be corrected for other gases. Leaks of less than  $10^{-5}$  atm – cc/sec are often assumed to be in the molecular flow regime [10], and from Chapter 1, we know that leaks in an encasement must be substantially smaller to meet the required specifications. In the molecular flow regime, the molecular mass of each species factors in to calculating an estimated leak rate for gases other than helium. In this regime, leak rates are modeled to be a linear function of differential pressure across the leak. Therefore, the

partial pressures of helium used for testing (often 1 atm) and the partial pressure of the other gas must also be considered—air has an oxygen partial pressure of 0.21 atm. Thus, for converting a physical helium leak rate to a physical oxygen leak rate,

$$Q_{O_2,phys} = Q_{He} \cdot \sqrt{\frac{m_{He}}{m_{O_2}}} \cdot \frac{P_{O_2}}{P_{He}}. \quad (2.1)$$

Permeation is more complicated; there is no simple correlation between helium and oxygen permeation. Instead, the permeability  $k$  of various polymers and elastomers must be measured or found in the literature [11, 12, 13, 14, 15]. Further complicating matters, permeability tends to vary from batch to batch and is a function of temperature, pressure, gas composition, and geometry, and data is often missing for certain material/gas combinations.

Oxygen permeability is typically determined by measuring the oxygen flow rate  $Q_{O_2,perm}$  through a membrane of area  $A$  and thickness  $D$ . The value of  $k$  can then be found using

$$Q_{O_2,perm} = k \frac{A}{D} P_{O_2}. \quad (2.2)$$

A more detailed analysis of permeation through elastomer o-rings follows in Section 2.4.1.

Permeation impacts both testing technique and interpretation of results. It is the job of the helium leak detector operator to differentiate between physical leaks and permeation. The only practical tool available is the time response of the helium leak signal. While physical leaks are expected to be detected almost immediately after application of helium, permeation may take several minutes to develop, as shown in Figure 2-2. This figure shows that permeation takes several minutes to become detectable, allowing the operator to differentiate between physical leakage and permeation.

Helium leak testing results of the Massachusetts Archives encasements are discussed at the end of this chapter. The procedure developed for helium leak testing is described in Appendix A.4.

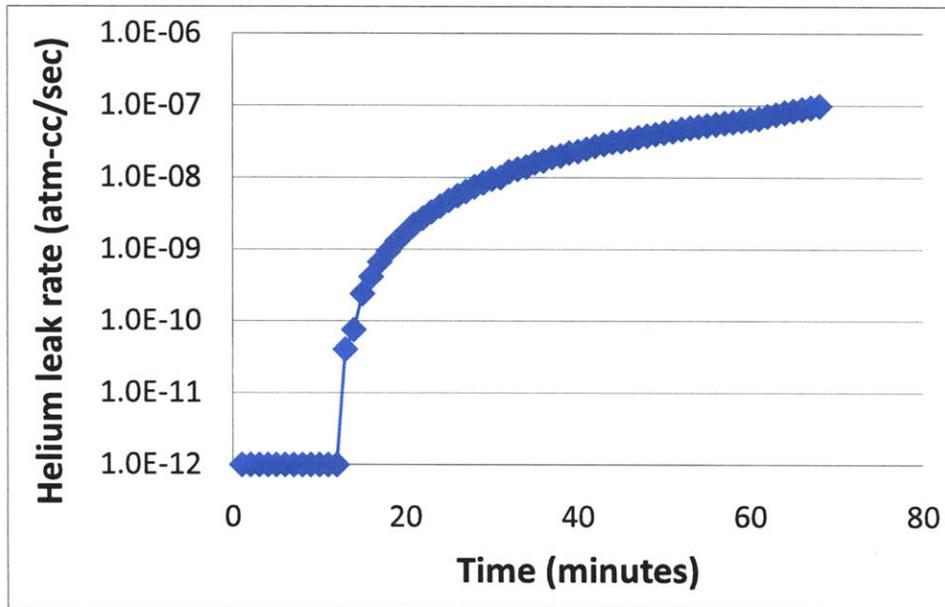


Figure 2-2: Time response of helium permeation through a -334 (2.6" ID × 0.210" CS) Viton<sup>®</sup> A o-ring using 35 % Helium/63 % Argon/2 % CO<sub>2</sub> tracer gas. Values of  $1 \times 10^{-12}$  atm – cc/sec indicate the lower detection limit of the Varian VS MD30 helium leak detector. Helium was not detected until 12 minutes after the flow of tracer gas was started.

## 2.2 Metal seals

Metal seals, at first glance, appear to be the ultimate sealing solution. These seals are typically constructed such that a spring energizes a softer material that forms the actual seal. They can withstand high temperatures, and they are expected to have very low leak rates for both physical leakage and permeation. The metal spring is expected to maintain contact between the seal and seal gland nearly indefinitely, unlike with elastomers that can creep. The spring may be a coil, or may be the seal itself, as shown in Figure 2-3. They are often used to seal the demanding aerospace and power generation applications.

These seals, however, have some shortcomings. Prominently, they are rarely used on aluminum components because the soft surface would be damaged by the seal. While this may be acceptable for a single use, many times it is desirable to reuse components without rework. Even with harder gland materials, these seals cannot be considered demountable. [16] shows that the leak rate of an all-metal seal increased

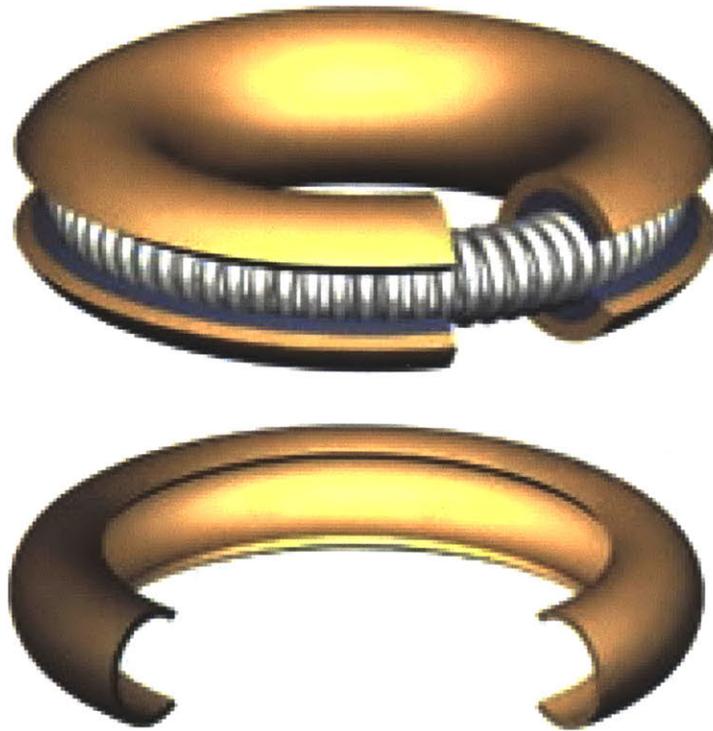


Figure 2-3: Helicoflex<sup>®</sup> and C-Flex<sup>™</sup> metal seals. *Garlock Helicoflex<sup>®</sup>*

by several orders of magnitude after a single disassembly and reassembly operation.

The Charters of Freedom project attempted to overcome this shortcoming by electroless nickel plating the seal glands. This provides a much harder surface than the aluminum, while also providing a more corrosion resistant sealing surface. However, the plating would require polishing if the cases were ever disassembled [17]. This plating operation also added significantly to the cost of the cases.

Sealing to glass also presents some difficulty for metal seals. First, metal seals are intolerant of variations in installed height. Since glass tolerance tend to be fairly wide ( $\pm 0.5$  mm for this project), the glass installation would have to be shimmed to establish the correct seal geometry. Second, differential thermal expansion between the glass and an aluminum tub generates high shear stresses in the seal. The stresses can be reduced by using different materials for the encasement; for example an encasement made of stainless steel or titanium would better match the thermal expansion of the glass. The processing, of course, of these materials is much more complicated

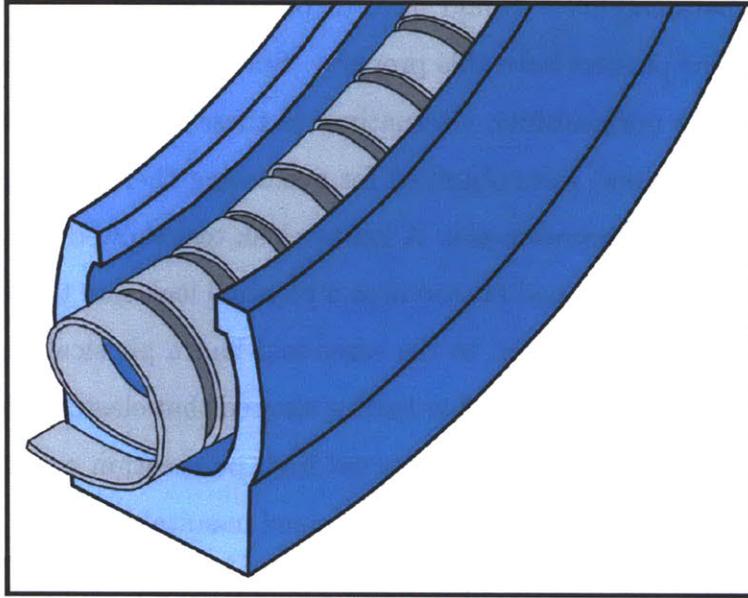


Figure 2-4: An energized plastic “OmniSeal®.” *St. Gobain Performance Plastics*

and expensive. As a result, metal seals were seen as too high cost and too high risk for the Massachusetts Archives project.

## 2.3 Energized plastic seals

Spring energized plastic seals blend some of the best features of all-metal seals and their elastomeric predecessors. Their combination of properties solve many difficult sealing applications. This type of seal was initially considered a front-runner when investigating seals for encasements. An energized plastic seal is depicted in Figure 2-4.

Because they have use a metal spring to maintain contact between the seal and the gland and jackets made of highly inert plastics, they can be expected to maintain their integrity nearly indefinitely. As a sales representative said, “They’ll be around when the sun goes nova.”

However, relative to an o-ring, these seals have a large area and small depth, making permeation a concern. Furthermore, the typical spring design does little to act as a barrier to permeation. To compete with an elastomeric o-ring in terms of

permeation, the permeability  $k$  would have to be substantially lower than for elastomers, and very few plastics have this property. Since the manufacturer was unable to provide a value for permeability, alternatives to these seals had to be considered.

Samples (in small sizes) were obtained for evaluating their performance with regards to use in document encasements. A helium leak test showed that a sample seal that had been installed twice had far too high a physical leak rate to meet the project specifications. An o-ring subjected to the same test had a physical leak rate several orders of magnitude smaller, and further testing showed that elastomeric o-rings could be successfully reused. Between the suspected high permeation and confirmed high physical leakage, energized plastic seals were deemed unsuitable for the Massachusetts Archives encasements.

## 2.4 Elastomer seals

In light of the above deficiencies, the venerable o-ring is hard to beat. After carefully considering the alternatives, o-rings were selected over other types of seals for the Massachusetts Archives encasements. O-rings are not without their own issues, however. The following sections detail the design considerations that must go into an o-ring application where leakage is to be minimized.

### 2.4.1 Designing for elastomer seals

**Materials** Selecting a material for a new sealing application is a balance between several parameters, including chemical compatibility, temperature resistance, out-gassing, permeation, and compression set. The ideal seal would let no oxygen through and return to its original shape after being compressed for a long period of time. It must not cause corrosion in the seal gland, which would cause the seal to eventually leak. To gain acceptance with conservators, the seal material must not hurt the document in any way. It must not fail due to chemical degradation over the course of several decades.

Of the dozens of base materials and even larger selection of compounds available,

fluorocarbon (trade name Viton<sup>®</sup>) provides the appropriate combination of parameters, with the largest emphasis applied to compression set and permeation. Values of these parameters for common elastomers can be found in the literature, such as the ERIKS O-ring Technical Handbook and the Parker O-ring Handbook [11, 12].

Viton<sup>®</sup> finds much use in high vacuum applications. In recent years, however, fluorocarbon materials have been compounded to provide greater resistance to motor vehicle fuels. These newer materials do not offer the same low permeation of the original Viton<sup>®</sup> A and should be avoided for vacuum applications.

## Dimensioning

Squeeze is easily the most critical parameter in designing for a sealing application. Squeeze is defined as the percent change in height of an o-ring from its free state to a compressed state; typical values range from 10 % to 30 %. Lower values are inadequate from a permeation perspective, while higher values result in significantly higher forces, which may require heavier construction. Above 30 % squeeze, the o-ring may be damaged, though smaller cross sections are more tolerant of heavy squeeze [12]. The cross sectional diameter of the o-ring plays a role in squeeze. Smaller cross sections are more tolerant of squeeze. However, the desire to use a small o-ring must be balanced with other design parameters, such as the available glass tolerance. When small diameters are combined with variations in component geometry, unacceptable squeeze values may result.

When ordering an o-ring, the length must also be specified. O-rings are typically sold by inside diameter, even if the o-ring won't be installed in a round gland. In this case, the inside circumference can be measured and an equivalent ID calculated. The o-ring should be ordered to be 1 – 2 % smaller than the actual ID needed; this quantity is known as “stretch”. An o-ring longer than the groove it must fit in can be difficult to seat during assembly, and an o-ring with too high a stretch tends to climb out of its groove and is more likely to fail in service [12].

The gland dimensions must also be chosen carefully. While gland depth is directly calculated from squeeze, width has no strict specification. While too wide a gland

Table 2.1: O-ring parameters for “floating glass” design. Red values are inputs, and black values are outputs.

	nominal	actual	unit
seal diameter	0.210	0.210	in
nominal squeeze	22.0	-	%
nominal seal thickness	0.164	-	in
nominal glass thickness	13.52	13.25	mm
glass tolerance (thick)	0.50	0.10	mm
glass tolerance (thin)	-0.50	-0.10	mm
minimum gap (glass to aluminum, per side)	0.005	-	in
glass pocket depth	0.562	0.562	in
gland width	0.255	0.255	in
gland depth	0.149	0.149	in
lower bound squeeze	17.3	18.5	%
upper bound squeeze	26.7	20.4	%
upper bound fill	88.2	81.2	%
lower bound fill	78.2	79.4	%

could result in movement of the o-ring in a high pressure application, an atmosphere of pressure will generally not provide enough force to overcome friction. Too narrow a gland, however, is a problem. Elastomers can be considered incompressible, so the gland must be large enough to accept the entire o-ring with room to spare. Typically, an o-ring gland should not be filled more than 90%. The gland width was selected in the example below to ensure that the percent fill did not exceed this value.

To simultaneously evaluate multiple o-ring parameters, it is useful to make a spreadsheet, as shown in Table 2.1. This spreadsheet is specific to a “floating” glass design, where the glass is sandwiched between two seals, as shown in Figure 2-5. This means that only half of the glass tolerance must be accommodated by each seal, resulting in less variation in squeeze. This spreadsheet has two columns of values—one is to calculate nominal values based on specifications, while the second column is to evaluate the effects of the actual parts that were measured upon arrival. Most notably, the glass was thinner than nominal, but was within specification. As a result, squeeze was reduced from nominal. If glass thickness had been accurately known while designing the rest of the encasement, the squeeze would have been increased.

Another thing to consider is the shape of the gland. A rectangular gland is the

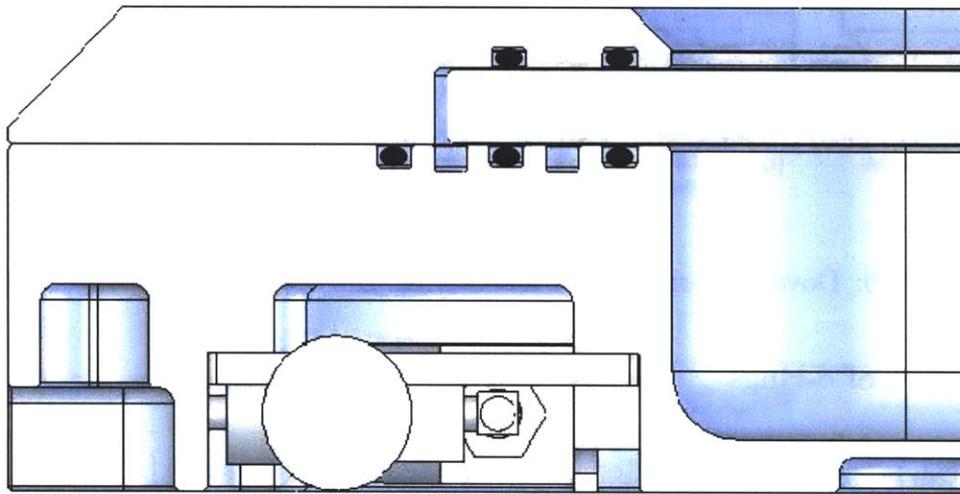


Figure 2-5: Cross section of encasement with floating glass.

most common and is easy to machine and finish polish; the Massachusetts Archives used rectangular glands. A rectangular gland, however, does little to retain the o-ring during installation, which turned out to be a minor nuisance during assembly of the Massachusetts Archives encasements. Dovetail glands overcome this assembly problem, and on a project of this scale, the delta cost is insignificant. While the Massachusetts Archives encasements used rectangular glands, a dovetail or half-dovetail gland would almost certainly be specified in a second-generation design.

Special design considerations for specifying dovetail glands include careful dimensioning and tolerance analysis, because the dovetail design limits the void area, which should be kept above 10% while allowing for wide glass tolerances and maintaining an acceptable range of squeeze. A good machinist will be aware that a dovetail cutter cannot cut as fast as a straight cutter of the same diameter. The designer can help the machinist by designating a location for a “drop hole”, which allows the large end of the dovetail cutter to pass through the top surface of the machined part. This allows the use of a significantly larger and more rigid cutter, increasing material removal rate and reducing the number of passes required. A typical machining sequence for a dovetail gland with drop hole allowance is shown in Figure 2-6.

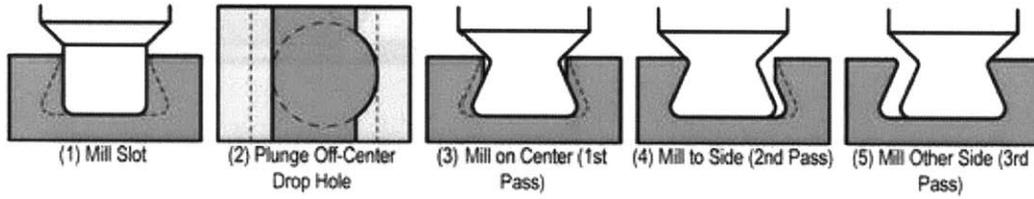


Figure 2-6: Dovetail o-ring gland machining. *Harvey Tool Company, LLC*

### Permeation of o-rings

The oxygen permeability  $k$  of Viton is often quoted in the literature [15, 18] to be  $1.1 \times 10^{-8}$  cc(STP)/sec – cm – atm. Permeability is most often determined by measuring leakage rates through elastomer membranes with an exposed area  $A$  and thickness  $D$ . The value of  $k$  can then be found using Equation 2.2.

However, the geometry of an o-ring is substantially different from a membrane. If the o-ring cross section is square with dimensions  $D \times D$  and the o-ring has length  $L$ , then

$$A/D = L, \quad (2.3)$$

assuming that  $L \gg D$ . These two equations can be combined, resulting in

$$Q_{O_2,perm} = k \cdot L \cdot P_{O_2}. \quad (2.4)$$

Most o-rings, however, do not have a square cross section. For the general case, we desire a relationship that looks like Equation 2.4, but is corrected for geometry and other factors. Such an equation might look like

$$Q_{O_2,perm} = K \cdot L \cdot P_{O_2}, \quad (2.5)$$

where  $K$  is the corrected permeability.

Now the challenge is to develop a relationship between  $K$  and  $k$ . As one might expect, the squeeze  $S$  plays an important role in the value of  $K$ . For the simple case of

Table 2.2: Geometry factors as a function of o-ring squeeze.

squeeze (%)	G
10	1.58
15	1.27
20	1.04
25	0.869
30	0.695
35	0.593

a square o-ring, developing this relationship is very easy. Compressing the o-ring both decreases the area exposed to gas, and increases the depth that gas must permeate through. Assume an incompressible o-ring of length  $L$  and with a  $D \times D$  square cross section. When the seal is compressed to a height  $D(1 - S)$ , the width must increase by a factor of  $1/(1 - S)$  to maintain the same cross sectional area. Thus, the ratio  $A/D$  can be calculated as

$$L(1 - S)^2. \tag{2.6}$$

Of course, most o-rings don't have a square cross section. To quantitatively evaluate the effects of crush on permeation through an o-ring of round cross section, a two-dimensional finite element analysis was performed using COMSOL. Various seal geometries were evaluated, using a partial pressure of 1 atm of oxygen on one side and vacuum on the other. The deformed shape of the o-ring was assumed to be an oval, as shown in Figure 2-7. The width of the flat portion was calculated to maintain the cross sectional area of the original uncompressed o-ring. The edges flattened by the sealing surface were treated as impermeable. It was assumed that permeation is a bulk property, *i.e.*, permeation is unaffected by the area or shape of the surface exposed to oxygen or vacuum, and this analysis does not account for possible interactions between gas species. The results of this study allow  $k$  and  $K$  to be correlated by some geometry factor  $G$ , the values of which are listed in Table 2.2.

The odd units of  $k$ , cc(STP), warrant some discussion. A cubic centimeter of gas, specified at a certain "standard temperature and pressure" (STP), is describing an amount of gas more correctly specified by an amount of substance. Similarly, leak

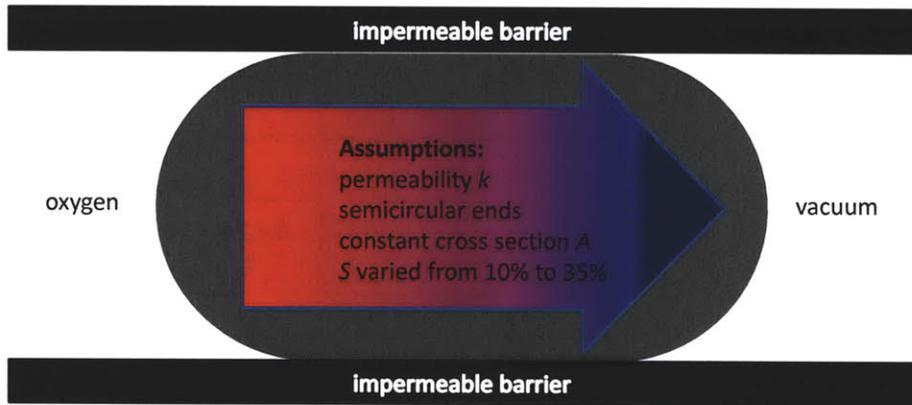


Figure 2-7: O-ring permeation study assumptions.

detection work is often carried out in atm-cc/sec, where an atm-cc implies a mass of gas occupying a cubic centimeter at atmospheric pressure and ambient temperature. STP usually means the gas volume is measured at 273.15 °K, though data will often be collected at ambient temperature. When using values of  $k$  from literature, one should be aware of the definition of STP in use and ensure that the measurements were performed at a temperature comparable to the intended application, since permeation is temperature dependent. Depending on the definition in use,  $K$  may have to be corrected for ambient temperature using the ideal gas law. The application of these scaling factors results in

$$K = G \frac{T_{ambient}}{T_{STP}} k. \quad (2.7)$$

From this equation and the values of  $G$  in Table 2.2, it can be seen that the values of  $k$  and  $K$  may not be drastically different; they are often treated as equal for estimating leak rates in vacuum work.

For the purposes of continuing this analysis in Section 2.5.1, it is recommended that values for  $k$  be measured or converted to cc(STP)/sec – cm – atm. Correcting for ambient temperature allows  $Q_{O_2,perm}$  to be measured in atm – cc/sec and  $K$  to be measured in cc/sec – cm. These measures are convenient for the analysis that follows in Section 2.5.1.

## 2.4.2 O-ring manufacturing

In the US, standard o-ring sizes are codified in Aerospace Standard 568, published by the Society of Automotive Engineers. The smallest standard o-ring (-001) has an ID of 1/32" and a cross sectional diameter of 1/32". The largest standard o-ring (-475) has an ID of 26" and a cross sectional diameter of 1/4". Even the largest standard o-ring is not large enough for many commercial applications.

Larger sizes are typically constructed by joining the ends of one or more pieces of stock. This stock may be extruded or molded, and the joint can be glued or molded. How the stock is manufactured and the joining process used appears to have a large impact on o-ring performance. Most stock for large o-rings is extruded. Physical properties, surface finish, and dimensional accuracy are typically worse than with molded materials.

The first set of o-rings ordered for the Massachusetts Archives project were extruded and centerless ground. Centerless grinding improves the dimensional accuracy and surface finish, as well as allows custom diameters to be easily fabricated. Four of the five encasements assembled with these o-rings had unacceptably high leak rates, even with careful preparation of the seal gland and o-rings. Upon inspection, many of these o-rings had defective joints. Figure 2-8 shows a split at an o-ring splice. Centerless grinding may also have contributed to high leak rates, since the grinding marks run tangentially, potentially creating leak paths around the o-ring.

On the leaking encasements, the o-rings were replaced. The second batch of o-rings was ordered from Greene, Tweed, & Co. These were constructed from large diameter standard molded o-rings, which were cut and then spliced, and were not centerless ground. Since these are made from o-rings smaller than the desired size, each o-ring had three splices. However, these splices were of much higher quality than the splices from the original batch and did not exhibit any visible defects. The manufacturer of these seals claims the joints, just like the stock, are molded, but process details were not disclosed.

It should be noted that molded components will likely have some visible flash,



Figure 2-8: A defective splice in an extruded and centerless-ground o-ring.

which could adversely affect sealing if the o-ring was oriented such that the flash contacts sealing surfaces. The flash on the Greene, Tweed o-rings appeared to have been removed by sanding or grinding, and the splices appeared to be sanded or ground all around. These finishing operations made the parting line difficult to see; though every effort was made to install the o-rings such that parting lines were oriented away from sealing surfaces. It may be possible to specify that the flash be left intact, though the parting lines would have to match between the multiple o-ring segments.

Despite the minor issue of the mold flash, the high quality of the Greene, Tweed o-rings combined with careful assembly work yielded impressive results. As demonstrated by the leak test data in Table 2.5, these replacement o-rings reduced physical leakage to orders of magnitude below permeation leakage.

## 2.5 Leakage analysis

This section provides the mathematical framework for analyzing a seal network of arbitrary complexity. To help build intuition, an analogy between seals and passive electrical components is developed. Approximations useful for early stage design are developed and discussed.

Table 2.3: Leak detection/electrical analogy.

leak detection		electrical	
component	variable	component	variable
seal “resistance”	$R$	resistance	$R$
volume	$V$	capacitance	$C$
leak rate	$Q$	current	$i$
pressure-volume product	$k_{PV}$	charge	$q$
pressure	$P$	voltage	$v$

### 2.5.1 Single seal, single volume analysis

It is convenient to draw an analogy between the seal system and an RC electrical network. The analog of a seal is a resistor, and the analog of a capacitor is a volume. Table 2.3 matches the variables used in leak detection work to those used in electrical work.

Boyle’s law states that the product of pressure and volume remains constant. This is analogous to the charge on a capacitor being equal to the product of capacitance and voltage:

$$k_{PV} = PV \iff q = Cv. \quad (2.8)$$

Of course, we must be careful in justifying the use of Boyle’s law instead of the ideal gas law, which includes the effect of temperature. This is an easy argument to make; large variations in temperature can damage a historic document. Encasing, leak detection, and display will likely be carried out between 15 °C and 25 °C. It is suggested that all calculations be carried out with leak rates having units of atm – cc/sec, which implies volume measurements at ambient temperature. The Varian VS series leak detector used in this work reports leaks in atm – cc/sec by default, and automatically performs temperature compensation to ambient temperature.

We will define the leak rate  $Q$  to be the time rate of change of the product  $PV$ :

$$Q = \frac{d}{dt}(PV). \quad (2.9)$$

Since  $V$  is a constant in our analysis, equation 2.9 can be integrated to determine

the pressure in a fixed volume as a function of leak rate and time, resulting in

$$P = \frac{1}{V} \int_{t_0}^t Q(\tau) d\tau + P(t_0). \quad (2.10)$$

In derivative form, equation 2.10 bears a strong resemblance to the current-voltage relation for a capacitor:

$$Q(t) = V \cdot \frac{dP_{in}(t)}{dt} \iff i(t) = C \frac{dv(t)}{dt}. \quad (2.11)$$

It is assumed that the leak rate will be a linear function of the partial pressure differential across the seal. This assumption is justified if the leak is in the molecular flow regime. This equation looks very much like Ohm's law:

$$Q = \frac{P_{out} - P_{in}}{R} \iff i = \frac{V}{R}. \quad (2.12)$$

Combining equations 2.12 and 2.11 results in the linear differential equation

$$RV \frac{dP_{in}}{dt} + P_{in} = P_{out}. \quad (2.13)$$

It can be assumed that  $P_{in} = 0$  at  $t = 0$ , since oxygen will have been flushed from the encasement. The exact solution to equation 2.13 for zero initial conditions is

$$P_{in}(t) = P_{out}(1 - e^{-\frac{t}{RV}}). \quad (2.14)$$

If  $t \ll RV$ , then  $P_{in}(t)$  can be approximated as

$$P_{in,approx}(t) = t \cdot \frac{dP_{in}(t_0)}{dt} = t \frac{P_{out}}{RV}. \quad (2.15)$$

We can rearrange this equation to find the time  $t$  at which  $P_{in,approx}$  reaches the limit of  $P_{in,max} = 0.005$  atm, resulting in

$$t = \frac{P_{in,max}}{P_{out}} \cdot RV. \quad (2.16)$$

Use of the approximate Equation 2.16 would result in  $t$  being underestimated by

1.2%, regardless of the value of  $RV$ . Given the accuracy of permeation data, such an approximation is both useful and justified when establishing design parameters for an encasement.

Now the only missing piece is the calculation of  $R$ . We will break  $R$  into two components,  $R_{phys}$  and  $R_{perm}$ , the first corresponding to the “physical” leak rate around the seal, and the second corresponding to permeation through the seal. These are separated because they must be measured in different ways. The physical leak rate  $Q_{He}$  can be readily measured with a helium leak detector. The physical leak rate is associated with surface defects in the seal or sealing surface. We can apply equation 2.12 to find the seal resistance from  $Q_{He}$ , but we are interested in oxygen ingress, not helium ingress. The seal resistance can be scaled to compensate for the increased resistance of the seal to the larger oxygen molecule by multiplying by square root of ratio of the molecular weights of oxygen to helium,  $2\sqrt{2}$ , resulting in

$$R_{phys} = \frac{P_{He}}{Q_{He}} \cdot 2\sqrt{2}. \quad (2.17)$$

$P_{He}$  is the partial pressure differential of helium across the seal. The side of the seal connected to the leak detector is almost always evacuated, and the opposite side is usually exposed to 100% helium at atmospheric pressure. For this situation  $P_{He} = 1$  atm. An example where  $P_{He}$  will have a different value is when an inner seal is tested with the inside of the encasement at atmospheric pressure but containing only 2% helium by volume; for this situation  $P_{He} = 0.02$  atm.

One additional caveat is that  $Q_{He}$  may need to be adjusted to a leak rate of room temperature gas as discussed previously.

With the resistance to physical leaks known, the next step is to calculate a resistance for permeation. To recap section 2.4.1, the oxygen flow rate through the seal due to permeation can be estimated to be  $KLP_{out}$ , where  $K = 2.23 \times 10^{-9}$  atm – cc/sec – cm. Applying 2.12 results in

$$R_{perm} = \frac{1}{KL}. \quad (2.18)$$

The total resistance can be found by treating  $R_{phys}$  and  $R_{perm}$  as adding in parallel; therefore

$$R = \left[ \frac{1}{R_{phys}} + \frac{1}{R_{perm}} \right]^{-1}. \quad (2.19)$$

Combining equations 2.17 through 2.19 results in

$$R = \left[ \frac{Q_{He}}{P_{He}2\sqrt{2}} + KL \right]^{-1}. \quad (2.20)$$

If the initial oxygen flow rate  $Q_{O_2}(t_0)$  into the encasement is desired, equation 2.20 can be combined with equation 2.12, resulting in

$$Q_{O_2}(t_0) = \frac{P_{out}}{R} = P_{out} \left( \frac{Q_{He}}{P_{He}2\sqrt{2}} + KL \right), \quad (2.21)$$

where one can clearly see the contributions to oxygen flow rate from both physical leaks and permeation.

## 2.5.2 Multiple seal, multiple volume analysis

From the development in the previous section, it should be clear that seals and volumes can be treated as linear circuit elements. Seals can be modeled as resistors and volumes can be modeled as capacitors. Little can be done to increase the resistance of a seal of fixed geometry and material, but seals can be added in series, increasing the equivalent resistance. Since the inner chamber of an encasement cannot be evacuated, at a minimum it is necessary to have an evacuated space between two seals for leak testing. Having two seals instead of one approximately doubles the time for oxygen to rise to a preset level. An encasement design could contain even more seals in an effort to curb leak rates, but there are practical limits to how far this can be taken.

In the analysis that follows, we will use  $R_{eq}$  to denote the equivalent resistance of a seal network. Seals in series add like resistors in series:

$$R_{eq} = R_1 + R_2, \quad (2.22)$$

and seals in parallel add like resistors in parallel:

$$R_{eq} = \frac{R_1 R_2}{R_1 + R_2}. \quad (2.23)$$

Figure 2-9 shows a schematic of an RC network representing the encasement cross section from Figure 2-5. There are a few ways to go about analyzing such a situation. The designer could model the network in SPICE or Simulink<sup>®</sup> or another suitable program, and plot the partial pressure of oxygen inside the encasement as a function of time. The designer could also crank through the differential equations. Alternatively, the network could be simplified such that it can be analyzed in a few lines of math. We will focus on that approach here.

In the case of the Massachusetts Archives encasements (and likely any other practical encasement design) the seal network can be reduced to a single first order RC filter. Since the volume of  $V_3$  is much greater than either  $V_1$  or  $V_2$ ,  $V_1$  and  $V_2$  have little ability to store oxygen and therefore have little effect overall performance. These volumes can therefore be eliminated from the analysis.

Further simplifications can be made based on the ability to test the physical leak rate of particular seals.  $R_1$ ,  $R_2$ , and  $R_3$  are not helium leak tested in the Massachusetts Archives design.  $R_1$  and  $R_2$  exist mainly to provide compliance for the glass to compensate for manufacturing tolerances and thermal expansion.  $R_3$  exists to allow helium to be applied across  $R_4$ . Unless there is a method to introduce helium across  $R_3$ , a physical leak rate cannot be established for this seal. Testing these seals would greatly complicate the design of the plumbing as well as the testing procedure.

Based on these simplifications, the  $RV$  time constant for this seal network can be estimated to be  $(R_4 + R_5)/V_3$ .

### 2.5.3 Leak budgeting

In Section 2.7, we show that with careful sealing surface preparation, the physical leak rate can be driven to near zero, *i.e.*, permeation dominates leakage through the seal. This makes it possible to estimate the time between flushes even though the

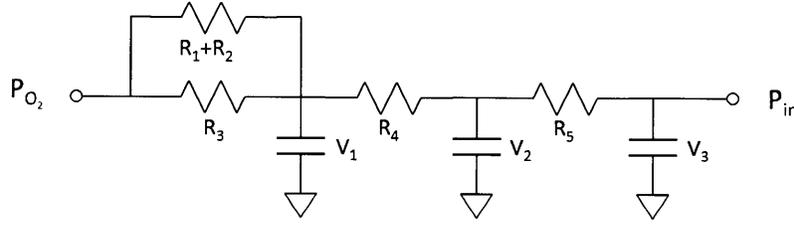


Figure 2-9: Schematic of seal network model.

Table 2.4: Predicted time until O<sub>2</sub> concentration inside encasement reaches limit of 0.5% for various encasement sizes using two Viton<sup>®</sup> seals.

size typical of	$l$ (cm)	$w$ (cm)	$h$ (cm)	$V$ (cc)	time to 0.5% O <sub>2</sub> (years)
8.5" × 11" letter	35	25	5	4375	4.5
Magna Carta	90	60	5	27000	11.0
Bill of Rights	100	80	5	40000	13.6
Waldseemüller Map	140	250	8	280000	44.1

physical leak rates cannot be known at this early stage.

Assume the encasement is a rectangular prism of length  $l$ , width  $w$ , and height  $h$  and that the length of the seal is the perimeter of the top face of the prism,  $2(l + w)$ . Substituting these dimensions as well as the maximum partial pressure of oxygen in the encasement into Equation 2.16 results in

$$t = P_{in,max} \cdot N \cdot \frac{lwh}{KP_{O_2} \cdot 2(l + w)}. \quad (2.24)$$

Table 2.4 summarizes the results for a few typical size documents for  $P_{in,max} = 0.005$  atm,  $K = 1.23 \times 10^{-8}$  atm – cc/sec – cm (20% squeeze) and the number of seals  $N = 2$ . One can see that large documents will withstand much longer periods between flushes. There are several options available to the designer to increase the time between flushes. The case can be enlarged or deepened, if space is available. More seals can be added, though  $N = 3$  is about the practical limit. Finally, the oxygen can be absorbed with the use of oxygen scavengers, which can maintain anoxic environments as long as the oxygen scavenger is not saturated and the seals are intact. This option will be explored next.

## 2.6 Oxygen scavenging

Oxygen scavenging (“getter”) products have recently gained acceptance in conservation work. Mitsubishi Gas Chemical Company’s (MGC) RP System<sup>®</sup> supplements their older Ageless<sup>®</sup> product. Unlike previous oxygen absorbing technologies, the RP System<sup>®</sup> is designed with conservation in mind. Setting this product apart from all other products, the RP System<sup>®</sup> “Type K” does not absorb moisture. This is an important feature in a conservation application where a constant relative humidity must be maintained. In cases where it is desirable to keep a specimen in an anoxic as well as dry environment, MGC also manufactures a “Type A” product.

The RP System<sup>®</sup> packets are rated in cc of air that it can remove the oxygen from, not the volume of oxygen absorbed. For example, a sachet with a rating of  $SR = 2000$  cc can absorb about 420 cc of oxygen, assuming a  $P_{O_2}$  of 0.21 atm. Let’s suppose that the length of time required to saturate the sachet is  $t_{sat}$ .

Assuming that the encasement will be purged when the oxygen concentration reaches a threshold, then  $t_{sat}$  can be thought of as the amount of time that oxygen scavenging can extend the service interval; *i.e.*, the encasement will need to be purged after time  $t_{perm} + t_{sat}$ .

However, oxygen scavenging also offers another possibility—one can keep the encasement essentially anoxic as long as the sachet is not saturated. Oxygen will ingress only after time  $t_{sat}$  has elapsed.

In either case, we can assume the flow rate of oxygen past the seal or seals is a constant  $Q_{O_2}(t_0)$ , because the partial pressure of oxygen surrounding the RP System<sup>®</sup> sachet is expected to be zero until such time that the sachet is saturated. We can then calculate  $t_{sat}$  in the more general terms of  $R$ , which can be determined from the approach outlined in Section 2.5.2.  $t_{sat}$  can then be calculated as:

$$t_{sat} = \frac{SR \cdot P_{O_2}}{Q_{O_2}(t_0)} = R_{eq} \cdot SR. \quad (2.25)$$

Note that  $R_{eq}$  should be calculated as the effective resistance between the getter volume and atmosphere, and will likely have a different value than the equivalent resistance of the encasement seal network. If the getter volume is separated from the volume containing the document as in the Massachusetts Archives design, the inside seal will not contribute to  $R_{eq}$ .

Using an approximate value of  $R_{eq} = 2.0 \times 10^5 \text{ sec/cc}$  (for the Massachusetts Archives encasement configuration), a single 2000 cc sachet can add almost 13 years between scheduled flushes, or if the getter is changed before it saturates and the seals are in good condition, there may be no need to perform a flush at all.

In any case, oxygen scavenging should not be seen as an excuse for careless o-ring gland preparation or a lack of cleanliness during assembly; instead, scavenging should be seen as a way to extend service intervals on a well-prepared encasement. Based on our very first attempts at obtaining an acceptable leak rate, the amount of RP System<sup>®</sup> used on the Massachusetts Archives encasements could have been exhausted in only a few years.

## 2.7 Massachusetts Archives encasement testing

To meet the required 20 years between service intervals, the Massachusetts Archives encasements employed oxygen scavenging sachets of the RP System<sup>®</sup> “Type K”. Four 2000 cc sachets were used. This provides a nearly anoxic environment for nearly 50 years, and approximately 60 years before the partial pressure of oxygen rises above the threshold of 0.005 atm. In practice, it is believed that the seals should be tested periodically to make sure that performance has not degraded significantly since initial installation.

Using the framework described previously, the leak testing results from the Massachusetts Archives encasements is calculated and tabulated in Table 2.5. Note that the  $t$  has been converted to units of years.

Table 2.5: Massachusetts Archives encasement leak testing results.

parameter	symbol	unit	Dec. of Ind.	Mass Const.	Bill of Rights	King Charles	Wm. and Mary
<b>constants</b>							
encasement volume	$V$	cc	4.23E+04	4.23E+04	4.23E+04	4.23E+04	4.23E+04
permeability	$K$	atm-cc/sec-cm	1.24E-08	1.24E-08	1.24E-08	1.24E-08	1.24E-08
<b>inner seal</b>							
seal length	$L$	cm	381	381	381	376	376
permeation resistance	$R_{perm}$	sec/cc	2.12E+05	2.12E+05	2.12E+05	2.15E+05	2.15E+05
helium leak rate	$Q_{He}$	atm-cc/sec	3.0E-10	9.0E-10	5.6E-09	3.0E-10	9.0E-10
helium partial pressure	$P_{He}$	atm	0.025	0.025	0.025	0.025	0.025
physical leakage resistance	$R_{phys}$	sec/cc	1.12E+09	3.74E+08	1.26E+07	1.12E+09	3.74E+08
total resistance	$R$	sec/cc	2.12E+05	2.12E+05	2.09E+05	2.15E+05	2.15E+05
<b>outer seal</b>							
seal length	$L$	cm	394	394	394	390	390
permeation resistance	$R_{perm}$	sec/cc	2.05E+05	2.05E+05	2.05E+05	2.07E+05	2.07E+05
helium leak rate	$Q_{He}$	atm-cc/sec	8.00E-07	1.60E-08	4.70E-07	3.90E-09	6.90E-09
helium partial pressure	$P_{He}$	atm	1.00	1.00	1.00	1.00	1.00
physical leakage resistance	$R_{phys}$	sec/cc	3.54E+06	1.77E+08	6.02E+06	7.26E+08	4.10E+08
total resistance	$R$	sec/cc	1.94E+05	2.05E+05	1.98E+05	2.07E+05	2.07E+05
<b>oxygen scavenger seals</b>							
total length (QTY 2)	$L$	cm	18.0	18.0	18.0	18.0	18.0
permeation resistance	$R_{perm}$	sec/cc	4.50E+06	4.50E+06	4.50E+06	4.50E+06	4.50E+06
<b>oxygen scavenger saturation</b>							
equivalent resistance (getter)	$R_{eq}$	sec/cc	1.86E+05	1.96E+05	1.90E+05	1.98E+05	1.98E+05
sachet rating	$SR$	cc	8000	8000	8000	8000	8000
time to saturate getter	$t_{sat}$	yrs	47.1	49.7	48.2	50.2	50.2
<b>overall performance</b>							
equivalent resistance (case)	$R_{eq}$	sec/cc	3.98E+05	4.08E+05	3.99E+05	4.13E+05	4.13E+05
time to 0.5% O <sub>2</sub> (no getter)	$t$	yrs	12.7	13.0	12.7	12.7	12.7
time to 0.5% O <sub>2</sub> (with getter)	$t + t_{sat}$	yrs	59.9	62.7	60.9	62.9	62.9

## 2.8 Conclusion

In summary, we have built a framework for the analysis of leakage through elastomer seals, including physical leakage and permeation. In practice, we have found that physical leakage can be controlled through careful surface preparation (described in Chapter 4) and assembly (described in Appendix A), resulting in permeation being the predominant type of leakage. Helium leak test data for the Massachusetts Archives encasements was presented and discussed. Based on these results, it is possible to accurately estimate seal performance from encasement geometry alone, allowing designers to rapidly explore available options for meeting desired performance specifications.



# Chapter 3

## Mechanical Design

This chapter focuses on the mechanical design of encasements. We will begin with an overview of the design process, and then dive into some details of the mechanical design and design process of the Massachusetts Archives encasements. The goal of this chapter is not to document every design detail, as we would quickly run out of space. Instead, we will attempt to develop the reader's understanding of how the system works as a whole, while giving extra attention to key design decisions. This chapter will also point the reader in the correct direction for companies that can process encasement components. All supplier contact information is included in Appendix C. Because a design is never done, Section 3.6 will outline desired design changes in a future second-generation encasement design.

### 3.1 A few words on design process

The design process has been described in the literature using spirals, circles, and block diagrams. In practice, it would be nice if the design process was this orderly. No matter how the design process is described pictorially, the theme is always the same: the designer must find a way to accommodate the constraints imposed by various influences on the project, and, with some hard work, the process often converges. Very likely, the final design could hardly be considered optimal; there will almost certainly be areas that can be improved on—examples include manufacturability,

ease of assembly, robustness, and aesthetics.

An idealized design sequence might go something like this:

1. Define project goals.
2. Conduct background research, including prior art and competing products.
3. Develop the underlying mathematical models.
4. Find off-the-shelf products that solve as many problems as possible.
5. Design the missing pieces and integrate everything.
6. Build, test, tweak.

The encasements designed for the Massachusetts Archives are similar to other design projects, and the above recipe is a good starting point. This chapter concerns itself with steps four and five, while the previous chapters have covered the previous steps in detail.

The importance of step four should not be underestimated. Many products already exist that solve difficult industrial problems—for an encasement, products used in the semiconductor and aerospace industries can come in very handy. Examples include seals, fasteners, fittings, sensors, and electrical & vacuum components. A good designer should arm oneself with catalogs and spend time getting to know the products. Every component will fall into one of three categories—purchased, purchased and modified, and fabricated. The goal of the designer should be to maximize the use of purchased components.

Step five is made much harder when working in uncharted territory. We had the great fortune of working with some of the members of the NIST team that designed the Charters of Freedom encasements, giving us access to valuable insights. Despite NIST’s extensive prior experience, many questions remained. To minimize risk, the Massachusetts Archives were designed to be as modular as possible. Of the five encasements, there are only two types, essentially one for “portrait” orientation and one for “landscape” orientation. Components are completely interchangeable on a single

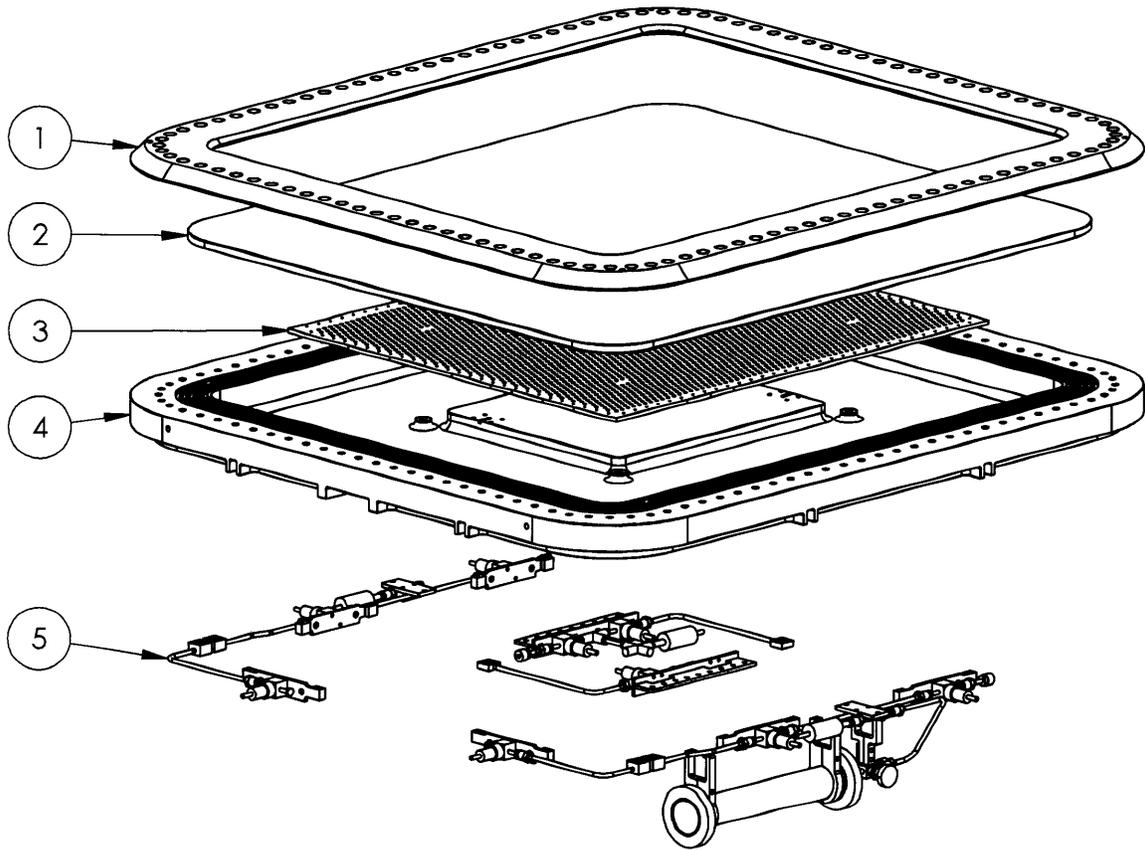
encasement type. Except for the few parts impacted by the change in size and orientation, component designs are shared between all five encasements. Extra mounting holes were drilled and tapped in the “tub” so that, in the future, new technologies such as updated instrumentation could be retrofitted. The plumbing was designed to allow replacement of sensors without breaking the seal. This modular approach, combined with defining key interfaces, allowed the longest lead time components to be ordered before the design was finalized, keeping the project on schedule.

## **3.2 Anatomy of an encasement**

Figure 3-1 shows the main components of the encasement. The largest and most expensive component is the “tub,” which in the case of the Massachusetts Archives encasements, is machined from a single piece of aluminum. The document rests on a “platen” which can be separated from the tub. The front of the encasement is a large sheet of glass, which is held on by a “clamp plate” that evenly distributes pressure to compress the seals (not shown). The bottom of the encasement houses the plumbing. The plumbing consists of three independent sections—the sections on the left and right of the figure connect between o-ring grooves, allowing helium leak testing and continuous monitoring, depending on the instrumentation connected. The section on the right of the figure also contains the oxygen scavenging components. The plumbing components in the center allow flushing of the encasement with humidified inert gas and continuous or periodic monitoring of conditions inside the encasement.

## **3.3 Tub, clamp plate, and glass**

The tub and clamp were machined by The Bechdon Company from 6013-T651 “Power Plate” donated by Alcoa, Inc. This material improves on the machinability and strength of 6061-T651. The platens were also machined by Bechdon, but from 6061-T651 aluminum. Figure 3-2 shows one of the tubs being machined on a horizontal machining center at The Bechdon Company. Aluminum parts were then hard anodized



ITEM	DESCRIPTION
1	clamp plate
2	glass
3	platen
4	tub
5	plumbing

Figure 3-1: Encasement exploded view.

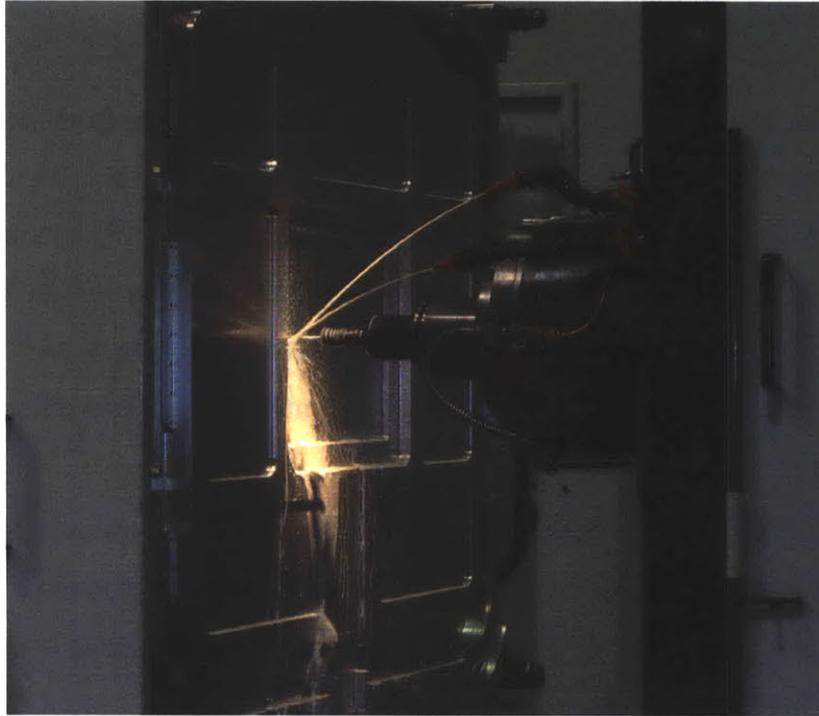


Figure 3-2: Machining the tub at The Bechdon Company.

and filled black by Alexandria Metal Finishers (AMF). The anodization process used on the encasements is the same as used for the interior of the Charters of Freedom encasements; the exact formulation is proprietary but has been approved by National Archives and Records Administration conservators.

The glass is Amiran<sup>®</sup> tempered glass with anti-reflective coating on both sides, produced by Schott AG. The glass is also two-layer safety glass, providing protection for the document from physical damage to the glass. Glass tolerances are notoriously wide, and as a result, impact the design of the tub and clamp plate. We were quoted  $\pm 0.5$  mm thickness tolerance and  $\pm 2$  mm width and height tolerance. Individual sheets, however, tend to be consistent in thickness. Too large a piece of glass will not fit in the recess of the clamp plate, and too small a piece of glass may not seal against the o-rings. As shown in Chapter 2, the glass thickness affects o-ring squeeze, which must be verified to be acceptable over the entire range of glass thicknesses.

## 3.4 Plumbing design

Because we aim to minimize leak rates, it makes sense to borrow from semiconductor processing technology, where maintaining ultra-high vacuum is critical. In small tubing sizes, Swagelok VCR<sup>®</sup> fittings are the gold standard for high vacuum fittings. These use metal seals to achieve leak-tight connections; usually, a helium leak detector will be unable to detect any leakage through these fittings. As VCR<sup>®</sup> fittings are commonly used on high vacuum systems, instrumentation is often available with this type of fitting.

While the VCR<sup>®</sup> fittings themselves are threaded, they are meant to be interconnected by welding to tubing or fittings such as crosses, tees, and elbows. These fittings give the designer great flexibility in locating components, but does require custom fabrication of bent and welded tubing assemblies. The designer should also be aware that for the various tubing fabrication tools to work properly, bends must have a minimum straight length on each end. When designing for tubing, one should be aware of these minimum tangent lengths for bending, trimming, and welding connections. These constraints are described in more detail in Chapter 4.1.

For general vacuum applications, fittings may be socket-welded, which can be welded manually by a skilled operator. For ultrahigh purity applications, fittings are often butt-welded to eliminate voids. While butt-welds can be performed by hand, orbital welding equipment is recommended for consistency. The minuscule amount of trapped contaminants in a socket-welded fitting would not adversely affect encasement performance; indeed the Charters of Freedom used socket welded components. However, the specialized orbital welding equipment necessary butt-welding had been purchased by an MIT laboratory and was made available for use on the Massachusetts Archives project. This equipment also enabled the use of Swagelok's Micro-Fit<sup>®</sup> fittings, which allowed for improved packaging of the instrument manifolds attached to the sensing grooves.

The Massachusetts Archives encasements required custom fittings for sealing to the aluminum tub were also required. One of these custom fittings is shown in Figure

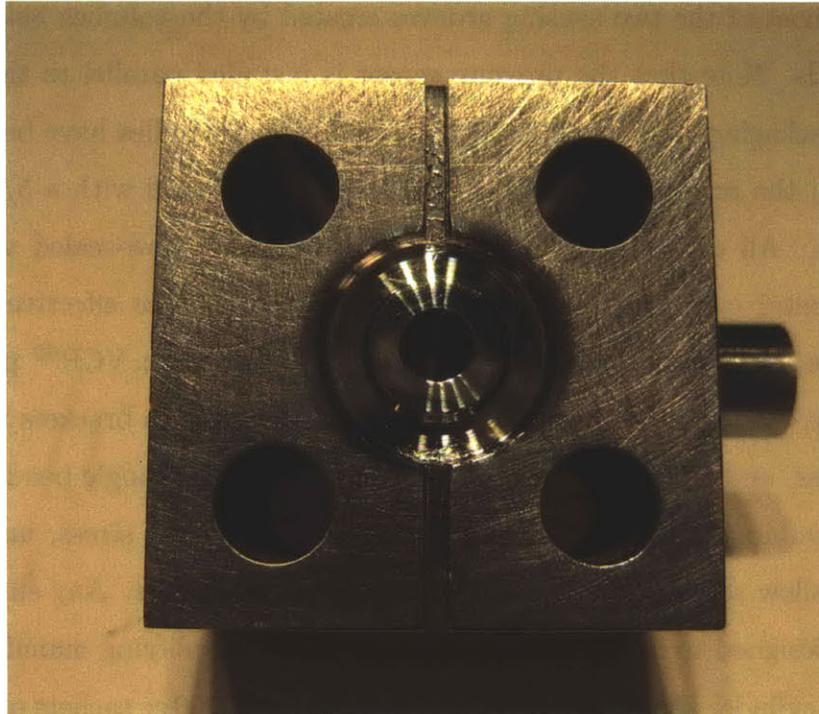


Figure 3-3: Custom fitting block for connecting stainless tubing to the encasement tub.

3-3. This block connects stainless steel tubing to the aluminum tub using an elastomeric o-ring. Note the two grooves connecting the o-ring gland to the edge of the block; these grooves are used for introducing helium to the seal gland for leak testing. Also note the very short short tube required for butt-welding; the compact design of this component is enabled by the Micro-Fit<sup>®</sup> orbital welding head.

The plumbing on the back side of the encasements consists of several manifolds, many of which are visible in the two panels of Figure 3-4. The top panels shows the plumbing and instrumentation connected to the interior of the encasements. This is where connections are made to flush the encasement with humidified inert gas. Also note the pressure and humidity/temperature sensors (described in Chapter 5), and that the instrumentation is installed between two valves, allowing the instrumentation to be isolated from the encasement. This allows instrumentation to be repaired or replaced without disturbing the document's atmosphere. It is even possible to perform a helium leak test of the replacement instrumentation without oxygen ingress. The bottom panel of Figure 3-4 shows the instrumentation and oxygen absorber installed

on the innermost of the two sensing grooves created by the volumes between the o-ring glass seals. Note that the pressure sensor is installed parallel to the main line to improve packaging. Also note that the original valve handles have been removed to save space; the replacement valve handles can be operated with a 5/16" wrench or nut driver. All of the valves are metal-seat, bellow-sealed valves. Due to their all-metal construction, permeation is expected to be effectively zero. In case the valve seat wears, the exposed ports are capped with VCR<sup>®</sup> plugs during final assembly, adding redundancy. The valves are mounted to brackets with slotted mounting holes, and where space allows, manifolds have right angle bends. The slots allow the plumbing to be installed with a minimum level of stress, and the right angle bends allow some flex to accommodate small movements. Any single piece of plumbing is designed to have bends in only one plane, simplifying manufacturing.

The large cylinder shown in Figure 3-4 contains four 2000 cc sachets of Mitsubishi RP System<sup>®</sup> "Type K" oxygen absorber. The cylinder is simply a KF50 union, which uses Viton<sup>®</sup> seals. The effect of oxygen absorber as well as the effects of permeation through the elastomer seals in the oxygen scavenging system is included in the analysis presented in Chapter 2.

As one can see from the wide variety of commercially available components, the designer has relative flexibility to solve packaging and process constraints. It is, however, worth noting a few ironclad rules applicable to designing around VCR<sup>®</sup> fittings:

- watch out for minimum bend, trim, and weld tangent lengths.
- allow for the uncrushed and crushed gasket thicknesses when designing and tolerancing (0.028" to 0.022").
- allow components to float axially during assembly; leave fasteners loose until fittings are tight.
- make sure there is adequate clearance to tighten nuts, including the use of a backup wrench.

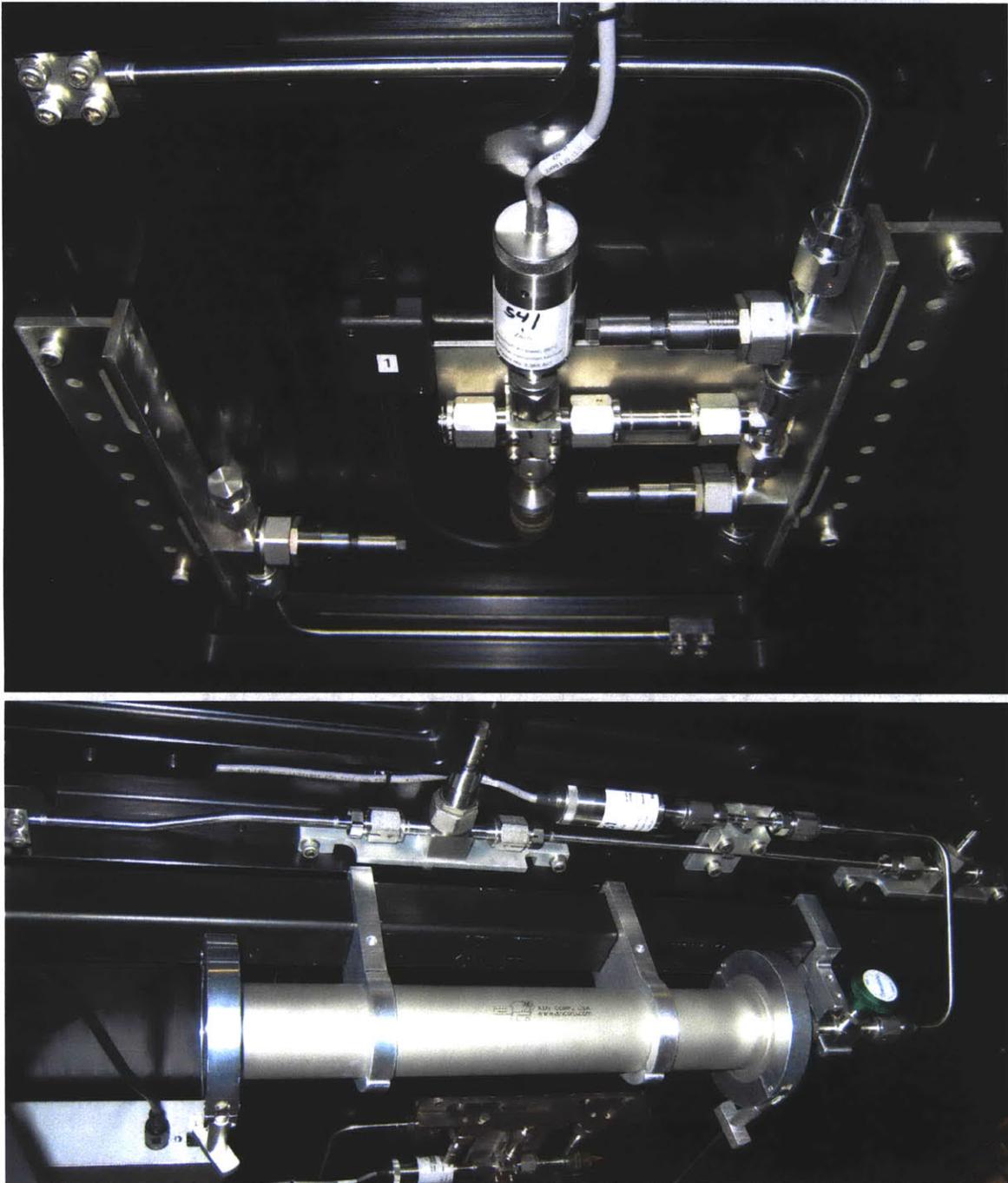


Figure 3-4: Top panel: Plumbing for the encasement interior volume. Bottom panel: Plumbing for the inner sensing groove. The large cylinder contains oxygen absorber.

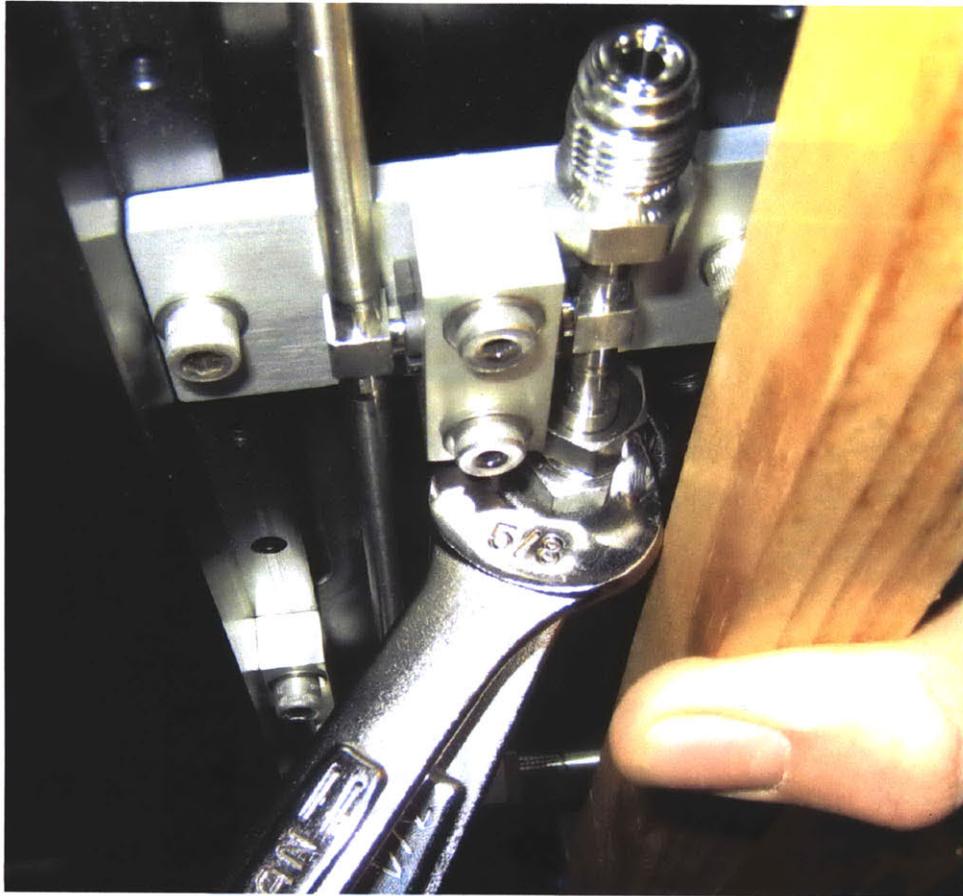


Figure 3-5: Testing clearances for a proposed design of a wheeled cart. The cart design required some minor changes to enable access to all fittings.

The final point is well illustrated by Figure 3-5, where it was noticed that the proposed design of a wheeled cart (which is part of the final museum display) would interfere with servicing the encasement. This mistake was caught before fabrication of the carts began, and the museum display designers were able to revise the design.

### **3.5 Platen design**

The platens support the document inside the encasement, but do not actually touch the documents. An acid free cardboard is installed between the document and platen. The following sections describe the process of digitizing the document outlines so that the platens could be fit to the shape of the documents, and also provide additional details on the design of the platen and the clips that secure the document to the

platen.

### 3.5.1 Digitization of document outlines

One of the most time consuming parts of the Massachusetts Archives project was producing accurate digital outlines of each document. It was desired that the platens would be cut to the outline of the document, with an additional 0.1” margin to allow for minor discrepancies in the tracing or movement of the document.

Tracings were made of each document on clear Mylar<sup>®</sup>. A French curve and a flexible ruler were used to draw smooth curves. These were then scanned by a local company with a large format scanner. The original tracings were scanned in black and white to maximize contrast.

These scans then required significant amounts of work to produce outlines suitable for machining platens. The scans will probably have extraneous black pixels, and the outline of the document will probably have islands of white. While an expert user of Adobe Photoshop<sup>®</sup> and Adobe Illustrator<sup>®</sup> could probably handle this situation gracefully, we found the process to have a steep learning curve. The process for conversion goes something like this:

1. The scans are opened in Adobe Photoshop<sup>®</sup>. Extraneous marks are removed, and the file is converted to grayscale.
2. The “paint bucket” tool is used to everything except the border gray.
3. Any remaining white areas are replaced with black using the “replace color” tool.
4. In exceptional cases, some of the outline may need to be repaired manually
5. The outline is then opened in Illustrator<sup>®</sup>.
6. The “live trace” tool is used to produce a vector outline that follows the scanned outline. The settings may require some adjustments to obtain the desired results.

7. The trace is exported as a DXF file.

The DXF file can then be imported into most CAD packages, such as SolidWorks<sup>®</sup>, where the holes for allowing the document to breathe, for attaching document clips, and mounting the platen can be created. A stack of three document outlines is shown in Figure 3-6. Note that the holes for allowing the document to “breathe” (refer to Section 3.5.2) are removed for clarity. Also note the irregular borders, and that the holes for document clips approximately follow the border. Ideally, the holes for attaching documents clips are placed at 0.5” from the edge, but the document clip design allows the hole to be placed as little as 0.4” from the edge.

The outlines from Figure were printed on clear Mylar<sup>®</sup> at full size and checked against the original documents before committing to the final design.

### **3.5.2 Platen design details**

The platens are constructed of 1/4” thick 6061-T651 aluminum, hard anodized and filled black. The anodizing, aside from being aesthetically pleasing, also prevents damage to the platens. The coating is hard enough to allow a cardboard backer to be cut with a razor, using the platen as a template, ensuring that the cardboard fits the platen exactly. The board specified by conservators was Kensington antique white 100% rag buffered mounting board, available from University Products.

The platens are perforated with a multitude of 3/16” diameter holes to promote gas contact with the back side of the document, as specified by conservators involved with the project. These holes are spaced approximately every 1/2”, as shown in Figure 3-7. The smallest document (the Declaration of Independence) had approximately 1800 perforations, while the largest document (the Bill of Rights) had approximately 3200.

### **3.5.3 Document clips**

The document clips consist of two components—a transparent Melinex<sup>®</sup> document clip that holds the document to the platen, and a black-anodized aluminum retainer

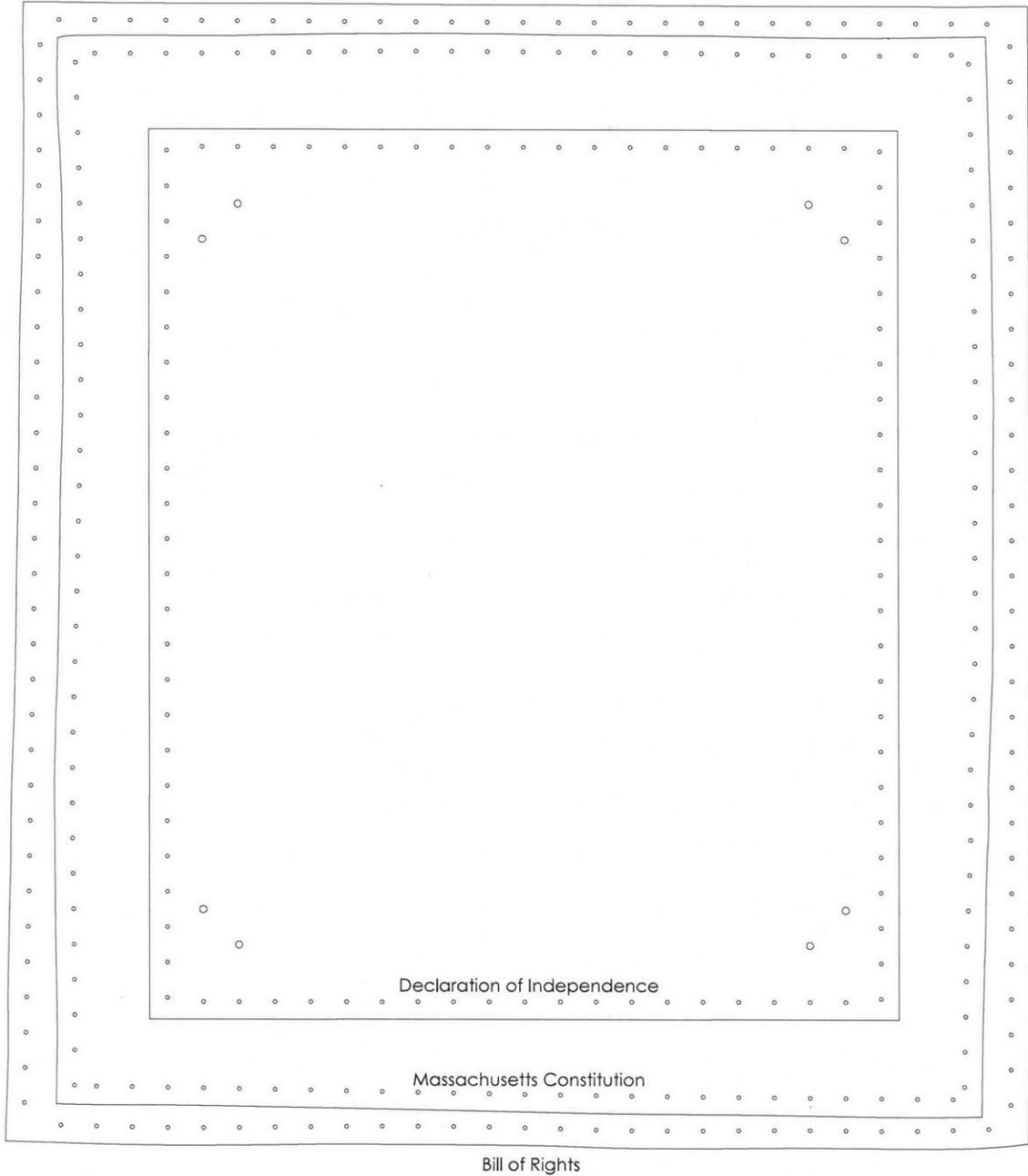


Figure 3-6: Platen outlines of the three “portrait” orientation documents.

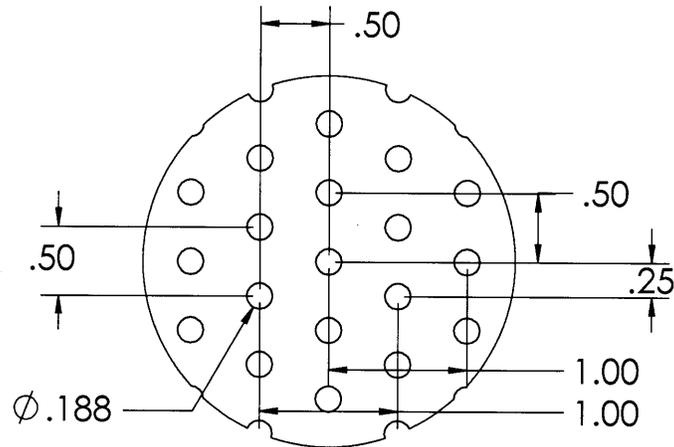


Figure 3-7: Platen hole pattern to promote gas contact with document.

that secures the document clip. Melinex<sup>®</sup> 515 and 516 polyester films are often used in framing and conservation work. The retainer fastens to the bottom of the platen with a small screw. In the low lighting of the museum, the retainer is nearly invisible and the transparent document clip is unobtrusive. Figure 3-8 shows the mechanical drawing of the document clip used for fastening documents. When the retainer is pressed to the edge of the platen, the acute bend in the document clip creates a downward force on the document.

Specialized tooling was needed to fabricate the document clips. A steel rule die (sourced from Apple Die) was ordered to cut the outline of the clip, and special bending fixtures were created from thin sheet metal. Steel rule dies allow short production runs of die cut pieces from thin sheet stock at extremely low cost. Most commonly, these dies are used to process gaskets and cardboard boxes. The dies are made from laser cut plywood, and the cutting edge is a sharpened steel strip. One die (~\$100) was able to cut over 100 document clips. The art for the die is shown in Figure die is shown in Figure3-8, and the actual die is shown in 3-9. Design for manufacturing is straightforward—the die manufacturer asked for a minimum radius of 0.063”, so the corners were radiused.

Two special bending fixtures (Figure 3-11) were designed to place the bends in the correct locations. The fixtures are made from three layers of stainless steel shim

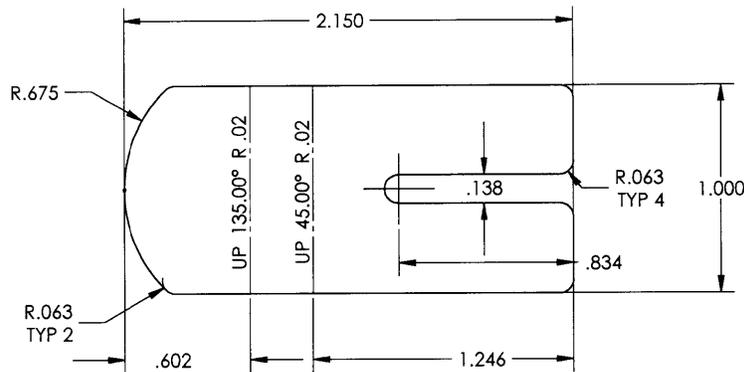


Figure 3-8: Die cutting pattern for the document clips. Material is 1000 gauge Melinex<sup>®</sup> 515.

create a bend. The obtuse bend was created cold, using the fixture shown in the top panel. The first acute bend was then made with the fixture shown in the bottom panel, and then a hot air source from a soldering station was waved over the bend to stress relieve the material, setting the bend.

### 3.5.4 Platen mounting

Kinematic couplings (KCs) are well-known for allowing repeatable assembly of components[19]. KCs mate assemblies with six point contacts, exactly constraining the design. KCs were used to mount the platen to the tub, eliminating both backlash and the possibility of the mechanism jamming. KCs need to be preloaded, and magnets are often the simplest solution. However, the use of iron gall inks on historic documents means that conservators will not allow magnets inside the encasement. Instead, KCs designed for Massachusetts Archives were preloaded with ball plungers that engage a ramp machined into a pin, as shown in Figure 3-12. The ball plunger (yellow) is adjusted by threading it in or out, until a suitable resistance is felt installing and removing the platen. The position of the plunger is then locked with the hollow lock screw (green), which is drilled through to allow the ball plunger to be adjusted without removing the lock screw.

Figure 3-13 shows the kinematic couplings installed on the bottom of the platen. The tub was designed to mount the platen in each of its four corners; of course, six is

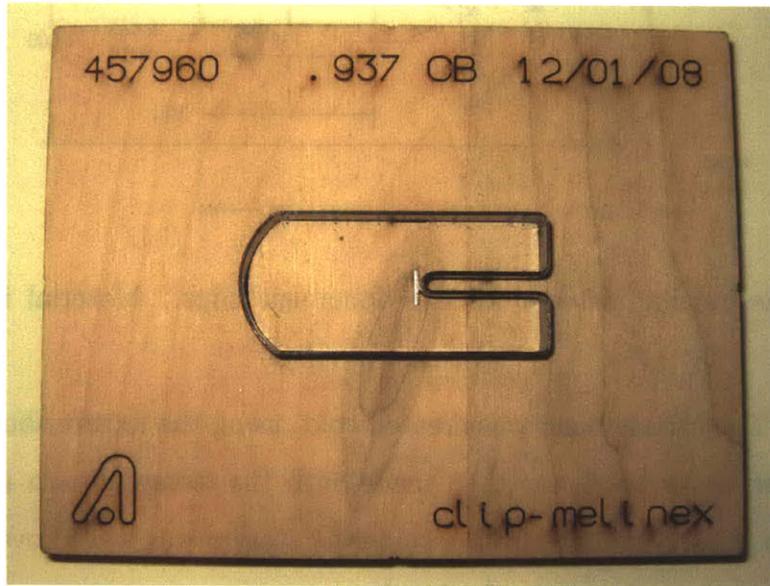


Figure 3-9: Steel rule die for cutting document clips.



Figure 3-10: Document clips in uninstalled and installed configurations.

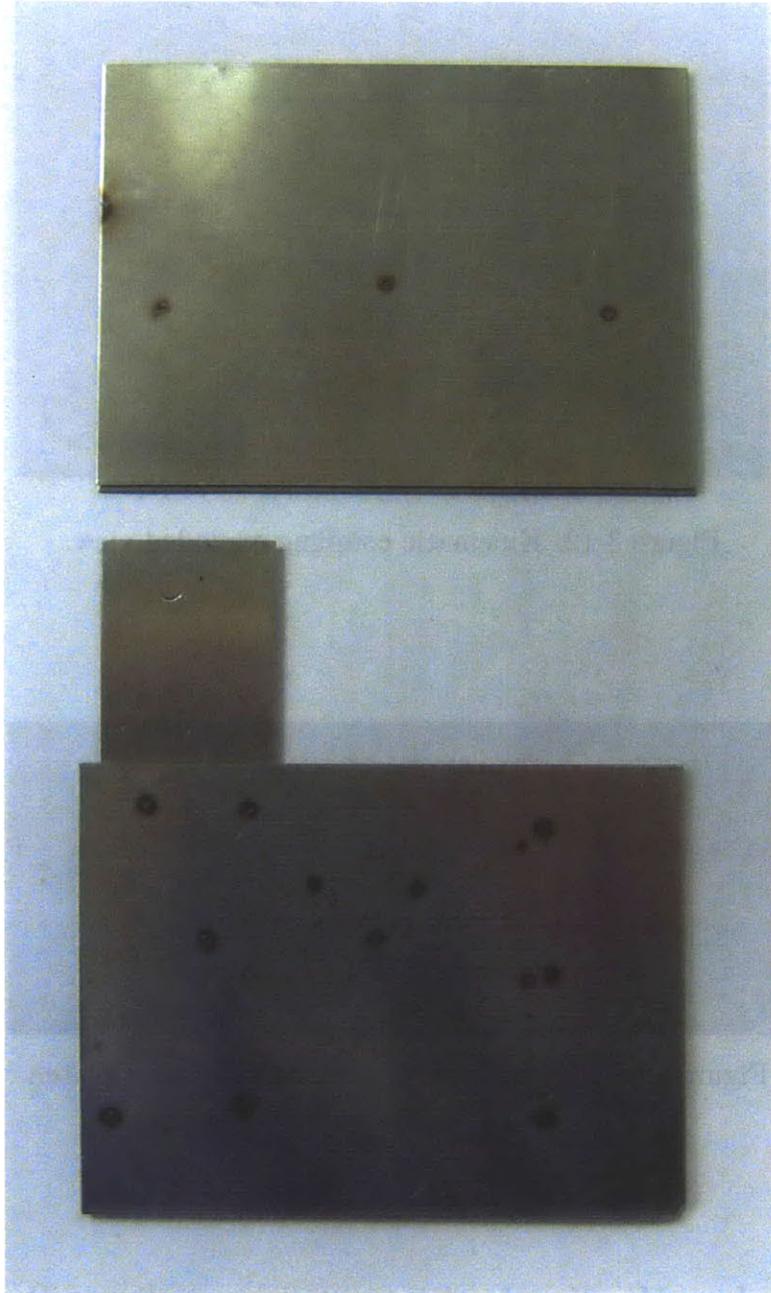


Figure 3-11: Document clip bending fixtures.

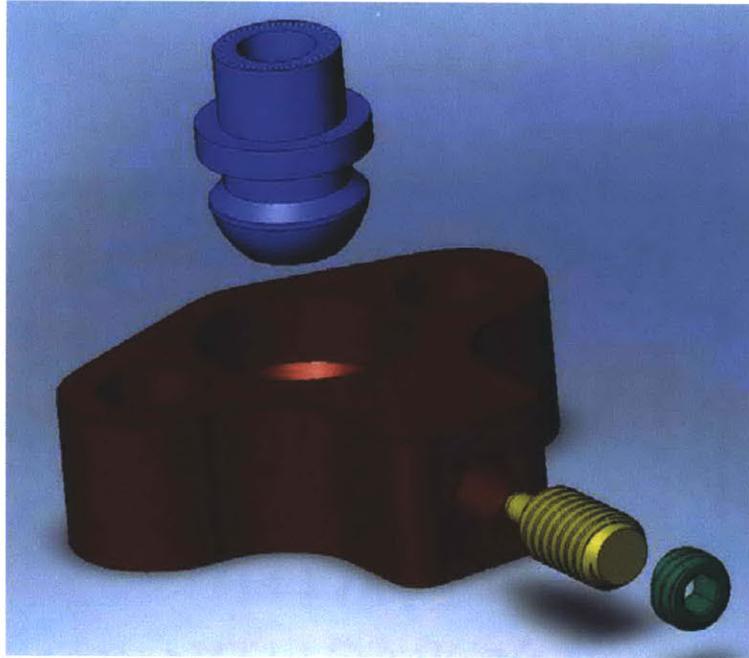


Figure 3-12: Kinematic coupling exploded view.



Figure 3-13: Kinematic couplings on document platen.

not divisible by four. Mounting the platen with three couplings with a contact pair each would exactly constrain the platen, but would mean that pushing down on a corner would likely overcome the coupling preload force and disengage. Instead, two of the couplings are designed to contact on only one point, exactly constraining the platen.

## 3.6 Second-generation encasement design

After the Massachusetts Archives encasements were installed, an updated encasement design was prepared, with the goals of honing CAD skills and investigating opportunities to encase other documents. Several refined design details are apparent in Figure 3-14:

- Smaller valves are used to decrease the border around the document.
- Smaller seals allow three seals to seat on the glass, and a procedure could be developed to test the outermost seal.
- The clamp plate bolts use nuts instead of threading into tub. This allows for a stronger interface and less chance of permanent damage to the tub.
- The glass is retained with clips, making installation easier and safer.

What is not so obvious from the figure is the sophistication of the CAD model. The extensive use of top-down solid modeling methods ensure that the major components fit together, even if encasement dimensions need to change. Critical dimensions are equation driven, so if the seal geometry changes, for example, both the tub and the clamp update to use the new dimensions. Extensive use of patterns ensures that all fasteners are shown in the model and that bolt holes line up between components. Such a detailed model also allows direct creation of bills of material and other documentation such as weight reports.

Other technical details were updated as well. One of the most significant changes involve the sensing grooves. A comparison of the layout between Massachusetts Archives encasements and the 2nd-generation encasement are shown in Figure 3-15. This new layout is expected to decrease the response time of the helium leak detector, as the “dam” that forces gas around the sensing grooves when flushing is no longer present. To aid in packaging, a single fitting block now contains ports for both sensing grooves. By machining the end at an angle, the tedious operation of creating an offset bend in a tube is avoided. The changes to the sensing grooves also

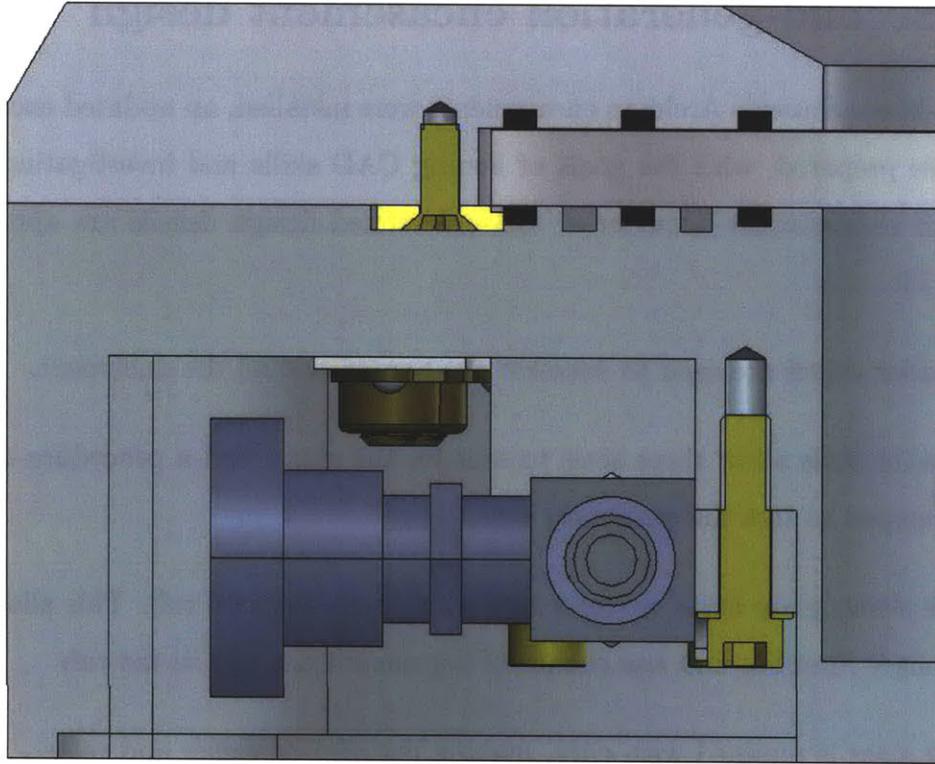


Figure 3-14: Second-generation encasement cross section.

allow both outer manifolds to be identical, decreasing fabrication costs. Figure 3-16 shows the updated manifold.

Finally, platen clips were designed to positively lock, which would retain the platen in case the encasement was inverted, which would increase safety if the document travels or was stolen. This platen clip concept is shown in Figure 3-17.

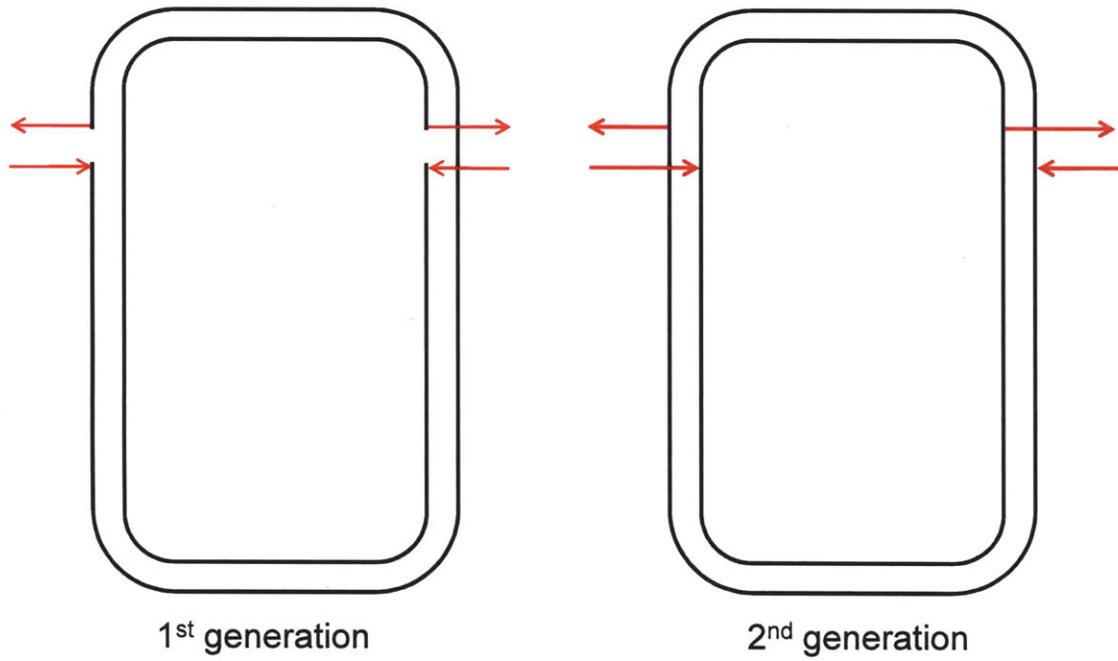


Figure 3-15: Revised sensing groove arrangement to allow for improved helium leak testing ability.

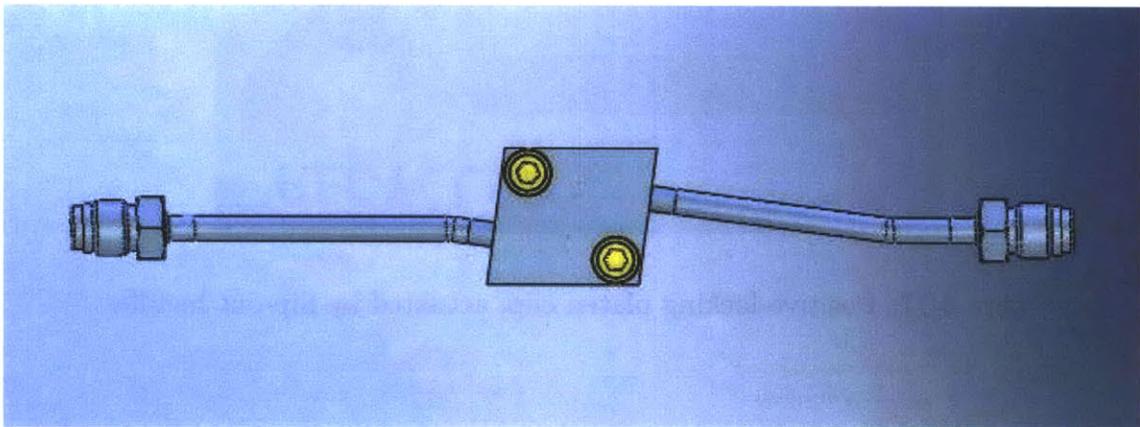


Figure 3-16: Revised manifold to simplify manufacturing and reduce parts count.

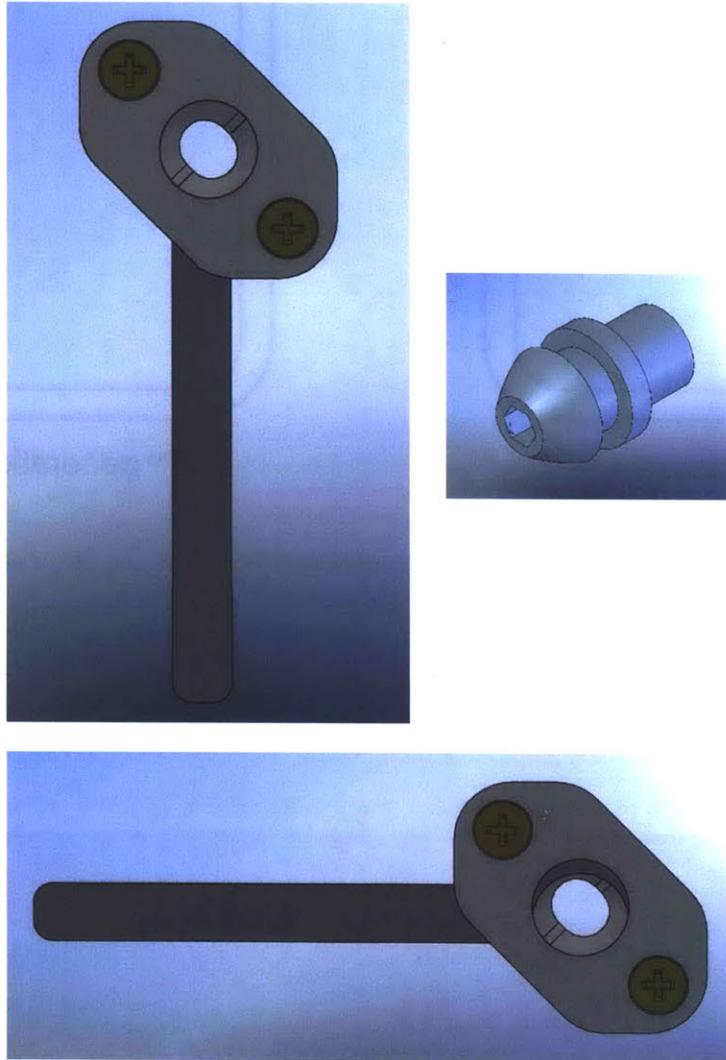


Figure 3-17: Positive-locking platen clip, actuated by flip-out handles

# Chapter 4

## Fabrication Tooling and Techniques

This chapter discusses the techniques used to fabricate certain components of the Massachusetts Archives encasements and describes the specialized tooling that was developed to aid in fabrication of these components. Much of this development was related to the production and sealing of the “plumbing” on the backside of the encasement, consisting of tubing, valves, sensors, and various fittings. These components must be made to tight tolerances, since the short tubing runs allow for only a minimal amount of flex. Furthermore, minimizing oxygen ingress means that sealing surfaces must be carefully machined and polished. This chapter is intended to serve as a how-to guide for when accuracy of fabricated tubing assemblies really matters, and for when sealing depends on having a perfect surface finish.

### 4.1 Tubing run fabrication

The short tubing runs on the back side of the encasements make the accuracy of fabricated tube assemblies critical. There are three components to maintaining high standards for accuracy: careful bending, careful trimming to length, and careful welding setup.

### 4.1.1 Tube bending

Tube benders place some constraints on which geometries can be successfully bent. For example, there must often be a straight section of tube on both ends of the bend (“end tangents”), because the bend must not interfere with functioning of the die or follower. For the Massachusetts Archives encasements, all tubes were bent using a Swagelok MS-HTB-4 bender. This tool is shown in Figure 4-1, along with dimensions of the tangents and the distance from the end of the tool to the start of the bend. These design-for-manufacturing (DFM) constraints should be considered when the shape of the tube is designed. If necessary, the tube can be trimmed shorter after bending, but should not be shortened beyond the minimum weld tangent. For the Micro-Fit<sup>®</sup> welding head used on this project, the weld tangent can be as little as 1/4”. Figure 4-2 shows the minimum tangents for various manufacturing operations.

Bending tubes accurately is especially easy when there is only a single bend in a tube. Even if the angle is somewhat off, this can be corrected by hand with 1/4” tubing. Due to the ease of manufacturing, the tubing runs were designed to have single 90° bends wherever possible. Two tubes on each encasement, however, required offset bends due to packaging constraints. These bends required careful work to place and to maintain planarity. It was decided that stops would be placed accurately on straight tubing, and that these stops would then be used to repeatably locate the bends. After bending, the tubing was then checked for fit in a fixture (Figure 4-3) and checked for planarity. The tube was then reworked or replaced if necessary. Figure 4-4 shows the offset bending technique using shaft collars as stops. The shaft collars were adjusted until the distance from inside face to inside face was correct; this dimension was determined as follows:

- 1) The centerline path length from start of first bend to finish of second bend was determined, as shown in Figure 4-2.

- 2) 1.125” was added to each end for the offset from the end of the tube bender.

- 3) 0.100” was added to compensate for effects such as springback. This value was determined experimentally, and would probably be different for angles other than 15°.

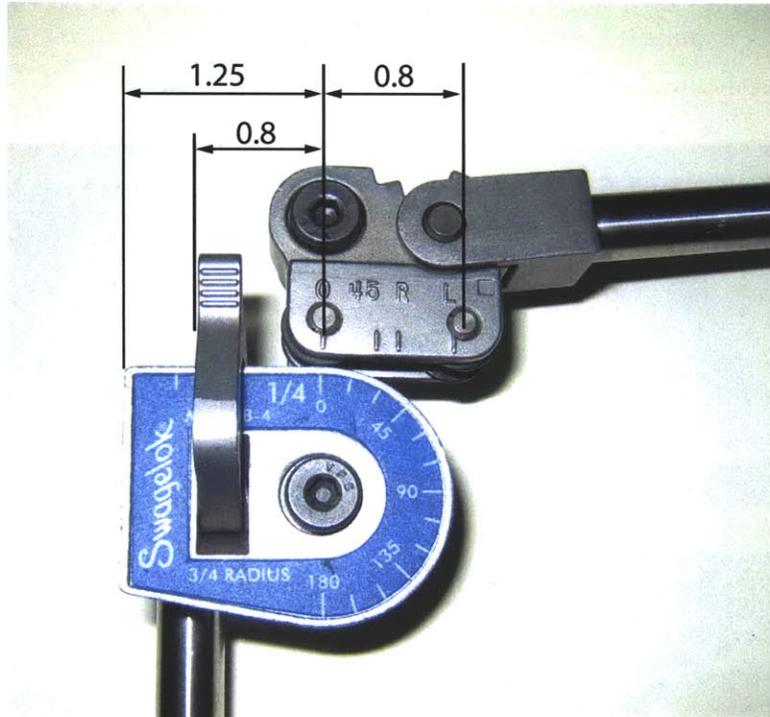


Figure 4-1: Minimum end tangents of a Swagelok MS-HTB-4 bender.

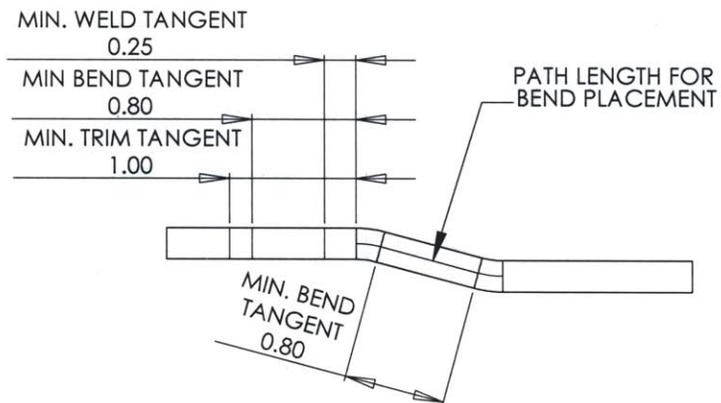


Figure 4-2: Minimum tangents for tube manufacturing and path length determination for an offset bend.

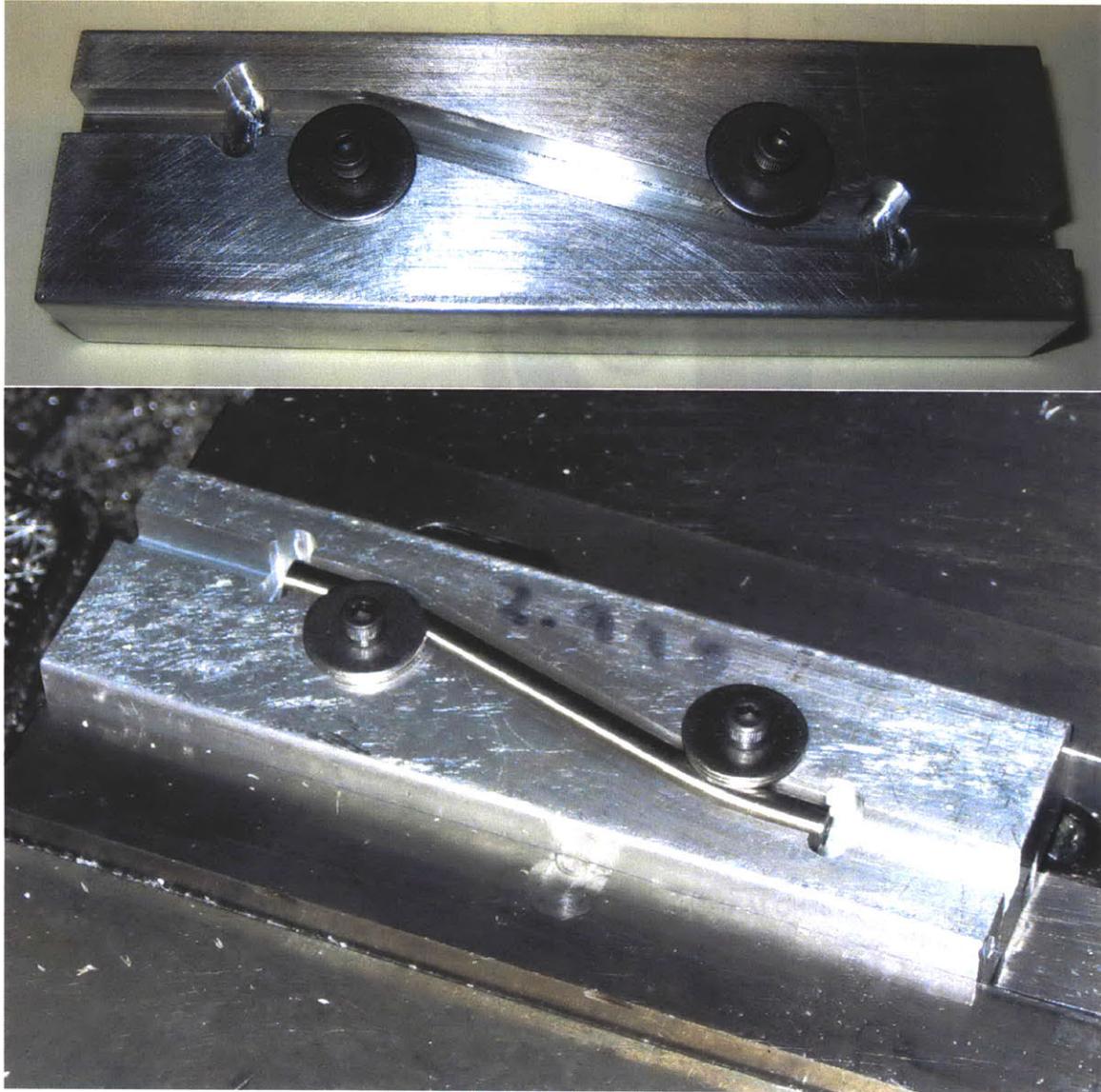


Figure 4-3: Photographs showing the offset bend fixture (top), which is used to hold a tube in the correct shape while trimming to length with a mill (bottom).

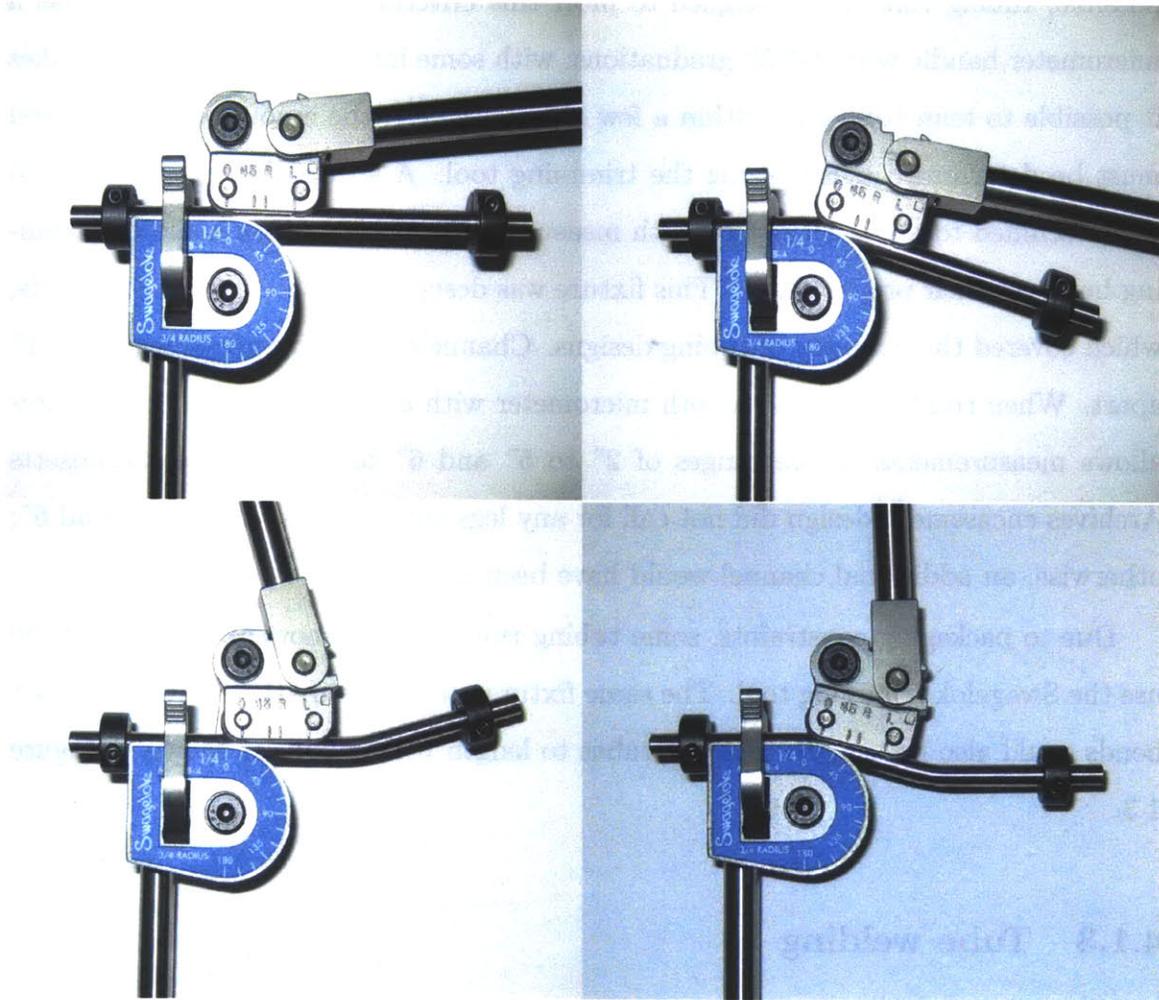


Figure 4-4: Bending sequence for an offset bend.

### 4.1.2 Tube trimming

For butt-welded connections, the ends of tubing should be prepped so they are flat and square, with no burr and minimum fillet or chamfer on the ID and OD. Swagelok manufactures a special tool for preparing the end of tubing, part number SWS-232EP-1. This tool, however only works when there is an end tangent at least 1" long. Where possible, tubing runs were designed to meet this criteria. The Swagelok tool has a micrometer handle with 0.005" graduations; with some interpolation, this tool makes it possible to trim tubing to within a few mils. However, the amount to be removed must be determined before using the trimming tool. A special fixture (Figure 4-5) was machined to allow accurate length measurements of bent tubes while constraining bends to their proper shape. This fixture was designed for tubing with 90° bends, which covered the majority of tubing designs. Channels in the fixture were spaced 1" apart. When combined with a depth micrometer with a 0" to 1" range, this fixture allows measurements in the ranges of 2" to 5" and 6" to 9". The Massachusetts Archives encasement design did not call for any legs with lengths between 5" and 6"; otherwise, an additional channel would have been machined.

Due to packaging constraints, some tubing runs had too short an end tangent to use the Swagelok trimming tool. The same fixture used to verify the position of offset bends could also be used to trim the tubes to length with a mill, as shown in Figure 4-3.

### 4.1.3 Tube welding

Welding was performed with a Swagelok orbital welder at the MIT Plasma Science and Fusion Center. Specialized tooling was developed to fixture components and shield components with inert gas for welding.

Figure 4-6 shows a welding fixture designed for manifolds with 90° bends. The tubing is bolted to a spacer, which holds the tubing level with the fitting block. The weld joint is left only weakly constrained, since the alignment of the weld is controlled by the Swagelok welding head. This tool was designed to be modular; it could be

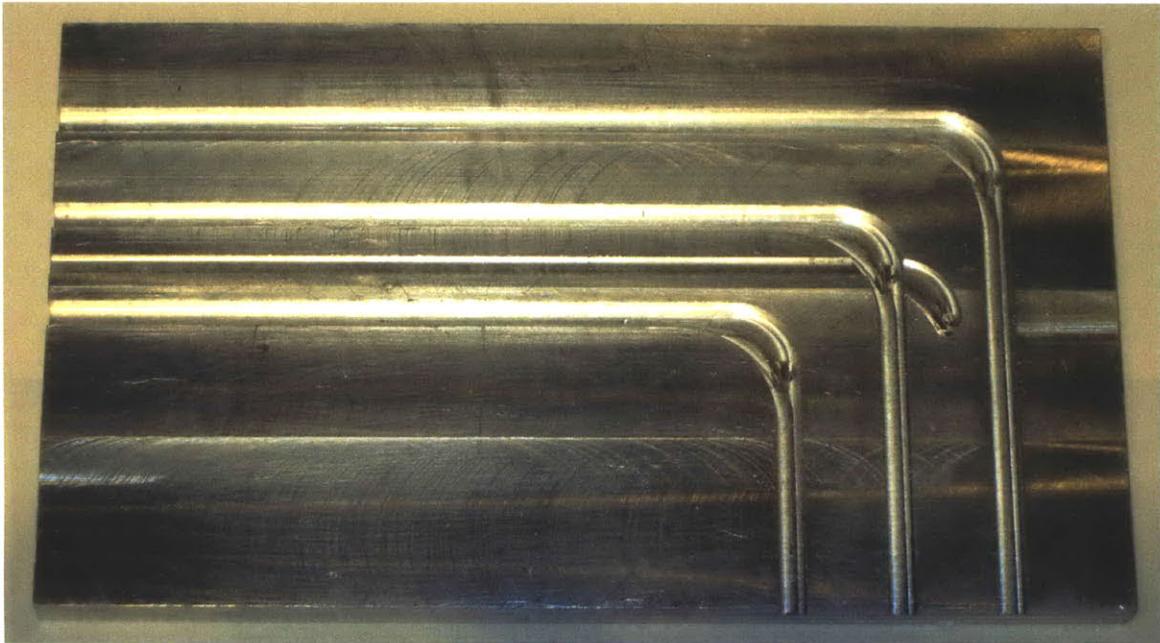


Figure 4-5: 90° bend fixture allows individual legs to be measured to determine how much material to remove.

used with tall or short fitting blocks (by substituting the spare spacer shown in the figure) and could be used with right or left-handed manifolds. The spacer could also be installed in any of three positions to accommodate legs of various lengths. This fixture is also used for “backpurging” the weld; the fitting on the left side of the figure is used for introducing argon into the tubing. Welding stainless steel requires protection from atmosphere on both sides of a weld. Swagelok manufactures a purge kit (P/N SWS-PURGE-KIT) that maintains a positive pressure of inert gas inside the tube while it is being welded. This kit includes fittings for adapting to tubing or fittings, restrictors, and a differential pressure gauge. This kit introduces inert gas from one end of a tube and generates a positive pressure by restricting the outlet. The flow rate is then adjusted to maintain correct pressure; for this project the pressure was set to 3” of water column (W.C.) ( 0.7 kPa). This unfortunate system of pressure measurement is the result of both the Swagelok purge kit and the Moisturematic (described in Chapter 6) using pressure gauges marked in inches of W.C. The Moisturematic can be used in place of the Swagelok purge kit, as described in Section 5.1.3.

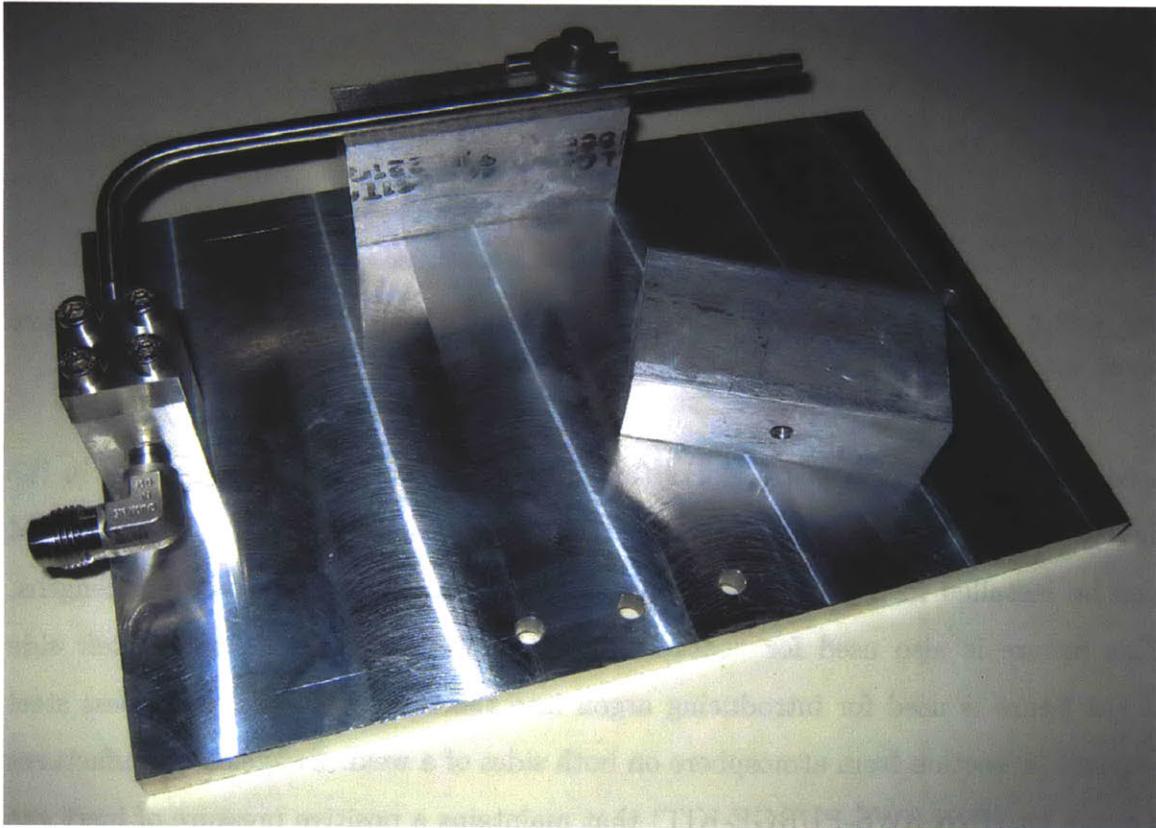


Figure 4-6: Welding fixture to align components and provide connections for shielding the inside of the tube with inert gas.

The Swagelok kit, however, is not capable of clamping onto the Micro-Fit<sup>®</sup> tees in the outer instrumentation manifolds, as the weld tangent is too short. Special purge adaptors were machined for this purpose, shown in Figure 4-7. These adaptors are designed for use with the fittings supplied with the Swagelok purge kit; the purge kit grips the cylindrical portion of the adaptor. Three types of adaptors were made to allow maximum flexibility while welding:

1. one with a large hole for introducing argon with minimum pressure drop,
2. one with a small hole for restricting the outlet,
3. and one with no hole for plugging passages.

Due to the assembly order chosen by the welder, only the restrictor and the plug were ultimately needed.

After welding, some minor finish work was required. Even with components fixtured, welding caused some distortion. When necessary, welded components were straightened by hand. Also, note that all plumbing components should be helium leak tested after final assembly. At a minimum, seals, welds, and bends should be tested to ensure no defects exist.

## 4.2 Finish polishing

Smooth surfaces are required for sealing. Parker recommends no rougher than a 0.4  $\mu\text{mRa}$  (16  $\mu\text{inchRa}$ ) surface roughness for high vacuum applications [12]. This finish was called out on all drawings for sealing surfaces custom components. Even more important than the value of surface roughness is the “lay”, the predominant direction of the marks left by machining operations. These should run with the circumference of the o-ring to avoid creating leak paths across the seal. On round parts, this pattern can be easily created by turning the part against a cutting tool or abrasive paper.

Due to the shape of the tub, the o-ring glands were milled, which creates lay marks perpendicular to the desired direction. Despite the manufacturer hand-working the

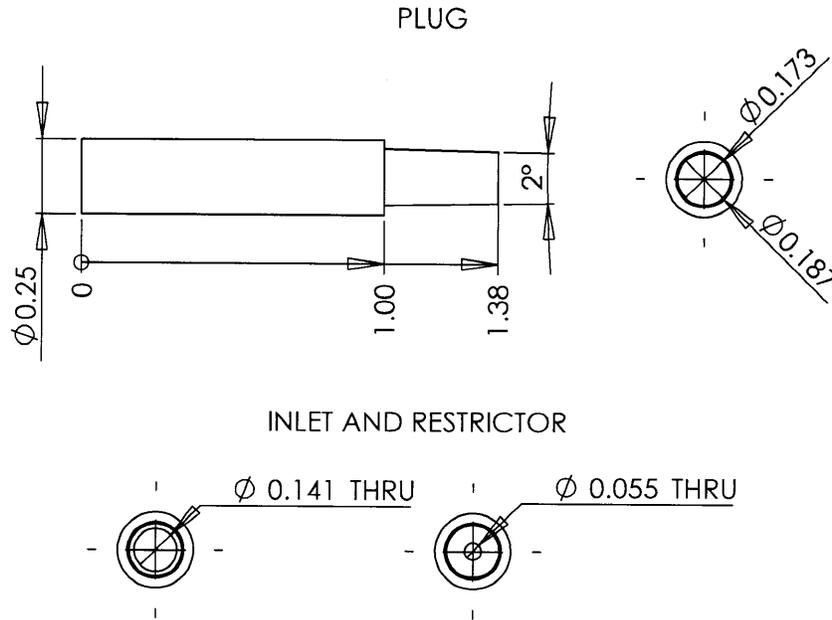


Figure 4-7: Custom purge adaptors for welding Swagelok Micro-Fit<sup>®</sup> tees.

sealing areas with Scotch-Brite<sup>®</sup> and the surface finish meeting the callout, these sealing surfaces leaked when assembled for the first time. In fact, several components, both custom and off-the-shelf, initially had difficulty passing a helium leak test. The sealing surfaces of these components were touched up using the techniques described here. After polishing, the helium leak rates of these components were all found to be acceptable, and many components exhibited no detectable leak.

#### 4.2.1 Fitting block preparation

The fitting blocks serve as the interface between the encasement and the instrumentation on the back side of the encasement. On these components, a single seal separates air from the atmosphere in the encasement. As a result, the surface finish on the sealing surfaces of these components must be held to a high standard.

The fitting block drawings specified that the machining marks run circumferentially; this essentially dictated the use of a lathe to machine the o-ring grooves. When the parts arrived, each one was inspected under a microscope, and it was noticed that the sealing surface had a rough appearance. While these parts may or may not have

met the surface roughness callout, it was thought that the surface was unsatisfactory but that the parts could be salvaged with careful polishing.

The o-ring gland is difficult to access for polishing. A felt polishing bob (McMaster 4811A17) was modified to fit inside the o-ring groove by drilling out the center and trimming the outside diameter with an X-Acto<sup>®</sup> knife. The bob was charged with an abrasive “cut and color” compound (McMaster 4784A2) and run against the fitting block in a drill press.

This technique was very successful. However, some of the blocks had defects that were too deep to be removed by polishing. Figure 4-8 shows a block that was not used due to a deep scratch in the sealing surface; it is thought that the machinist scratched the o-ring gland while removing a burr. Aside from the damaged area, the polishing operation left the surface well-prepared for sealing.

In hindsight, this defect probably could have been avoided if the o-ring grooves had been CNC milled instead of turned on a lathe. The polishing operation completely removed any machining marks, so turning the groove was an unnecessary complication.

## **4.2.2 Encasement polishing**

Though the encasement o-ring grooves met the surface finish callout on the drawing, the original helium leak tests consistently revealed unacceptably high leak rates. This may have been due to defective o-rings, imperfect groove finish, etching of the grooves from anodizing preparation, or a combination of these factors. It was decided that the best course of action was to replace the o-rings, refinish the seal grooves, and reassemble the encasement for retesting.

MicroMesh<sup>®</sup> MX series “cushioned abrasives”, manufactured by Micro-Surface Finishing Products, Inc., were used to refinish the o-ring glands. This product consists of silicon carbide particles embedded in a compliant latex and cloth backing. These products are said to produce a more consistent finish than sandpaper, and the abrasive sheets resist clogging.

The grooves were first polished with 180MX abrasive (said to fall between P360

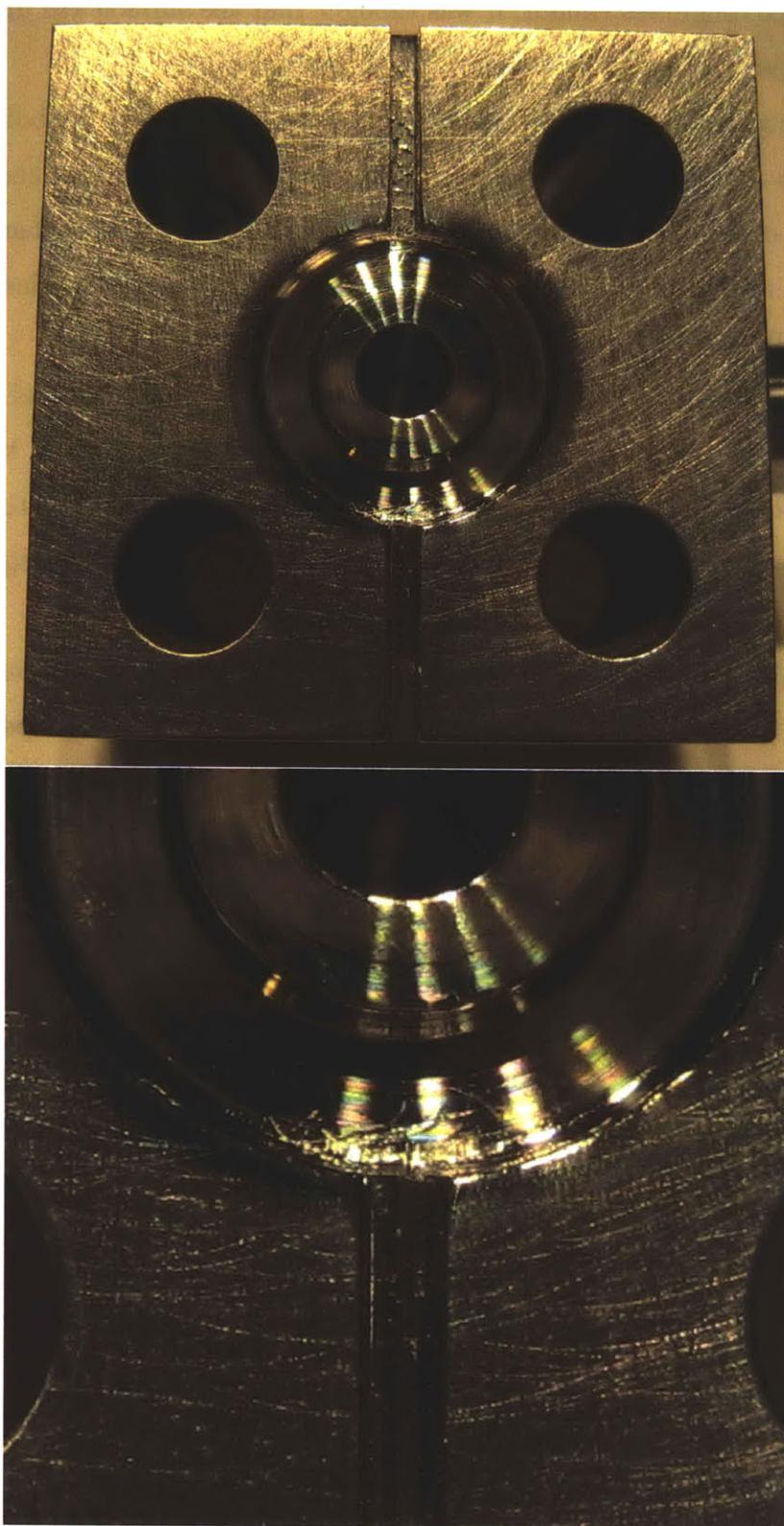


Figure 4-8: Polished block with defect in sealing surface



Figure 4-9: The o-ring groove is polished using foam poster board as a soft sanding block.

and P400 grit) and finish polished with 240MX abrasive (approximately P800 grit). The grooves were wet sanded; with wet sanding, water both acts as a lubricant and also flushes away aluminum particles and spent abrasive. Because the encasements already had instrumentation components installed, the same plugs that were used for helium leak testing (Section A.2.3) were used to keep the instrumentation clean.

To reach into the o-ring glands, strips of abrasive cloth were cut into 1/4" wide strips. The grooves were then sanded using foam board and pencil erasers as sanding blocks, as shown in Figure 4-9. The grooves were sanded in a continuous motion. After several passes, the dirty water was wiped up and refreshed. On the final passes with the finer grit abrasive cloth, special attention was paid to the lead-ins and lead-outs to ensure lay marks across the seal were not developed.

The encasement was then inspected under various lighting conditions. If any flaws were found, the entire circumference was polished again to avoid multiple lead-ins and lead-outs. Finally, the encasement was cleaned with an ethanol/water solution and allowed to dry. Final assembly immediately followed, as described in Appendix A.2.



Figure 4-10: The encasement is inspected with low ambient light and a flashlight.

### 4.2.3 O<sub>2</sub> getter bottle polishing

The getter bottles were off-the-shelf KF50 unions, designed for vacuum work. Oddly, these components had a bead-blasted finish; when subjected to a helium leak test, the seals leaked badly. These components were salvaged by polishing the sealing surfaces. Just like with the other components, sealing is best accomplished with the lay running along the o-ring. This pattern is easily created by rotating the part in a lathe against a stationary strip of sandpaper, but the shape of the getter bottle makes it difficult to grasp and its length makes holding the part from one end very dangerous. A small chuck was used to grasp the ID of the tube, and a bung was machined to support the far end with a live center. Both ends of the union were then sanded with 320 grit sandpaper. This technique is depicted in Figure 4-11. Note that the ways of the machine should be protected from abrasive particles with newspaper.

The end caps used to seal the getter bottles had a turned finish and did not require any finishing work to seal properly.

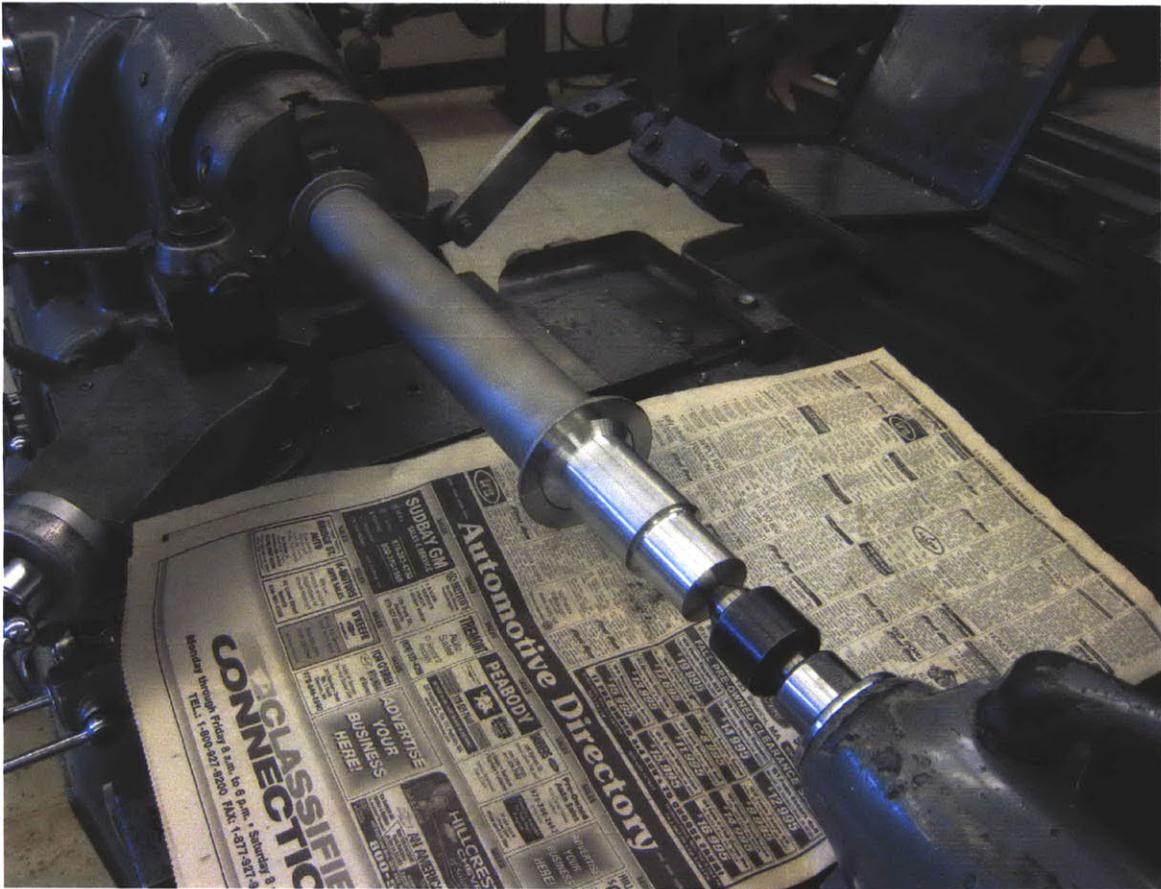


Figure 4-11: Getter bottle polishing.



# Chapter 5

## Instrumentation

This chapter is devoted to describing the instrumentation needed to verify the performance of the Massachusetts Archives encasements, as well as discussing various sensor technologies. It is hoped that the design and construction details discussed in this chapter, in the future, would allow a technician to easily service the instrumentation on the Massachusetts Archives encasements. Most electronics simply are not intended to last 20 or more years, and odds are the system will require servicing before the encasements need refilling.

### 5.1 Massachusetts Archives instrumentation design

This section describes the design of the instrumentation installed on the Massachusetts Archives encasements. This system measures and records temperature, humidity, and encasement and gap pressures on all five encasements.

#### 5.1.1 Overall layout

The Massachusetts Archives gallery design places the encasements in a group of two and a group of three, on opposite sides of the room. It was desired that the data would be collected and recorded in a central location, but signal integrity concerns required that analog samples be sampled at each encasement, and information transmitted

digitally to a utility closet near the gallery.

Of the many protocols available, Ethernet is probably the best known, and consequently, has the best product availability. Products exist to convert from Ethernet to just about any other protocol, making Ethernet very flexible. CAT6 Ethernet was specified to connect each encasement to the utility closet, which electricians installed during renovations of the museum gallery.

It was desired that off-the-shelf components be used to make servicing broken equipment easier. Even in the event that a component became unavailable, it is likely that a direct substitute or a substitute requiring only small modifications to the existing hardware could be found. However, there were no off-the-shelf, Ethernet-based data acquisition devices on the market with a reasonable price, at least three  $\pm 10$  V analog inputs, and a digital interface capable of directly communicating with the humidity sensor selected for the project; hence it was decided to pursue a serial or USB data acquisition system in conjunction with an Ethernet bridge.

Lantronix makes several Ethernet bridges for industrial applications. The two devices that were strongly considered were the UBox<sup>®</sup> 2100, a two port USB/Ethernet bridge, and the XPort<sup>®</sup>, a single port RS232/Ethernet bridge. These are pictured in Figure 5-1. The UBox<sup>®</sup> is a packaged product, ready for USB devices to plug in. The XPort<sup>®</sup>, which is barely larger than an Ethernet jack, is designed to be integrated into custom circuit boards. Lantronix also offers RS232/Ethernet bridges in standalone form factors, as well as serial/wireless bridges. A situation where a wireless bridge would be very useful is if power wiring was preinstalled, but data wiring was not.

Ultimately, a National Instruments USB-6008 (for acquisition) and a Lantronix UBox<sup>®</sup> 2100 (for interfacing) were chosen. The integration of these components into a complete system is described in Section 5.1.4.

### **5.1.2 Pressure sensing**

MKS Micro-Baratron(R) 870B pressure sensors were selected for sensing encasement and sensing groove pressures. The model selected had a range from 0 to 1000 torr,

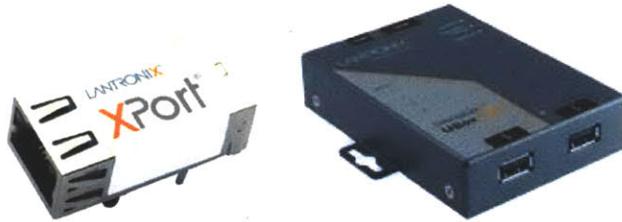


Figure 5-1: Lantronix Ethernet bridges

a 0 to 10 V output, 6 foot flying leads, and a female 1/4" VCR fitting. The part number for this combination is 870B13TCD2GF1. This sensor has all-metal wetted components, eliminating permeation as a concern.

While MKS could not provide a drift rate, an MKS engineer acknowledged that the largest contributor to drift is pressure cycling, of which sensors installed on encasements will see very little.

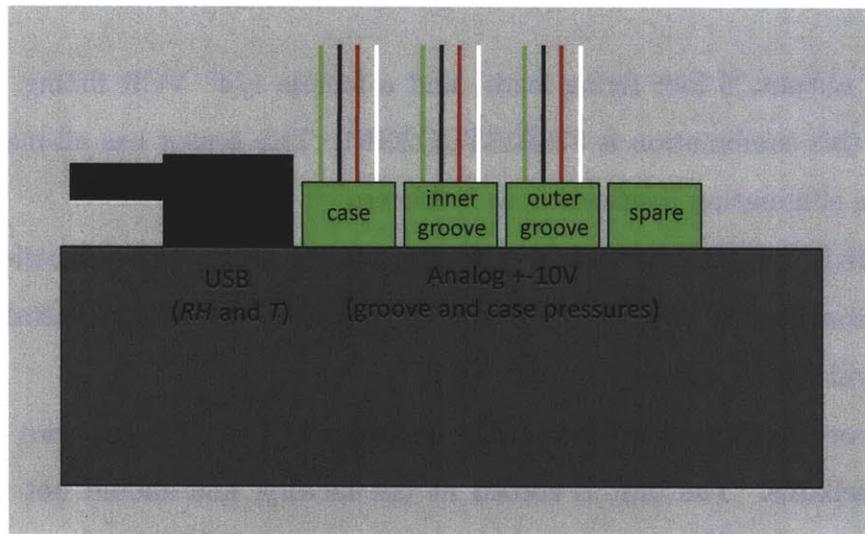
The sensor should be installed on the encasement such that the zero potentiometer is reachable. The unit is zeroed at the factory, and should not require an initial calibration. After installation, the sensor and connections should be helium leak checked. If the sensor does not read zero when helium leak checked, the zero potentiometer should be adjusted.

Assuming the sensor will be interfaced to the data acquisition hardware described in Section 5.1.4, the connectors should be installed as shown in Figure 5-2. Note that the color code is different for units with 10 foot flying leads.

### 5.1.3 Humidity sensing

A Sensirion SHT75 humidity sensor was selected for the Massachusetts Archives encasements. This particular sensor stood out from other capacitive or resistive humidity sensors because:

- The digital interface simplifies connection of the sensor to the data acquisition system.



DAQ box pin (1 is closest to USB port)	wire color	function
1	green	power (+24 V)
2	black	signal (-)
3	red	signal (+)
4	white	ground

Figure 5-2: Pressure sensor electrical connections.

- Calibration of individual sensors is performed at the factory, making the sensors interchangeable.
- Its rated accuracy and drift are among the best available. The accuracy is rated at  $\Delta RH = 1.8\%RH$  over the temperature and humidity range of interest. Long term drift is rated at  $0.5\%RH$  per year.
- Its form factor makes it very easy to integrate with a hermetically sealed housing.

### **Overview of housing fabrication and assembly**

A hermetically sealed housing for the sensor was fabricated from a combination of commercially available and custom components. The final product is shown in Figure 5-3, and a schematic and bill of materials for the final design is shown in Figure 5-4. While searching for a compact and reliable method to connect the sensor to the encasement, it was noticed that the SHT75 sensor could fit in the bore of a Swagelok HVCR<sup>®</sup> series fitting. These fittings are compatible with 1/4" VCR<sup>®</sup> fittings, but are used with 3/8" tubing. A machined adapter, the drawing of which is shown in Figure 5-5, was designed to connect the HVCR<sup>®</sup> gland to a high vacuum feedthrough using welded connections. A printed circuit board (PCB), shown in Figure 5-6, was designed to interface the sensor to the feedthrough. A header is soldered to the board so that the sensor can be plugged in after the housing is welded. According to the sensor datasheet, the accuracy of the sensor can be affected by the heat of soldering and should be installed in a header to maintain the rated accuracy. It was thought that welding the housing with the sensor installed would also damage the sensor, so the sensor is installed after the soldering and welding operations.

Attention should be paid to the proper housing assembly sequence, because some components are welded into the assembly and cannot be added or removed after welding. Orientation of the internal circuit components is also critical. While it would be possible to change the external connections if a mistake was made during assembly, this is obviously not practical on a project with multiple encasements that

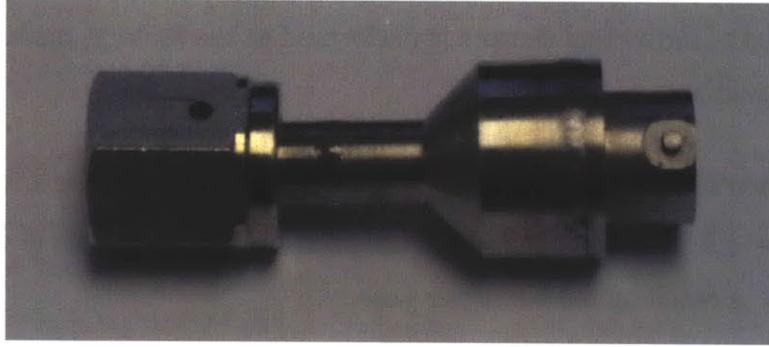


Figure 5-3: Picture of finished hermetically sealed humidity sensor.

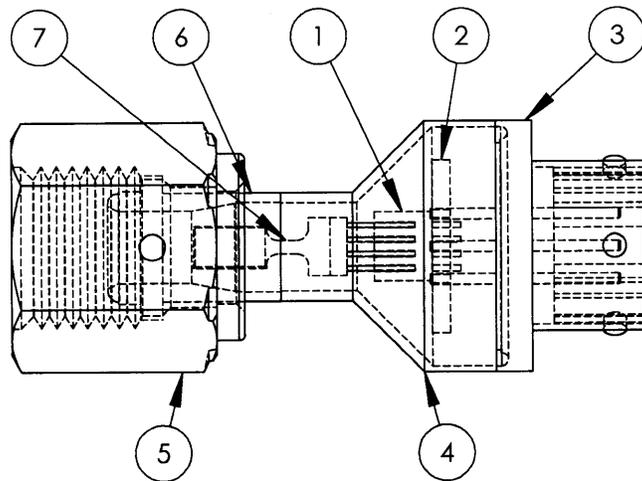
are expected to have interchangeable components. The correct assembly order and orientation is:

1. The header (1) is soldered to the circuit board (2). The traces should be on the component side of the board.
2. The circuit board and header assembly is soldered to the vacuum feedthrough (3). If oriented properly, pins A and D will not connect to the header.
3. The HVCR<sup>®</sup> gland (4) is inserted through the HVCR<sup>®</sup> nut (5) and welded to housing (6).
4. The two subassemblies are then welded together.
5. The humidity sensor is inserted with tweezers. The “hump” of the sensor should face pin A.

## **Welding**

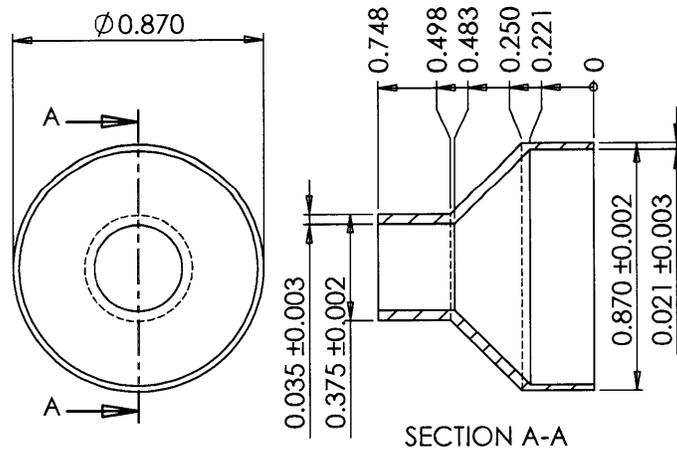
Welding the humidity sensor assembly requires a few special precautions, but can easily be assembled by a competent welder with some experience with vacuum fittings. First, the parts should be completely free of oil. Second, stainless steel must be protected from oxygen on both sides of the weld. Third, the vacuum feedthrough should be welded with a minimum of heat input, or it may develop a leak.

To remove oils from the machining process, the adapter was ultrasonically cleaned in degreaser, rinsed, and dried. This process is described in detail in the Appendix.



Item	Description	Manufacturer	Part Number
1	4 pin female header	Mill-Max	851-43-004-10-002000
2	circuit board	N/A	N/A
3	vacuum feedthrough	Solid Sealing Technology	FA16808
4	housing	N/A	N/A
5	HVCR <sup>®</sup> female nut	Swagelok Company	SS-4-HVCR-1SR
6	HVCR <sup>®</sup> gland	Swagelok Company	6LV-4-HVCR-3-.60SR
7	Humidity sensor	Sensirion AG	SHT75

Figure 5-4: Assembly drawing and BOM for the hermetically sealed humidity sensor design.



UNLESS OTHERWISE SPECIFIED:  
 DIMENSIONS ARE IN INCHES  
 TOLERANCE:  $\pm 0.005$   
 MATERIAL: 316L

Figure 5-5: Drawing of the adapter used to connect the HVCR<sup>®</sup> fitting to a high vacuum feedthrough.

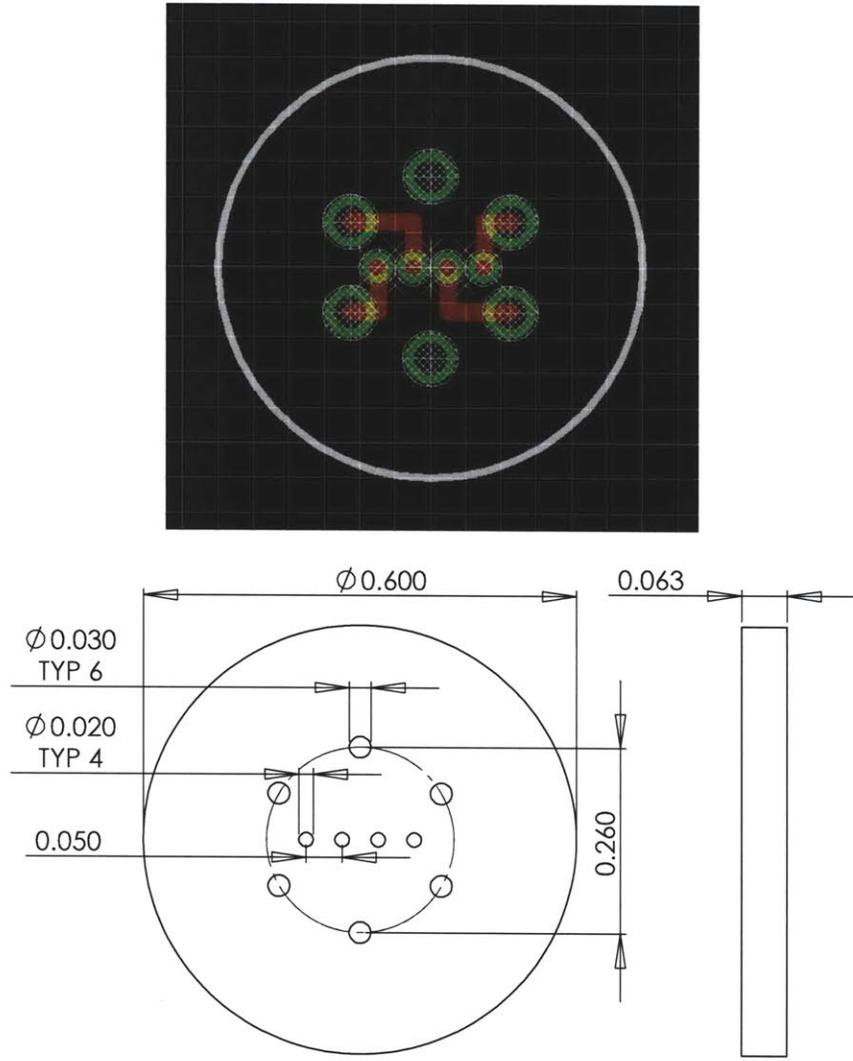


Figure 5-6: EAGLE circuit board artwork and mechanical drawing. The traces (in red) are on the component side of the board.

The vacuum feedthrough and Swagelok gland and nut come individually cleaned and bagged, and do not require any preparation before welding.

Protection from oxidation is best accomplished by flooding the inside of the sensor with inert gas while welding. Taken a step further, the purge can be used to create a slight positive pressure inside the components being welded to ensure that no oxygen can get in through leaks in the system, including the joint being welded. However, at a fixed flow rate, the pressure tends to increase as the weld is completed, because gas can no longer escape the joint. The increased pressure can cause the weld to blow out, leaving a hole that is difficult to fix. Using the “Moisturematic,” described in Chapter 6, to backpurge welds can eliminate the blowout problem because of its built-in low pressure relief valve.

To use the Moisturematic for backpurging, the relief valve should be set to the minimum setting ( $\sim 3$ ” W.C.). The saturator should be bypassed to ensure the purge gas is dry; the outlet hose of the pressure controller will screw directly onto the inlet of needle valve on top of the saturator. The hose normally used to supply argon to an encasement is connected to the assembly being welded, and a restrictor ( $\sim .060$ ” orifice) is placed on the other end of the assembly so that a positive pressure is created within the assembly. The Moisturematic’s needle valve is then opened until the pressure gauge indicates the relief valve setting. As the assembly is welded and the resistance of the joint increases, the relief valve will bleed off excess volume to prevent the pressure from rising.

Back purging generally dictates the order in which stainless steel assemblies will be welded. In the case of the humidity sensor, it is easiest to weld the HVCR<sup>®</sup> fitting to the adapter first, and place a plug (with the bleed orifice, of course) in the wide end of the adapter.

Heat input can be minimized with good fitup and proper welding technique. The edges to be welded should be flat and mate with no visible gap. The assembly can then be welded without filler, decreasing the amount of material to be melted and increasing travel speed.

Welds shrink as they cool, which can cause gaps to open on the side of an assembly

opposite the weld. To ensure that the gap remains closed, the assembly should be tack welded in several places before the seam is fully welded. Then short sections (~1/4") should be welded. Between welds, the assembly can be cooled off with a clean rag dampened with alcohol.

The housing were manually welded with the following parameters: GTAW process, 1/16" sharp tungsten, pulsed DC, 200 Hz, 50 % duty cycle, 25 % background current. The 0.020" thick section was welded with maximum peak current set to 30 amps, and the 0.035" section was welded at 40 amps. On most GTAW machines, the operator has the ability to reduce current with a foot pedal. The housings were helium leak checked after welding. None had a detectable leak.

### **Interface circuit**

The SHT75 sensor has a proprietary two-wire serial interface, making communication with the device somewhat difficult. An evaluation kit that interfaces the sensor to a RS232 port is available from the manufacturer, but it is not cost effective at over \$500 dollars for the kit. In a typical application, a microcontroller is used to bit-bang the proprietary interface. Example code is available from the manufacturer and other sources.

“Bit banging” the protocol using the USB-6008 digital ports was considered, but was ruled out due to latency issues. It was planned that a custom circuit board would be designed to interface the sensor to a USB port. A simpler and cheaper solution was found in the DLP Design DLP-232PC Miniature USB Microcontroller Module, shown in Figure 5-7. This board combines an FTDI FT232RQ USB UART and a Microchip PIC18F2410. The FT232RQ allows devices with legacy serial ports to communicate with a host over USB using either “Virtual COM Port” (VCP) drivers that expose a COM port on the host, or through direct calls to the FTDI driver (D2XX).

Note the 5 pin header opposite the USB connector in the photograph of the DLP-232PC; this is the PIC programming interface. It was noticed that this interface also had the appropriate signal and power connections to communicate with the SHT75. If the humidity sensor is connected to this interface, firmware can be loaded through

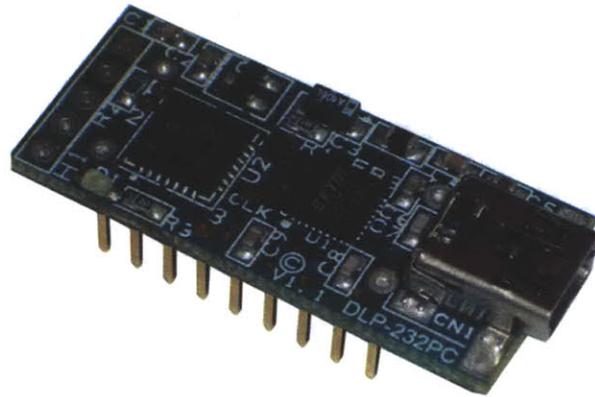


Figure 5-7: DLP-232PC used for interfacing the Sensirion SHT75 humidity sensor to USB

Table 5.1: Connections between DLP-232PC and Sensirion SHT75.

PIC pin function	DLP-232PC pin #	wire color	connector pin letter	SHT75 pin #
VPP	1	green	A	not used
RB6/PGC	2	orange	F	DATA (4)
VDD	3	red	B	VDD (2)
GND	4	black	E	GND (3)
RB7/PGD	5	blue	C	SCK (1)

the sensor cable. Of the PIC interface's 5 wires, the SHT75 only requires 4. The 5th wire is connected to the PIC's VPP/MCLR pin. This connection needs to be brought out to the connector for programming, but is not used to communicate with the sensor. Fortunately, the vacuum feedthrough has 6 pins, which allow the VPP pin to be accommodated. Table 5.1 indicates how the connections are made between the PIC and the SHT75.

In this revision of the sensor and interface box, the PIC programming clock is connected to the SHT75 data line, and the PIC programming data line is connected to the SHT75 clock. These connections must not be reversed, even though the PIC's PGC and PGD connections are for general purpose (when not programming) and can usually be used interchangeably. The reason for this arrangement is that the SHT75

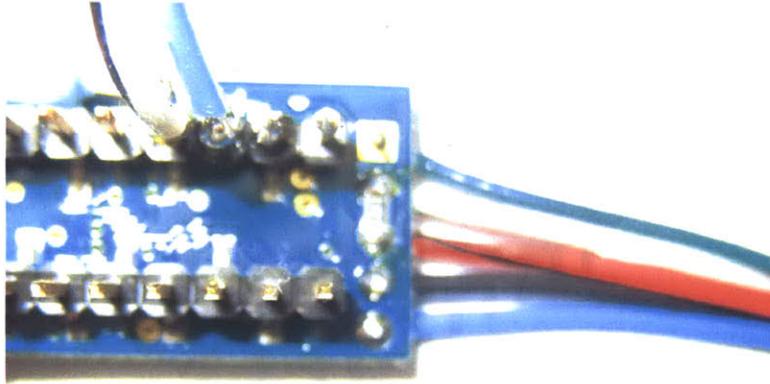


Figure 5-8: Pullup resistor installed between SHT75 VDD and DATA pins.

requires a pullup resistor on the data line. Either the PIC or the SHT75 may pull the line low, so the SHT75 DATA line must not be driven high. This pullup could interfere with programming if it were on the PIC's PGD line. Microchip does not recommend a pullup on either PGC or PGD, because the Microchip ICD2 programmer has 5.6 k $\Omega$  pulldown resistors on both lines. In practice one can get away with weak pullups, especially on PGC, which appears to be driven both high and low by the programmer. As long as the PIC can be programmed and verified, this pullup will not interfere with normal operation. The PGC line is next to VDD, so a 10 k $\Omega$  0805 package resistor was soldered directly between the PGC and VDD pins. This modification is shown in Figure 5-8.

In future revisions, the possibility of interference between the pullup resistor and programming could be eliminated. The pullup resistor can simply be designed into the circuit board installed in the hermetic sensor housing. When the cable is disconnected from the sensor, the pullup would also be disconnected. The sensor housings were already fabricated when the problem was noticed, and it would have been difficult and time consuming to cut open the housings, install new circuit boards, and reweld the housings.

With the programming connection being used for all external connections, the male header on the bottom of the board was no longer necessary. This header was desoldered so that the DLP-232PC board could fit into a thinner case. The DLP-

232PC was then glued to a sheet of Delrin laser cut to fit inside the Hammond Industries 1551FBK enclosure. A sheet of 3M™ VHB™ (very high bond) double sided tape was cut to fit the bottom of the enclosure, which is used to fasten the assembly to the encasement. A picture of the completed assembly (with lid off) and BOM is shown in Figure 5-9.

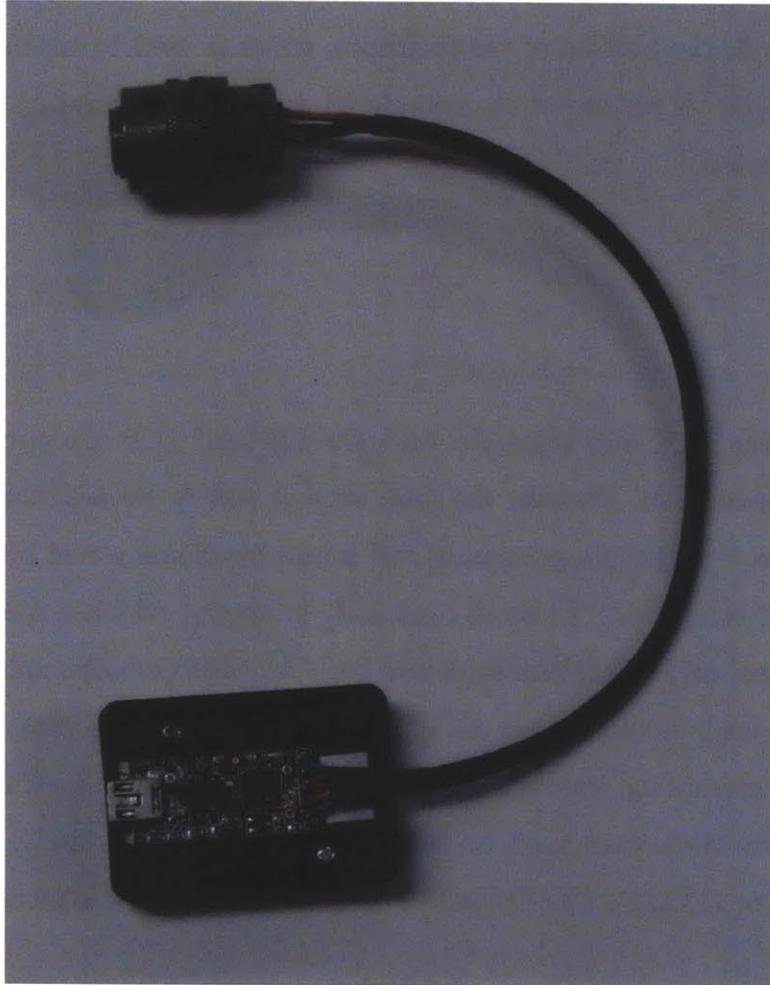
## Software

Code was written in C and compiled with the MPLAB C18 compiler; the code is included in Appendix B. Because the final version was to be programmed through the sensor cable, both the programming and sensor interfaces would not normally be accessible at the same time. To avoid constantly swapping cables, a special version of the humidity data acquisition hardware was built for code development. This version had an additional humidity sensor cable connected to alternate pins of the device, freeing the programming interface for exclusive use by the programmer. #IFDEF preprocessor directives were used to route the signals appropriately, depending of whether DEBUG or RELEASE was defined. This development tool is pictured in Figure 5-10.

The microcontroller also has several configuration bits that may need to be changed from their default settings. In particular, there are two settings that are dictated by the DLP-232PC board design:

1. Low voltage programming must be disabled, because the PGM pin is left floating on the DLP-232PC board. If a logic high is detected, the PIC will enter programming mode.
2. The oscillator selection should be set to 'EC', because the FT232RQ provides a 24MHz external clock. Lower speed clocks are also available.

Communication with the microcontroller can be established using either the VCP or D2XX driver. In either case, the FT232RQ should be set to communicate with the



Description	Manufacturer	Supplier	Part Number
connector, MIL-C-26482, 6POS	Amphenol	Digikey	MS3116E-10-6S
USB/microcontroller module	DLP Design	Digikey	DLP-232PC
10 k $\Omega$ 0805 resistor	Panasonic	Digikey	ERJ-6ENF1002V
extruded aluminum enclosure	Hammond	Digikey	1551FBK
mounting board for DLP-232PC	N/A	N/A	N/A
screws, #2 – 32 $\times$ 3/16"	unknown	McMaster	90056A075
wire, 22 AWG, irradiated PVC	unknown	McMaster	various
heat hrink tubing (3/16" ID $\times$ 7" L)	unknown	McMaster	7864K22
heat shrink tubing (1/4" ID $\times$ 1/4" L)	unknown	McMaster	74965K53
USB cable, RA A to Mini B, 12"	unknown	usbgear.com	USBG-1FABM
VHB™ double sided tape	3M	McMaster	7170A25

Figure 5-9: BOM for humidity sensor interface box and picture of finished assembly (cover removed).

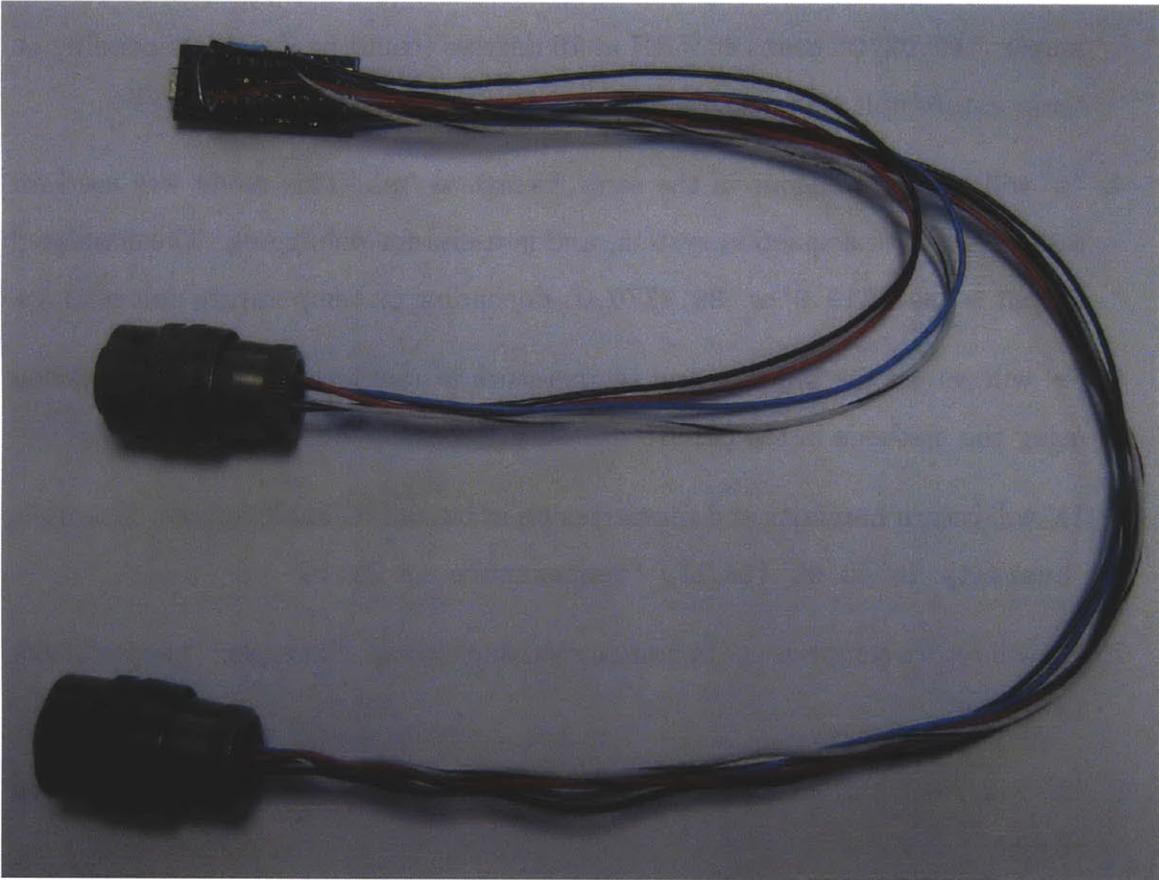


Figure 5-10: Development tool for humidity data acquisition with alternate connections to humidity sensor.

PIC at 19200 baud, with 8 data bits, no parity, and 1 stop bit. After communication is established with microcontroller, the following commands can be used:

1. 'F' will set the temperature units to Fahrenheit (default).
2. 'C' will set the temperature units to Celsius. If power is interrupted, the default will be restored.
3. 'm' will return the humidity and temperature in an easily parsed format. Example: '40.0%70' means 40 %RH at 70 degrees (could be C or F, depending on temperature unit settings).
4. 's' will simulate output in the same format as 'm'. This mode was used for testing the data acquisition system, and is useful for debugging. The simulated output is '39.7%19.9' or '39.7%70.0', depending on temperature unit settings.
5. 'r' will return the humidity and temperature in raw format (before conversion using the methods in the SHT75 datasheet). Example: '6000%1200'
6. 'h' will return humidity and temperature in human readable format. Example: 'humidity is 40.0%' [CR/LF] 'temperature is 21.1C'
7. 't' will return temperature in human readable format. Example: 'temperature is 21.1C'
8. 'R' will reset the serial connection of the SHT75 and return 'connection reset'.

### **Changes for future design revisions**

Humidity sensing is thought to be a very successful component of the Massachusetts Archives instrumentation. Like most designs, however, there is still room for improvement. To recap the design changes suggested above:

1. The resistor should be on the humidity sensor circuit board, so that there is no chance that it could interfere with programming. A stronger pullup could then be used, and communication speed could be increased.

2. The board artwork should be revised to include orientation markings, which would decrease the chance of assembly errors.

#### 5.1.4 Acquisition hardware

The data acquisition hardware consists of a Lantronix UBox<sup>®</sup> 2100 Ethernet/USB bridge, a National Instruments USB-6008 OEM data acquisition board, and a custom printed circuit board (PCB) with various connectors on it. The PCB's function is to route signals appropriately to simplify wiring, and also serves to mount the Lantronix and National Instruments components inside an enclosure. Because the PCB contains no active components, it is few failure points and can be easily repaired if necessary.

The pressure sensors requires a power supply of 12 – 36 VDC, while the USB components require a 5 VDC supply. The dual voltage requirements imposed by this sensor limits the selection of suitable power supplies. An Astrodyne AMP5002-03 power supply was found to meet the voltage requirements and exceed the minimum current requirements. The Astrodyne AMP3002-03 would have been a better match for the current requirements, but was out of stock. Both Astrodyne power supplies have a 5 pin DIN connector outputting 5 VDC and 24 VDC.

The acquisition hardware has several external connections: 4 analog inputs (each also provides +24 volt power and ground to the sensors), a USB port for the humidity sensor, a DIN connector for connection to a power supply, and a panel mount Ethernet patch cable, which connects internally to the UBox<sup>®</sup>. Internal connections include USB jumpers from the UBox<sup>®</sup> ports to the USB-6008 and the external humidity sensor USB connection and a +5 volt power connection for the UBo<sup>®</sup>. The USB-6008 is powered by the UBox<sup>®</sup> over USB. The acquisition hardware is contained in an extruded enclosure manufactured by Hammond Industries, P/N 1455T2201. An extruded enclosure was chosen because the opposite faces of the of the enclosure are parallel, making it easy to machine and mount. Molded plastic and die cast aluminum enclosures have a draft angle, making them more difficult to machine. Brackets were designed to allow the assembly to securely clamp onto the encasement stands, which are made from 1.5" square steel tubing. Top and end views of the acquisition hardware

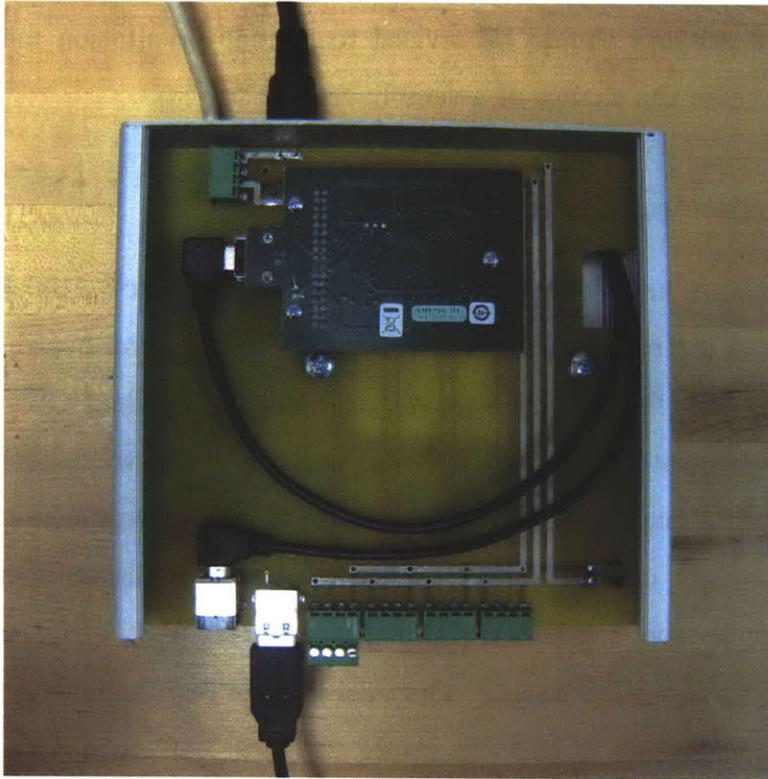


Figure 5-11: Top view of assembled instrumentation box

are shown in Figures 5-11 and 5-12.

### **Hardware construction**

This section contains the Bills of Materials and instructions for assembling instrumentation as applied to the Massachusetts Archives encasements. It should be noted that the QTY column is the number of pieces needed to assemble a unit for one encasement. Package quantities for small parts such as fasteners and wiring supplies can

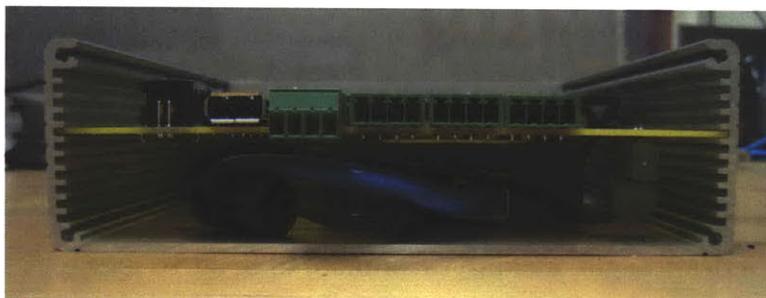


Figure 5-12: End view of assembled instrumentation box

be significantly more than needed; in many cases, one package was enough to finish all five encasements. If ordering spares, the required quantity should be evaluated.

The first step is to install soldered components on the PCB. The bill of materials for this step is listed in Table 5.2. The components include various connectors and a cable for providing power to the UBox<sup>®</sup>. The power cable is cut to 8” in length, stripped, and tinned. When soldering the power cable to the board, polarity must be observed. It is also important to be careful of the orientation and placement of the connectors. The DIN connector and power cable are on the “bottom” side of the board, while all the other connectors are on the “top” side. The 34 position connector is also keyed; the key must face towards the middle of the board.

Table 5.2: BOM for soldered components (1st PCB operation)

QTY	Component Description	Manufacturer	Supplier	Part No.
1	custom PCB	Advanced Circuits	Advanced Circuits	N/A
1	power cable, 2.1 mm	Tensility Intl.	Digikey	CA-2191
1	34 POS boardmount socket	3M	Allied	6834-4500PL
1	USB A connector	Tyco Electronics	Digikey	292303-1
1	USB B connector	Tyco Electronics	Digikey	292304-1
4	receptacle, 4 POS, 3.81 mm	Tyco Electronics	Digikey	284513-4
1	DIN receptacle, 5 POS	Tyco Electronics	Digikey	5212044-1

Next, the bolt-on components are installed on the PCB. The BOM for this step is listed in Table 5.3. The PCB acts as structure to mount the UBox<sup>®</sup> and NI board; the whole assembly slides into the enclosure. The Lantronix box is held on with #10-32 hardware and special washers to prevent the mounting tabs from bending. These washers were laser cut from a Delrin sheet. The National Instruments board is held on with #4-40 screws and standoffs. For proper engagement of the 34 pin connector, 7/16” long standoffs were trimmed to 0.415” length. The odd size required was a consequence of mating the 3M 2500 series connector that comes presoldered to the National Instruments board with a 3M 6800 series connector. This combination

results in a lower assembled height than with the 3M 8500 series connector recommended by National Instruments, which would have resulted in interference with the enclosure. 3M considers the 2500 and 6800 series to be compatible [20], but does not publish a recommended standoff height. Instead, the proper standoff height was calculated from the connector drawings. After the components are mounted, the power cable and USB cables should be connected.

Table 5.3: BOM for bolt-on parts (2nd PCB operation)

QTY	Component Description	Manufacturer	Supplier	Part No.
1	Ethernet/USB bridge	Lantronix	Amazon.com	UBox® 2100
1	data acquisition board	Natl. Inst.	Natl. Inst.	USB-6008 OEM
2	USB cable, 1 ft, A-B RA	unknown	usbgear.com	ARBR-0104B
1	panel mount Ethernet jack	DataPro	DataPro	1770-01C
6	#4 – 40 × 7/16” standoff	unknown	McMaster	91780A431
3	#4 – 40 × 1/4” screw	unknown	McMaster	90272A106
2	#10-32 nylock nut	unknown	McMaster	90633A411
2	screw, #10-32x1/2”	unknown	McMaster	90272A829
4	washer, NAS620C10	unknown	McMaster	90945A741
2	washer, 5/8” OD, 3/16” ID, 3/32” T	N/A	N/A	N/A

The enclosure requires a significant amount of machine work to prepare it to receive the assembled PCB. The enclosure and lid are shortened to 6” length, the endcaps are machined to have the appropriate connector cutouts, and the brackets which clamp the electronics to the encasement stand are machined. Because the PCB limits access to the screws that attach the brackets to the enclosure, the brackets should be bolted on before the PCB is installed. After the enclosure is prepared, the PCB is loaded into the enclosure and the lid and endcaps are installed. The BOM for the enclosure is listed in Table 5.4. Drawings of the enclosure components are shown in Figures 5-13 through 5.1.4.

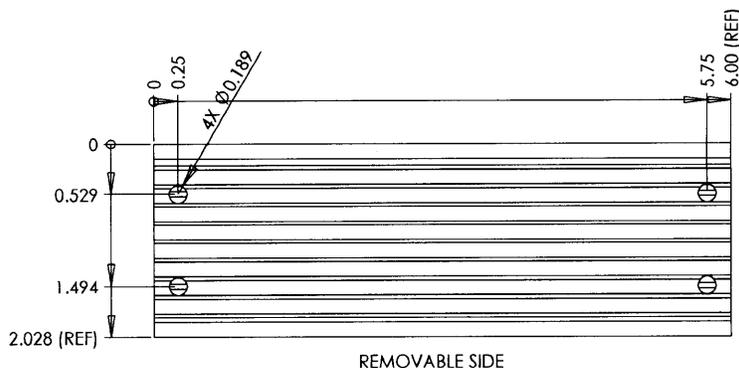


Figure 5-13: Instrument enclosure drawing.

Table 5.4: BOM for instrument enclosure

QTY	Component Description	Manufacturer	Supplier	Part No.
1	enclosure	Hammond Industries	Allied Electronics	1455T2201
2	bracket, fixed side	N/A	N/A	N/A
2	bracket, movable side	N/A	N/A	N/A
4	screw, #10 – 32 × 5/8”	unknown	McMaster	92185A971
4	washer, NAS620C10	unknown	McMaster	90945A741
2	screw, 1/4” – 20 × 1 – 3/4”	unknown	McMaster	92196A548
2	washer, NAS620C416L	unknown	McMaster	90945A760

The instrument boxes should be assembled as shown in Figures 5-11 and 5-12. They are then ready to be installed on the encasements. The supplies needed for installation are listed in Table 5.5.

When the instruments were installed at the Massachusetts Archives, cable ties, adhesive backed cable tie mounts, and heat shrink tubing were used to keep the installation neat and orderly. The mating connectors that are used with the pressure sensors are also listed in the BOM; these should be installed as shown in Figure 5-2. The power supplies were attached to the encasement stands with VHB™ tape, since the power supply does not have a long enough cable for the “brick” to rest on the floor. A picture of the completed installation is shown in Figure 5-16.

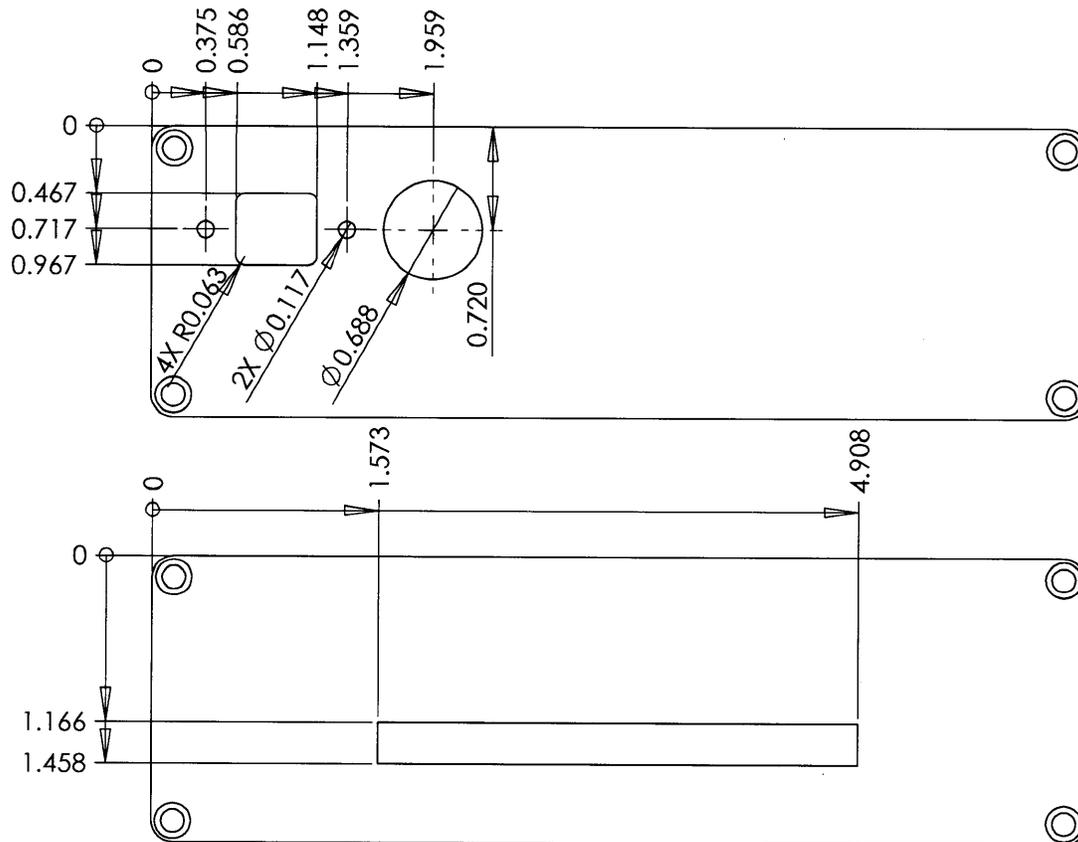


Figure 5-14: Enclosure endcaps drawing.

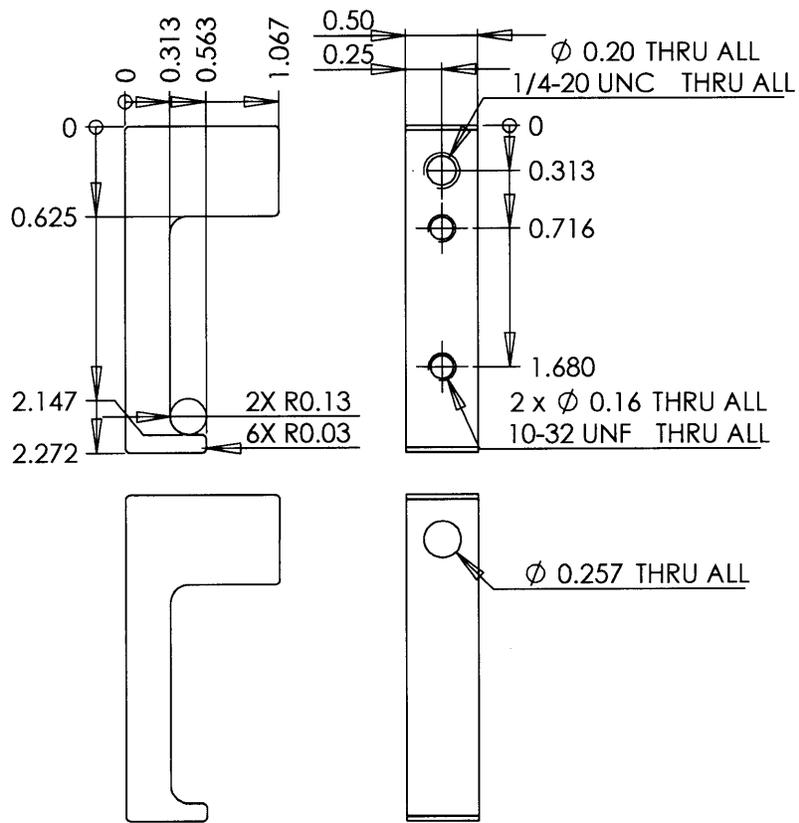


Figure 5-15: Enclosure bracket drawing. Note that two parts are shown (with same outline, but different holes); two of each are required for one assembly.

Table 5.5: BOM for instrument installation supplies.

QTY	Component Description	Manufacturer	Supplier	Part No.
1	power supply	Astrodyne	Astrodyne	AMP5002-03
1	power cord, 3'	unknown	Amazon.com	B00007JO37
3	plug, 4POS, 3.81 mm	Tyco Electronics	Digikey	284507-4
~ 15	cable ties	unknown	McMaster	7130K52
~ 15	cable tie mounts	unknown	McMaster	7582K11
1	heat shrink tubing	unknown	McMaster	74965K63
1	VHB™ tape (cut in half)	3M	McMaster	7170A25

### 5.1.5 Software

Data acquisition software was written in LabVIEW. LabVIEW is easy to use and code can be developed quickly. LabVIEW programs are written graphically; programs typically consist of a user interface, known as the “front panel,” and a block diagram that controls the flow of data. The front panel for the Massachusetts Archives data acquisition system is shown in Figure 5-17. A stripped down block diagram (reduced due to space constraints) is shown in Figure 5-18. This diagram shows the acquisition and logging portions of the software for a single encasement. Not shown is the graphing portion, which displays the previous 24 hours of temperature and relative humidity data for all five encasements. Pressures are not graphed, because they are not expected to change quickly.

The LabVIEW functions used will be familiar to those that have used LabVIEW before. To summarize the functions of the code: pressure information is acquired from analog to digital converters on the USB-6008 boards, and temperature and relative humidity data is acquired by the block marked “HUMD”. The data is recorded to a file using the “Write to Measurement File” function. A time and date stamp is written along with each line of data. All of this is contained in a conditional loop that executes

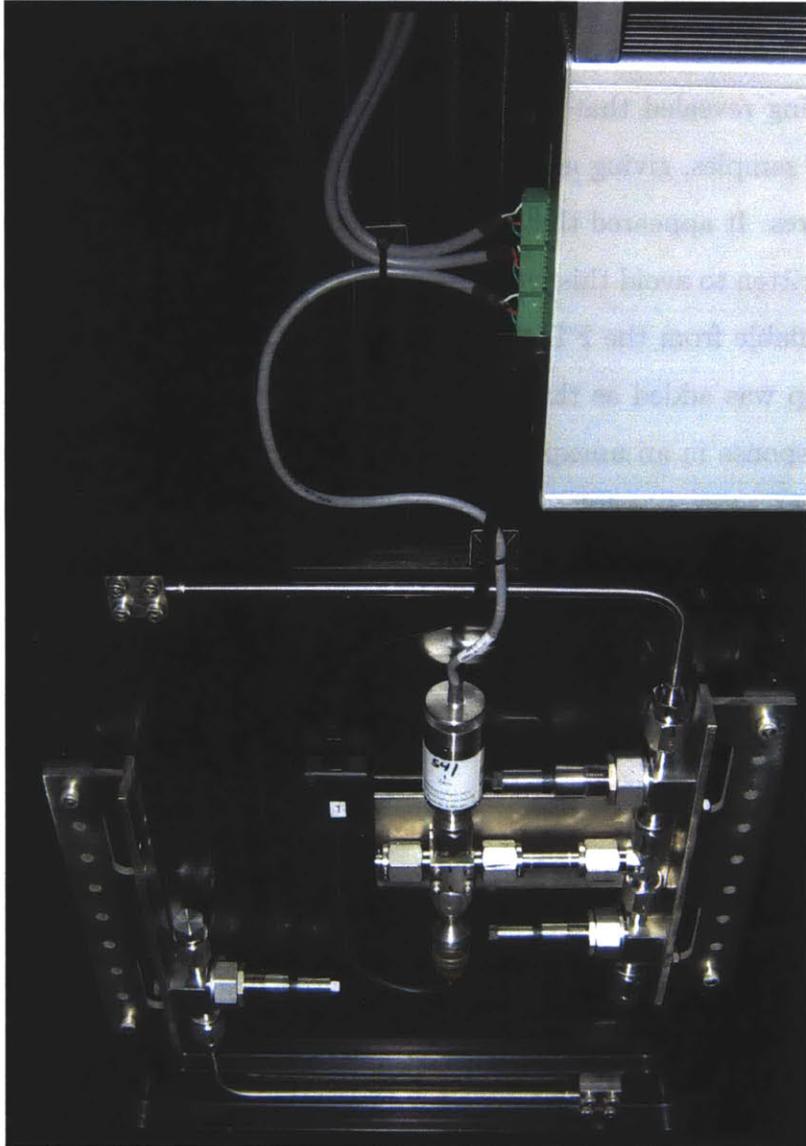


Figure 5-16: Completed instrument installation.

once every 12 times the outer timed loop executes, which occurs every 5 seconds. This results in samples being acquired once a minute. Without the conditional loop and the faster-running timed loop, it could take up to a minute for the code to respond to a request to stop execution.

The “HUMD” block requires a discussion of its own. Originally, the “Instrument I/O Assistant” was used to communicate with the humidity sensors using the VCP drivers. Testing revealed that the software would consistently freeze after approximately 15000 samples, giving about 10 days of run time, an unacceptably short time between failures. It appeared that the Instrument I/O Assistant was hanging, so the code was rewritten to avoid this module. A sample LabVIEW VI that uses the D2XX drivers is available from the FTDI website. This example was turned into a subVI. A parsing step was added as the last frame in the sequence, if it receives an empty string or a response in an unexpected format, an error is generated. The outer loop will exit if data appears valid, and will retry up to three times if it does not. This error is not latched; it will attempt three more times if the subVI is called again. The block diagram for this subVI is shown in 5-19. Changing from the VCP to the D2XX greatly improved reliability.

### **5.1.6 System preparation**

Several steps are necessary to configure the computer used for data acquisition. These steps are outlined below.

#### **Hardware setup**

It is possible to start by connecting the UBoxes and USB devices. For some USB devices, installing the hardware before the drivers would not be advisable, but the UBoxes won't connect USB devices to the host before the UBox<sup>®</sup> software is installed.

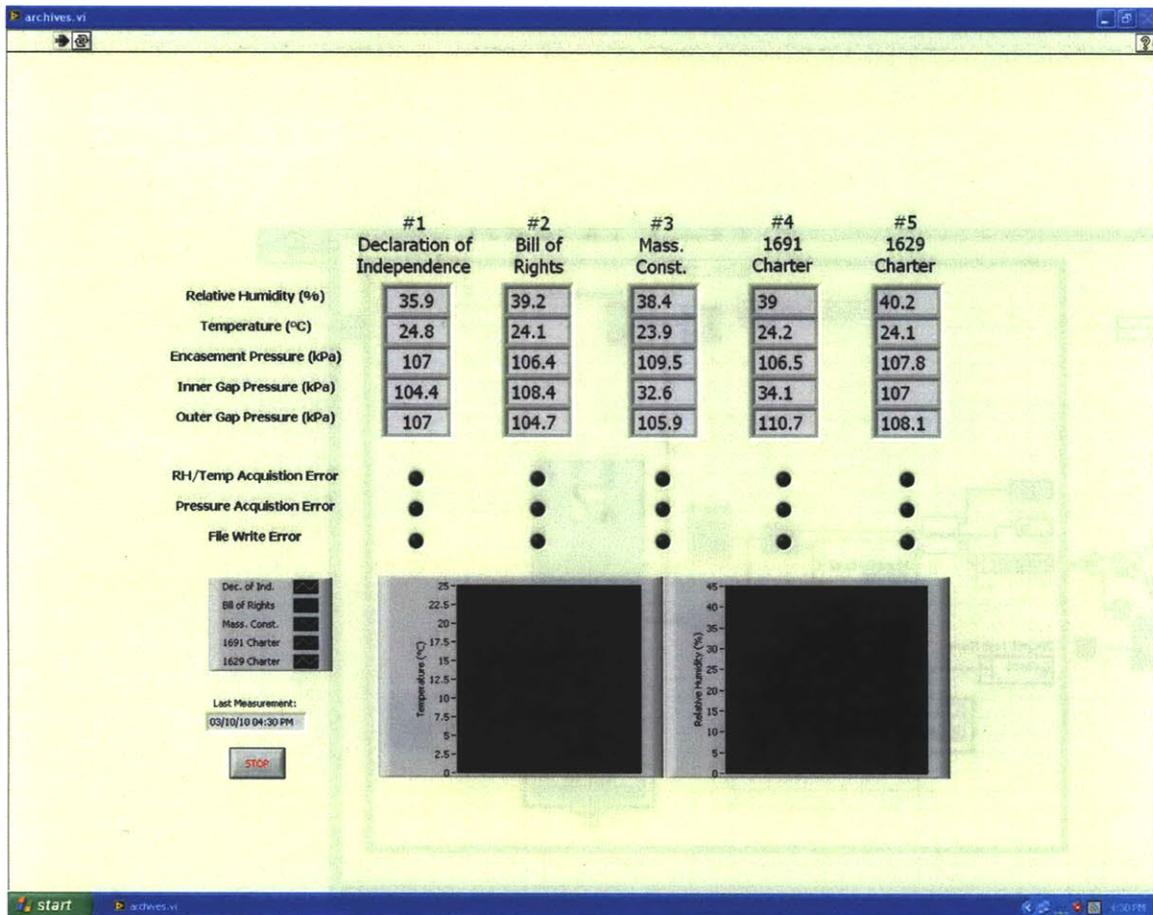


Figure 5-17: Data acquisition front panel.



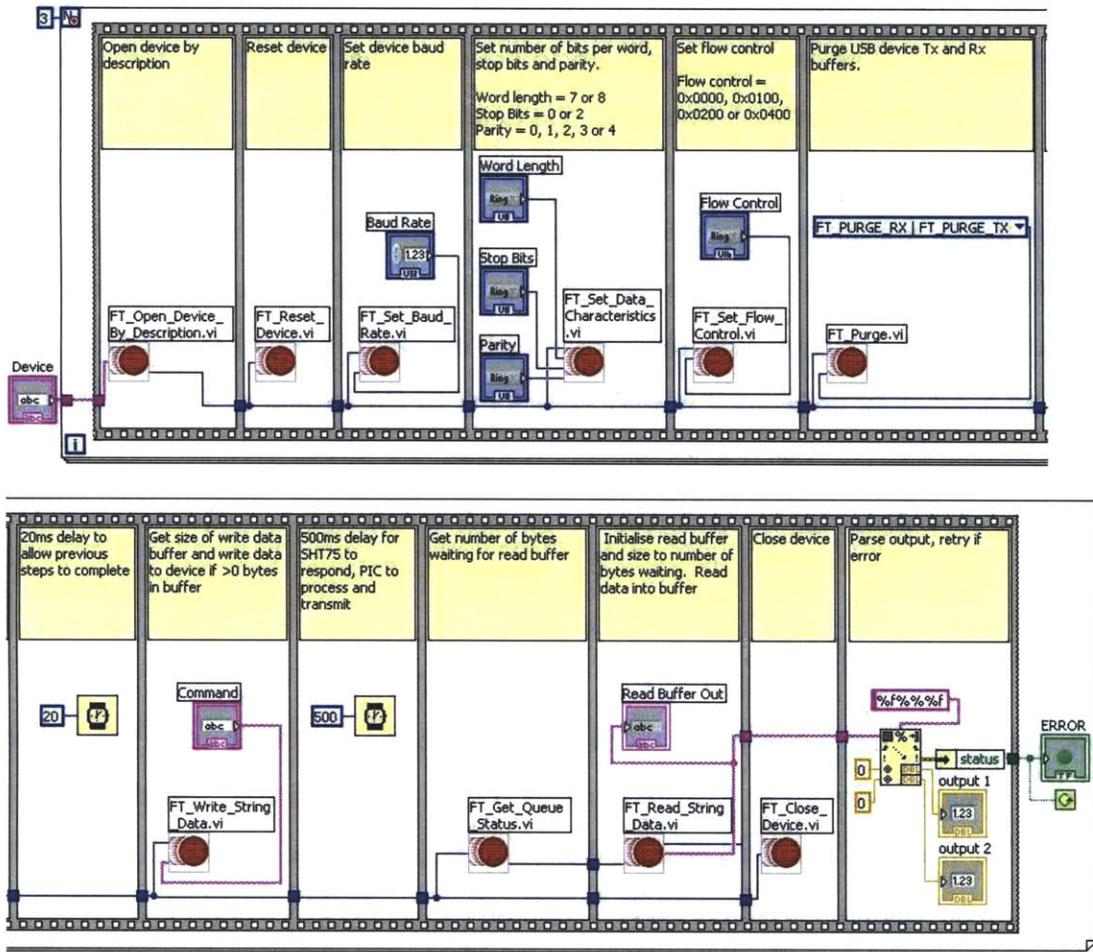


Figure 5-19: Modified D2XX driver subVI example.

Table 5.6: Network configuration for host and UBoxes.

Device	IP address	subnet mask	gateway
Host	192.168.0.1	255.255.255.0	none
UBoxes	192.168.0.11-192.168.0.15	255.255.255.0	192.168.0.1

### UBox<sup>®</sup> software and network setup

Next, the UBox<sup>®</sup> software should be installed. Refer to the UBox<sup>®</sup> manual for full instructions. The essential steps are to (1) open the firewall to TCP and UDP traffic on the correct port, (2) configure the host network adapter, and (3) configure the UBox<sup>®</sup> network settings. Static IP addresses were used to aid in identifying which UBoxes are connected, which is especially useful during development work. Windows, however, will recognize the USB devices by serial number, so one of the alternate UBox<sup>®</sup> configuration methods could be used. Note that the host would also need a gateway assigned if it were connected to the Internet. The settings used for the Massachusetts Archives are shown in 5.6.

### DLP-232PC board setup

Next, the DLP-232PC boards should be connected one at a time with the UBox<sup>®</sup> software. Windows will ask for the location of the correct driver, which should be downloaded from FTDI. The current FTDI drivers combine the VCP and D2XX drivers into a “Combined Driver Model” (CDM). This allows communication using a COM port or direct calls to the FTDI drivers.

The FT232RQ has several options that need to be configured if multiple humidity sensors will be connected to a single host. Otherwise, it would not be possible to know which sensor the host is communicating with.. A program can be downloaded from FTDI to configure the device; FT\_PROG is the current utility. A screen capture of this program is shown in Figure 5-20. Note that for the first chip, the product description is the string ‘HUMIDITY 1’ and the serial number is ‘00000001’. Each device was programmed with sequential product descriptions and serial numbers.

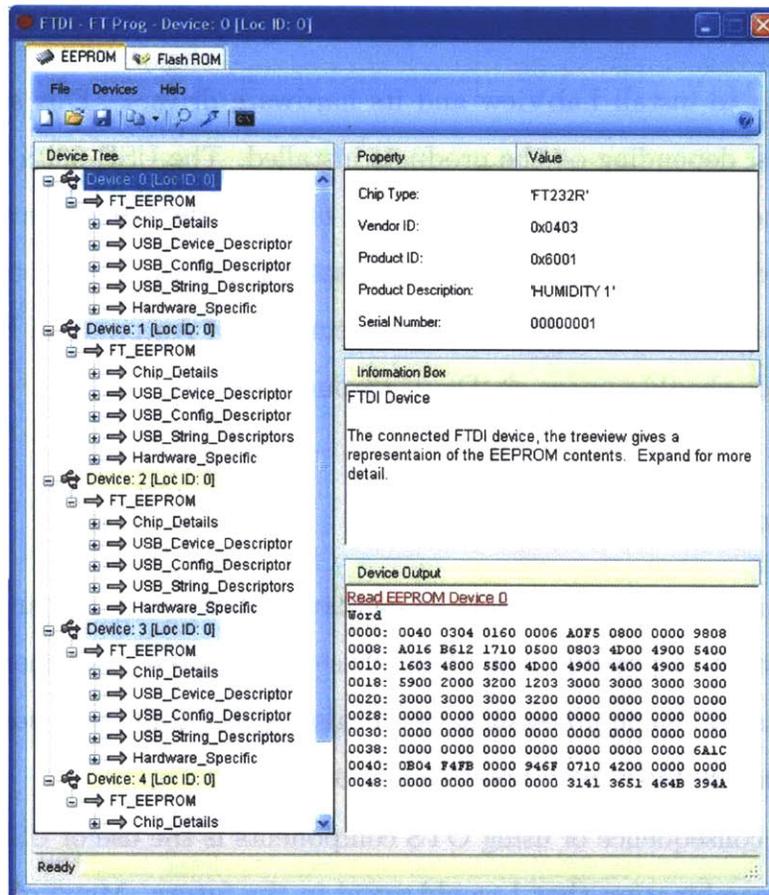


Figure 5-20: Screen capture from the FTDI programming utility, FT.PROG.

Windows uses the serial number to assign a COM port, so these must be unique if VCP drivers are used. Once assigned, the COM port will remain associated with a specific device. If using the D2XX drivers, either the serial number or description can be used to open a specific device.

It may also be desirable to configure Windows so that the COM ports are assigned sequentially if the VCP drivers will be used for debugging or other purposes. For the Massachusetts Archives project, COM11 through COM15 were assigned to the DLP-232PC boards so that the 2nd digit would correspond to the encasement the board is connected to.

## LabView Setup

The final step is to install LabView and its hardware drivers. This step alone can take a few hours, depending on the products installed. The USB-6008 boards should then be connected one at a time, in the desired order. If the UBox<sup>®</sup> is set to ‘shared’ mode for USB devices (default), the UBox<sup>®</sup> software must be used to connect the devices individually. In the LabView “Measurement and Automation Explorer,” the USB-6008 boards should appear as ‘Dev1’ through ‘Dev[n]’.

### 5.1.7 Conclusion

Given the constraint of building a data acquisition system using as many off-the-shelf (OTS) components as possible, the Massachusetts Archives instrumentation meets the requirements. OTS components, however, also heavily influenced some key design decisions that in hindsight, may have been poor.

The biggest consequence of using OTS components is the use of USB over Ethernet, which is a combination that I would avoid in the future. Many of the problems initially experienced were resolved by updating the UBox<sup>®</sup> driver and firmware update, but some minor glitches continued. If USB devices must be used, USB extenders that utilize Ethernet cable (but not the protocol) might be a safer, though inelegant, solution. The decision to use USB over Ethernet was driven by the selection of the USB-6008 data acquisition module, which was based on its ability to handle  $\pm 10$  V signals. Using a pressure sensor with a 5 V analog output or a digital output would have affected component selection throughout the chain. The use of 5 V signals enables much more flexibility in component selection.

Even though the system was constructed almost entirely from off-the-shelf components, some of the components required a significant number of modifications. If one of these components fails, a substantial amount of technical skill is required to diagnose problems and perform repairs. Since ease of repair was originally used to justify the use of OTS components, this requirement should perhaps be reevaluated. Circuit boards can easily be designed with future repairs in mind; for example, del-

icate components can be socketed and test points can be provided to help diagnose problems. In the future, certain OTS components may become unavailable, while a competent technician would have no trouble repairing a well-documented custom circuit board. To illustrate this point, the manufacturer of the UBox<sup>®</sup> has released a product discontinuation notice between the time the UBox<sup>®</sup> was selected and the time this thesis was written. Furthermore, a custom solution would likely cost less, certainly in parts and likely in labor. With OTS parts, design time is largely spent sourcing components and attempting to fit them together physically and electrically, which is made much harder if space constraints are imposed.

Designing a custom datalogging solution would also provide some other benefits. First, such a system could be designed to fit in a much smaller space; the electronics could be made to fit within the envelope of the encasement and wires could be routed internally to the encasement and planned in CAD, resulting in a much more elegant installation. Second, such a design would likely consume less power, removing a heat source that resides directly below the document. In conclusion, given the opportunity to redo the electronics on the encasements, I would not have been adamant to use OTS electronics.



## Chapter 6

# Humidity Generation

Conservators consider a constant humidity to be essential for preservation of documents. The Massachusetts Archives specified that the gas in the encasements be humidified to 40 %RH at 70.0 °F (21.1 °C), and that the encasements would be filled to approximately atmospheric pressure, 101.4 kPa at sea level.

The temperature had to be specified in addition to relative humidity because relative humidity is temperature dependent. Isobarically cooling or heating a parcel of gas starting with the conditions prescribed above will significantly alter the relative humidity. At 6.8 °C, that parcel would have a relative humidity of 100 %, and water would begin to condense. This temperature is known as the dew point.

Changing the temperature of a sealed encasement, however, is not an isobaric process. Instead, we must consider the effects of changing the temperature of a gas at fixed volume. Isochorically altering the temperature of a vessel containing humidified gas will affect pressure, relative humidity, and even dew point. The actual mass of the water and dry gas will not change, and thus neither will the number of molecules of each species. The mixture therefore has a constant mixing ratio.

The goal of this chapter is to describe methods to humidify a gas such that it has the correct mixing ratio, and to discuss the design of a machine capable of accurately producing this gas. It is still convenient to work in terms of relative humidity, as can be seen from the equations below. It is important to keep in mind that specifying relative humidity, temperature, and pressure will correspond to a particular mixing

ratio.

## 6.1 Humidity generation methods

There are many methods of generating a gas of known humidity. Sections 6.1.1 through 6.1.3 will describe three practical approaches and their strengths and weaknesses. The methods below all involve saturating a gas stream with water, and then either diluting the saturated stream, or manipulating temperature or pressure to reduce the relative humidity. Another method in common use is to pass a stream of gas over a saturated salt solution. The resulting humidity is dependent on the type of salt and the temperature of the solution. This method is often used to calibrate humidity sensors, as it can generate a known relative humidity, but not an arbitrary value of relative humidity. The methods suggested below can humidify a gas stream with an arbitrary value of relative humidity.

### 6.1.1 Dilution method

Conceptually, this method is the simplest to describe, analyze, and implement; saturated gas is simply diluted with a dry gas. For example, if 50% RH gas is desired, then dry gas could be split into two streams of equal flow rate. One stream is then saturated, and the streams are then recombined. While this method was used successfully for filling the Waldseemüller map encasement and some commercial humidity generator designs rely on this method, it has a few drawbacks:

- Typical rotameter accuracy is 2% of full scale, and two rotameters are needed which compounds error.
- The ratio of saturated to dry gas is controlled open-loop and may vary due to disturbances such as downstream or upstream pressure.
- The saturation pressure of water, and thus humidity, has a strong temperature dependence. Temperature, therefore, should be controlled.

None of the commercial systems surveyed close the control loop on flow rates or temperatures, and thus are limited in accuracy. Certainly more complex systems could address these shortcomings; using mass flow controllers in place of rotameters would address the flow accuracy and open-loop control issues, and temperature of the saturator could also be controlled with additional equipment. Controlling all of these variables, however, would likely result in a greater system cost than if using one of the other suggested methods.

### 6.1.2 Two temperature method

Another conceptually simple approach is to saturate a gas at a reduced temperature; the gas will then have a lower humidity when warmed to room temperature. The equation

$$RH = \frac{e_w(T_s)}{e_w(T_c)} \quad (6.1)$$

describes the situation where where a gas is saturated at some temperature  $T_s$  and is then heated isobarically to a temperature  $T_c$ .  $e_w$  is the saturation pressure of water as a function of temperature. This equation is a simplified version of equation 6.2, and neglects the small effect of the enhancement factor, described in section 6.1.3. 40% relative humidity at  $T_c = 21.1^\circ\text{C}$  corresponds to  $T_s = 6.8^\circ\text{C}$ , which will be the dew point of the gas.

### 6.1.3 Two pressure/two temperature method

A natural extension to the two temperature method is to include the effect of pressure change on a humidified gas as well. This effect is strong and can be used to generate wet gas over a wide range of humidities. The relative humidity of a gas saturated at one temperature and pressure and then expanded and heated or cooled is given by [21]

$$RH = \frac{f(P_s, T_s)}{f(P_c, T_c)} \times \frac{e_w(T_s)}{e_w(T_c)} \times \frac{P_c}{P_s}. \quad (6.2)$$

where  $e_w$  is the saturation pressure of water, and  $T$  and  $P$  are the temperatures and pressures of the saturation vessel or in the encasement, denoted with  $s$  and  $c$  subscripts respectively. For the purposes of this thesis,  $P_c = 102.6$  kPa and  $T_c = 21.1$  °C.

Thus, expanding a saturated gas will lower its relative humidity, and increasing the temperature of a humidified gas will lower its relative humidity. The enhancement factor  $f$  describes a weak interaction between species that results in slight changes in relative humidity as a result of temperature and pressure changes.  $f$  relates the partial pressure of a saturated gas to the saturation pressure of water alone. Over a wide range of temperatures and pressures, the value of  $f$  is close to unity [22].

The saturation pressures  $e_w$  can be found from various thermodynamic tables or calculated from any of several representations. For this work, IAPWS-IF97 equations were used; this representation was chosen because the equations were available at no charge in the form of Excel macros [23], which made the incorporation of the equations into a spreadsheet straightforward.

For many applications,  $RH$ ,  $P_c$  and  $T_c$  will be constrained by the process. For example, preservation experts associated with Massachusetts Archives specified that the encasements be filled with a gas having a relative humidity of 40 % at an encasement pressure  $P_c = 102.6$  kPa and encasement temperature  $T_c = 21.1$  °C. Another constraint is that condensation must be avoided. After the gas passes through the saturation vessel, the relative humidity must be lowered by reducing the pressure and/or increasing the temperature. To minimize startup time as well as risk of condensation, it may be desirable to keep  $T_s$  close to  $T_c$ . Therefore, a logical constraint is that  $P_c \approx P_s$ .

Determining the enhancement factor is not as straightforward. One formulation for the enhancement factor of humid air is [22]

$$f(P, T) = 1.00072 + 3.2 \times 10^{-5} \cdot P + 5.9 \times 10^{-9} \cdot P \cdot T. \quad (6.3)$$

where  $T$  is in °C and  $P$  is in kPa.

This equation, however, is invalid for any gas other than air. A review of the

Table 6.1: Parameters suitable for generating humidified gas with a relative humidity of 40 %.

variable	symbol	value
desired humidity	$RH$	40 %
desired temperature	$T_c$	21.1 °C
desired pressure (abs)	$P_c$	102.6 kPa
saturator temperature	$T_s$	25 °C
saturator p.p.H <sub>2</sub> O	$e_w(T_s)$	3.170 kPa
encasement p.p.H <sub>2</sub> O	$e_w(T_c) \cdot RH$	1.001 kPa
pressure ratio	$P_c/P_s$	3.165
saturator pressure (abs)	$P_s$	324.8 kPa

literature did not yield a usable value of  $f$  for humid argon. Most work focuses on extreme pressures or temperatures, where the effect is much more significant. At low pressure, this factor is often sufficiently close to unity that it can be ignored. For example, a saturator held at 25.0 °C and 320 kPa has an enhancement factor of 1.011 and a test chamber held at 21.1 °C and 101.4 kPa has an enhancement factor of 1.004. The combined effects would create an error of 0.7 % if ignored, which would be acceptable for many applications. We expect the effect to be even smaller with argon; this can be seen by comparing the enhancement factor in argon and nitrogen in [24] and [25]. We can conclude that ignoring the enhancement factor will not result in significant error in the relative humidity of a humidified argon mixture. Though we will assume  $f = 1$  for calculating setpoints, the effects of this factor will be included in an error analysis in Section 6.4.6.

Table 6.1 summarizes parameters suitable for humidifying a gas to a relative humidity of 40 % at the desired encasement temperature and pressure. Note that the encasement pressure is slightly higher than atmospheric due to the use of a back pressure regulator in the humidity generator circuit (refer to Section 6.4 for more information).

Table 6.2: Commercially available humidity generators

Manufacturer	Model number	Method
General Eastern	DPG-300	dilution
Xentaur	N/A	dilution
LI-COR	LI-610	two-temperature
Sable Systems	DG-4	two-temperature

## 6.2 Commercial humidity generation equipment

Several humidity generators are commercially available; the dilution method and the two-temperature method are both common. The dilution method tends to be used on simpler machines, as a result none of the commercially available machines found control saturator temperature. One can see from the analysis above that saturator temperature needs to at least be known for accurate humidity generation, and preferably controlled. A further complication is that many commercially available units are not intended for flow-through operation, instead they have a humidity controller chamber for calibration of small instruments.

Table 6.2 lists several commercially available dewpoint generators. This list is probably not exhaustive, and only lists humidity generators with a small footprint ( $< 0.2 \text{ m}^2$ ) and that are intended for flow-through operation. Of these, the dilution method units were considered unsuitable due to the accuracy concerns cited previously. Either of the remaining systems would have required additional hardware to be added to make encasement filling safe and user-friendly. A custom machine based on the two pressure/two temperature method was also evaluated along with the commercial units. Ultimately, the custom machine was determined to offer the best combination of capability, packaging, and cost. This machine is described in Section 6.4, “The Moisturematic.”

### 6.3 Purge parameters

With the decision made to fabricate a device for generating humid argon, some basic machine requirements must be calculated, such as how long a purge should last and how much argon and water will be consumed.

A volume of dry gas will expand in volume at constant pressure and temperature when water vapor is added. The relationship between these volumes must be known because gas is likely to be metered in its dry state, but the flow rate of wet gas figures into determining purge parameters. The flow rates of wet and dry gas can be related with

$$\dot{V}_w = \dot{V}_d \frac{P_s}{(P_s - e_w(T_s))}. \quad (6.4)$$

Note that at room temperature, the flow rates of wet and dry gas are nearly equal, and can be approximated as such when generating humid gas for document preservation purposes. As saturator temperature increases, however,  $e_w(T_s)$  becomes significant compared to  $P_s$ .

The next step in determining how much gas and water is required is to calculate how long purging should last. Assuming perfect mixing of incoming gas, the concentration of oxygen in the encasement while purging is described by the first order ordinary differential equation

$$\frac{V}{\dot{V}_w} \frac{dP_{O_2}}{dt} + P_{O_2} = 0. \quad (6.5)$$

We then expect the oxygen concentration in the encasement to follow

$$P_{O_2}(t) = P_{O_2}(t_0) \cdot (1 - e^{-t(V/\dot{V}_w)}). \quad (6.6)$$

With an encasement volume  $V$  of 40 L and a flow rate  $\dot{V}_w$  of 1.5 L/min, the time constant is about 27 minutes. 8.4 time constants are needed to drive the oxygen concentration inside the encasement below 0.005%, two orders of magnitude less than the 0.5% maximum oxygen concentration specified by conservators. Therefore,

a purge time of 4 hours is appropriate.

Water and gas consumption can then be calculated. The density of water vapor at the temperature of the saturator must be determined; this value can be found using the Excel macros from XSteam. The mass of water consumed in a purge is

$$m_w = \rho_v(T_s) \cdot RH \cdot \dot{V}_w \cdot t. \quad (6.7)$$

and gas consumption is, of course,

$$V_t = \dot{V}_d \cdot t. \quad (6.8)$$

## 6.4 The Moisturematic

We call our solution to the needs of the Massachusetts Archives the “Moisturematic.” The Moisturematic is a portable machine designed to fill encasements with a humidified inert gas, while minimizing danger to the document, the encasement, and the operator. The completed Moisturematic is shown in Figure 6-1

### 6.4.1 Design Approach

To begin the design, a short list of functional requirements was generated:

1. The machine should accurately humidify argon (or other inert gas), especially near the conditions specified by Massachusetts Archives.
2. The machine should regulate the flow through the encasement during purging.
3. The machine should prevent overpressure of an encasement.
4. The machine should be simple to operate and be tolerant of operational mistakes.
5. The machine should be able to introduce a measured volume of helium for leak checking purposes.



Figure 6-1: Completed Moisturematic (front and rear views)

6. The machine should be portable.

These were then translated into design parameters:

1. The two pressure/two temperature method shall be used, and the saturator shall incorporate closed loop control over temperature and pressure
2. The operator shall be able to set the flow rate with a metering valve and measure the flow rate with a rotameter.
3. A low pressure ( $< 4$  kPa) relief valve shall be incorporated.
4. Operator controls shall be kept to a minimum. An incorrect valve position must not compromise the document.
5. The machine shall be able to switch on-the-fly between argon or helium gas, and shall be able to measure the flow rate of either gas.
6. The machine and gas source should be incorporated into a wheeled cart.

These design parameters were the basis for the design of a saturator for generating humidified gas, development of a component layout and selection of off-the-shelf components to achieve the required functionality, and the integration of the pieces into a portable unit. These three phases of the project are described in detail in the next three sections.

### **6.4.2 Saturator Design**

The basic function of the saturator is to bring gas and water in contact for enough time to equilibrate; this process can be accelerated by increasing the contact area between the water and gas and the time that the two are in contact. Most practical saturator designs would likely fall into one of two categories:

- A “bubbler” design where gas is bubbled through a column of water.
- An “extended surface” design where gas is brought into contact, but not bubbled through, water

Both methods have been used successfully in precision humidity generation machines [21, 26]. Both methods were considered viable for designing humidification equipment for document preservation, and both were pursued in at least some level of detail. Ultimately, the Moisturematic used a bubbler design. Also presented is a conceptual extended surface saturator design that address some of the shortcomings of previous designs.

### **Bubbler saturator design**

The bubbler saturator design was chosen for the Moisturematic because the device was easy to construct, the form factor fit better into the overall system, and data on saturation efficiency was readily available [27]. This article predicts that 99 % saturation of a nitrogen stream could be achieved in as little as 4.8 mm of water depth, and experimentally verified that > 99 % saturation had occurred at 13 mm of water depth, the minimum tested. The authors argue that their mathematical model is insensitive to changes in carrier gas, justifying the application of the results to a pressurized argon carrier gas. The authors suggest a depth of at least 40 mm would be suitable for a wide range of liquids and gases.

Their analysis is valid for the single bubble regime, *i.e.*, when the flow rate is low enough that bubbles do not interact and join together into larger bubbles or a single stream. Their analysis begins when the bubble breaks free from a sparger, which is a device used to generate bubbles. In practice, saturation begins as soon as a bubble begins to form on the surface of the sparger, increasing the time the bubble is in contact with the water. The ideal sparger therefore has a large surface area and a large number of fine pores. A Sweetwater® Fine-Pore diffuser, normally used for fish tank aeration, was used for sparging. The desired combination of fitting and diffuser size was not available off the shelf, so a 1" × 1" × 3" diffuser was cut down to 1.25" in height, and a stainless fitting was bonded in with Hysol® M-21HP epoxy. This assembly is shown in Figure 6-2. The manufacturer claims to provide custom fittings on request, and would likely be able to provide a diffuser with an appropriate fitting.

To minimize corrosion, the saturator is constructed from stainless steel fittings.

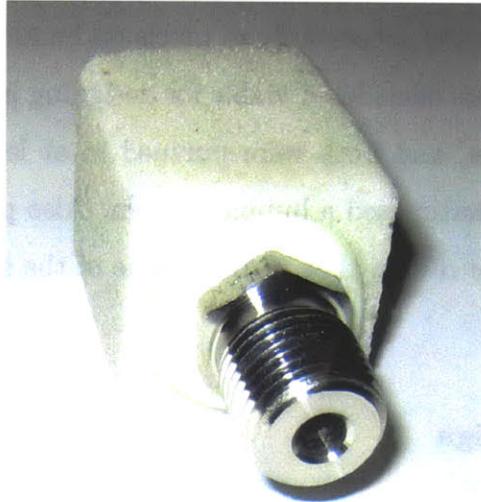


Figure 6-2: Gas diffuser, modified with bonded-in stainless steel fitting.

Sanitary “Tri-Clamp<sup>®</sup>” fittings were used to construct the body of the saturator. These fittings provide a unique combination of features not available with other types of fittings. They can be quickly disassembled and reassembled without the use of tools. They are gasketed and thus do not require sealants such as PTFE tape, which can create debris capable of plugging small orifices. They are also available in relatively large diameters; 2.5” tubing and fittings were used for this project. This size is rated to 2750 kPa (400 psi) when using the wing nut style clamp, and even higher pressures with a bolted clamp.

Stainless compression fittings were welded on to the Tri-Clamp<sup>®</sup> caps for the inlet, outlet, and drain. The body of the saturator also has three additional ports, a compression fitting for the pressure sense port, a thru-drilled compression fitting for the temperature sensing RTD, and a 3/8” NPT port for a sightglass, which can be used to visually gauge the quality of the bubbles. Two coupling nuts were welded on the back side for mounting. The unit was then assembled with valves, gaskets, and a heater sheet for temperature regulation. The finished assembly is shown in Figure 6-3.

The saturator is filled such that the sightglass is completely full. This liquid level places the RTD just under the surface of the water.

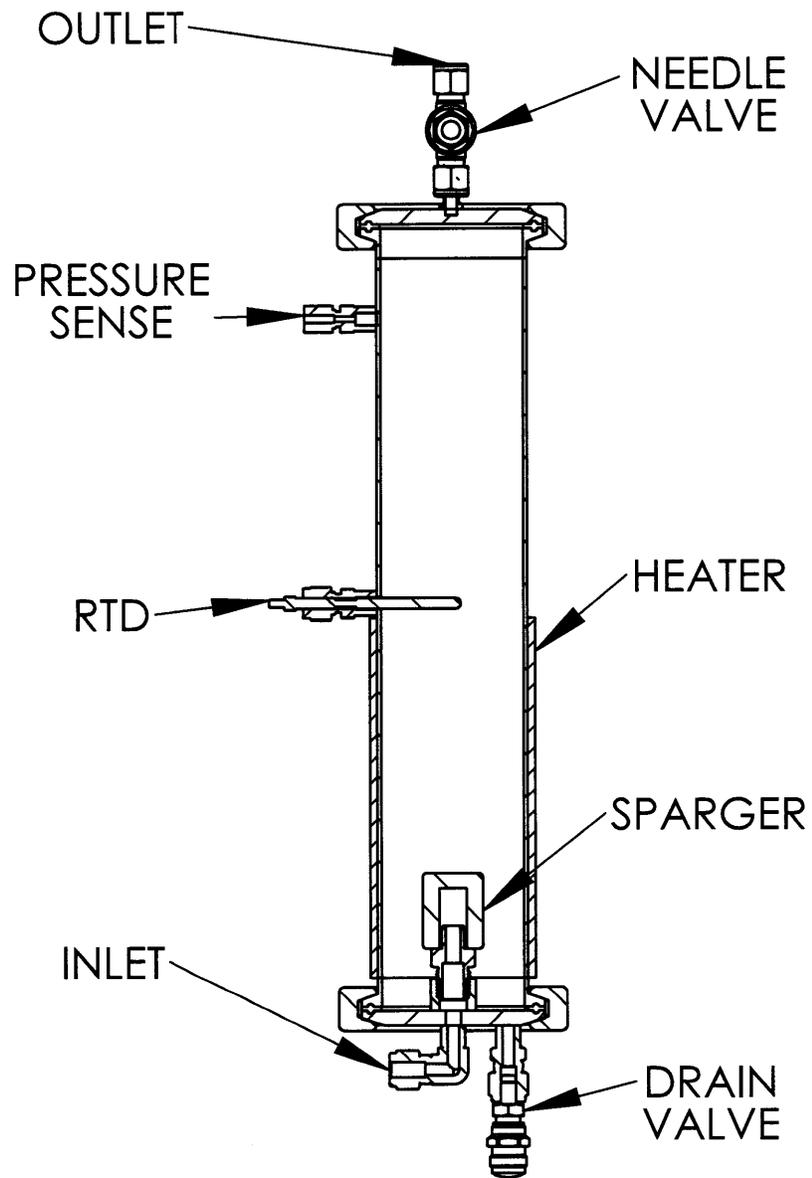


Figure 6-3: Saturator assembly drawing.

A few scenarios were envisioned that could result in inaccurate humidification:

- Bubbles breaking the surface of the water could aerosolize water droplets, which could get entrained in the gas stream
- If the saturator does not operate in the single bubble regime, saturation efficiency could be impaired.
- If the expansion valve was colder than the saturator, water could condense in the line. If the condensation drips down, humidity will be lower. If the condensation is entrained with the air, humidity of the output gas may vary.
- Humidity regulation might be further improved by insulating the assembly to reduce spatial temperature variation

The first and second issues could be addressed with an alternate type of saturator, two of which are described below. The third and fourth issues could be addressed by regulating the temperature of the valve and insulation; alternate saturator designs would also be susceptible to this.

### **Conceptual extended surface and condensing saturator designs**

In case the bubbler saturator proved inadequate, two other designs were conceived of but were never built. The extended surface saturator design of [21] consisted of a copper coil half filled with water. Such a design has a large footprint and must be kept level; this is undesirable for a portable machine.

To reduce the footprint, a saturator consisting of stacked plates was conceived, which is shown in Figure 6-4. The design is modular; increasing the number of plates would increase the surface area of the water/gas interface. To minimize fabrication costs, the unit consists of only three plate designs. The top and bottom plates are unique, while the middle plates are identical. Every other middle plate is simply rotated 180° during assembly. O-rings seal between each of the plates. Just like the bubbler saturator design, provisions for controlling temperature and pressure are necessary.

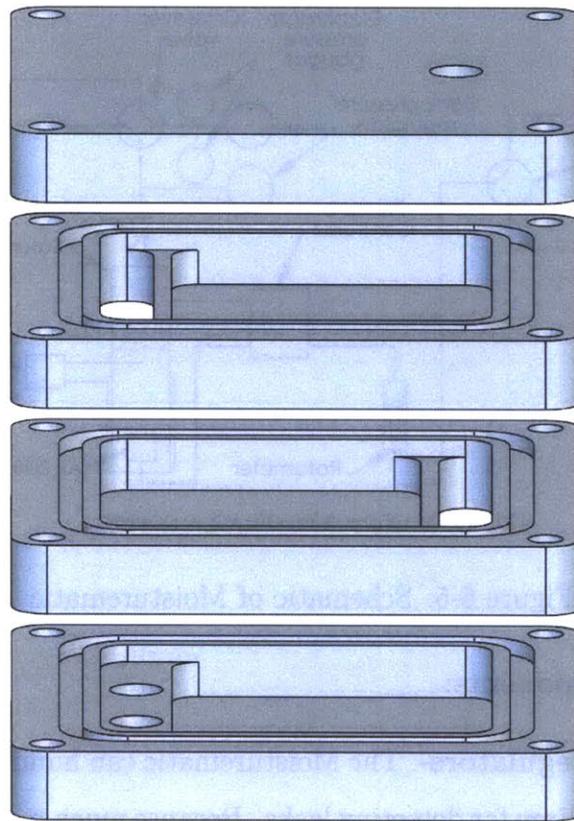


Figure 6-4: Conceptual extended surface saturator

Filling of the saturator is accomplished by simply pouring water in the top. As each plate fills, water spills to the plate below. The bottom plate has a drain; when water spills out the drain, the assembly is full. Unfortunately, a consequence of this design is that draining the unit requires that it be turned upside down. Alternatively, drain plugs would have to be installed on each level.

### 6.4.3 Schematic and description of components

The design parameters were the basis for the development of a component layout and selection of off-the-shelf components to achieve the required functionality, the design of a saturator for generating humidified gas, and the integration of the pieces into a portable unit. The layout of these components is shown schematically in Figure 6.4.3.

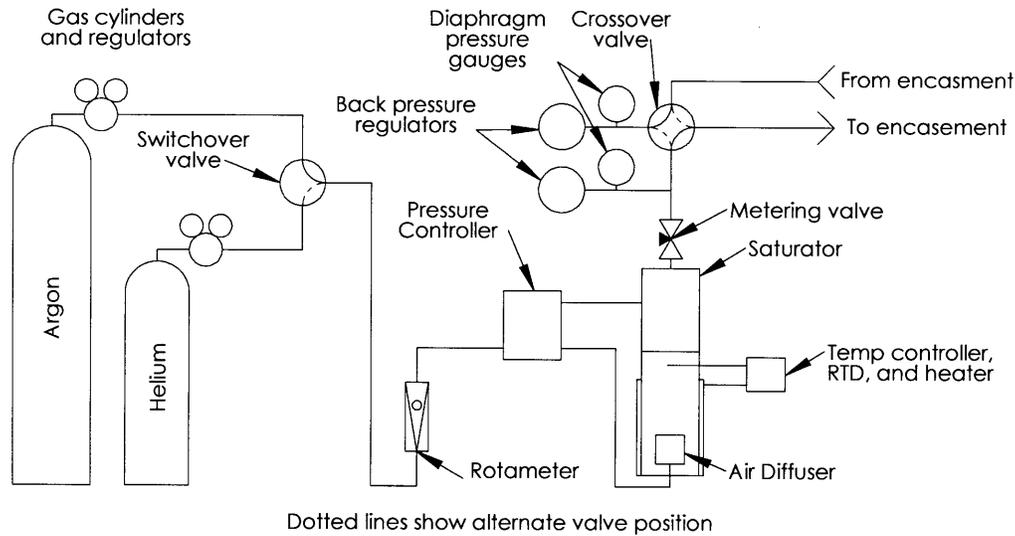


Figure 6-5: Schematic of Moisturematic

### Moisturematic components:

**Gas cylinders and regulators-** The Moisturematic can humidify argon for encasement filling or helium for detecting leaks. Because much more argon than helium is needed, the Moisturematic carries a 200 cubic foot cylinder of argon and an 80 cubic foot cylinder of helium. The regulators used have a maximum outlet pressure of 100 psig. Just about any CGA580 regulator with the correct pressure output can be used, though more expensive high purity models have metal diaphragms.

**Switchover valve-** A Swagelok SS-43GXF4 three port switching service valve is used to select helium or argon gas for humidification.

**Rotameter-** An Omega FL-3651G-NV is used in conjunction with the needle valve on the saturator to set the appropriate flow rate, usually 1.5 L/min. This particular rotameter will allow measurements from 0.19 L/min to 1.9 L/min of dry argon at 60 psig.

**Pressure controller-** An Alicat Scientific PC3-100PSIA-D is used on the inlet of the saturator to control absolute pressure within the saturator. It has a remote pressure sensing port, which is connected to the headspace of the saturator.

The unit has a maximum outlet pressure capability of 100 psia (690 kPa). The pressure controller requires 24 VDC for operation, which is supplied by an ICC/Elpac MSM0724 power supply contained in the same enclosure as the temperature controller.

**Temperature controller-** An Omega CNI16D24-C24 temperature controller and an Omega PRCTL-2-100-A-3/16-24-40 RTD are used to measure the temperature of the water in the saturator. This controller's internal solid state relay directly controls a McMaster 35765K228 heater strip adhered to the surface of the saturator vessel. This model also has a dual display, which displays both the current temperature and the setpoint. This helps to let the operator know when the machine is warmed up.

**Saturator-** The custom built saturator is described in Section 6.4.2.

**Metering valve-** A Swagelok SS-4MG-SC11 needle valve is used to regulate the flow rate of gas through the Moisturematic. This valve expands gas saturated under pressure to near atmospheric pressure, resulting in a dryer gas. A Swagelok NY-2M-K6 micrometer handle kit was installed to aid in setting the flow rate repeatably.

**Diaphragm pressure gauges-** two Omega PGL-25L-35 gauges measure pressures in the inlet and outlet lines of the encasement. These gauges measure from 0" to 35" inches of water column (W.C.) (0 – 8.6 kPa).

**Back pressure regulators (BPRs)-** two Emerson 289U-4 BPRs, adjustable from 5" to 25" W.C. (1.2 – 6.2 kPa) act as relief valves to limit pressure on both the inlet and outlet hoses connected to the encasement. The outlet BPR creates a slight positive pressure in the encasement while purging. The inlet BPR prevents overpressure of the encasement, even if a hose is stepped on or a valve is inadvertently left closed.

**Crossover valve-** A Swagelok SS-45YF4-1466 four-port crossover valve allows purging to be started ("purge" position) or stopped ("idle" position). The function

of this valve is to alter the path of humidified gas to add a loop through the encasement when the valve is in the purge position. When the crossover valve is in the idle position, the encasement is isolated, but gas will still flow through the saturator and out of the BPRs to allow warmup and adjustment. When idling, the encasement and hoses are maintained at a slight positive pressure, preventing oxygen ingress from any leakage in the hoses and fittings. Combining control of the purge and isolation functionalities into a single valve simplifies operation and increases safety to the encased document.

#### **6.4.4 Integration**

The functional requirement of portability could have been addressed in a variety of ways, but the simplest solution was to incorporate a hand truck directly into the design of the Moisturematic.

Of the many hand trucks on the market, the best match for the Moisturematic had these features:

1. “Continuous” handle design would be less likely to interfere with component placement.
2. Made of steel, so it could be easily modified.
3. Available locally, so it could be inspected for quality and configuration surprises.

After looking at several hand trucks in person, it was found that trucks designed specifically for moving gas cylinders would require more modifications than a standard truck and were not as well made as standard hand trucks.

A Dayton 2W179 hand truck was bought from Grainger. Measurements were made of the available space; it was decided that the Moisturematic components (excluding the cylinder and regulator) would be arranged to fit on a rectangular plate 11” × 32”, and that the components would be mounted such that they do not protrude from this mounting plate by more than about 4.5.” This allows the handle to protect the components when the machine is transported.

Solid models were made of the plate and the selected components. The components were arranged to fit within the required envelope while making the connections as direct as possible. The cartoon of Figure 6.4.3 reflects the final component placement, while Figure 6-6 shows the actual Moisturematic control panel. Three horizontal crossmembers were welded to the hand truck frame to mount the plate and components.

To hold the argon cylinder, two TH-109 cylinder brackets were purchased from 4B's Bracket Company. These are intended for a cylinder between 9" and  $9\frac{3}{4}$ " in diameter; other sizes are available. These brackets mount the bottle very securely, but allow for easy bottle changes. These were welded directly to the top and bottom crossmembers. The helium cylinder was added later, and was simply strapped to the Moisturematic with a ratchet cargo strap. Two small cylinders would be a better fit, this is discussed in Section 6.4.8.

The Moisturematic electronics enclosure contains the temperature controller, fuses, and a power supply for the pressure controller. The electronics package is shown in Figure 6.4.4. The left panel of Figure 6.4.4 shows a side view of the electronics package; the depth of the temperature controller stands out as an obvious packaging problem, since the available depth was constrained by the desire to shield components from physical damage, as mentioned previously. To solve this problem, a hole was cut in the mounting plate of the Moisturematic and the base of the electronics enclosure (riveted to the front of the mounting plate), and a 2nd enclosure on the opposite side of the mounting plate was used to house the far end of the temperature controller. This has the added advantage of making the controller connections easily accessible. The right panel of Figure 6.4.4 shows the base of the 2nd enclosure riveted to the mounting plate of the Moisturematic.

The bill of materials for the electronics is listed in Table 6.3. This BOM is intended as a reference for if the Moisturematic as delivered to the Massachusetts Archives needs repair. General wiring supplies such as zip ties, heat shrink tubing, and electrical tape are not included.



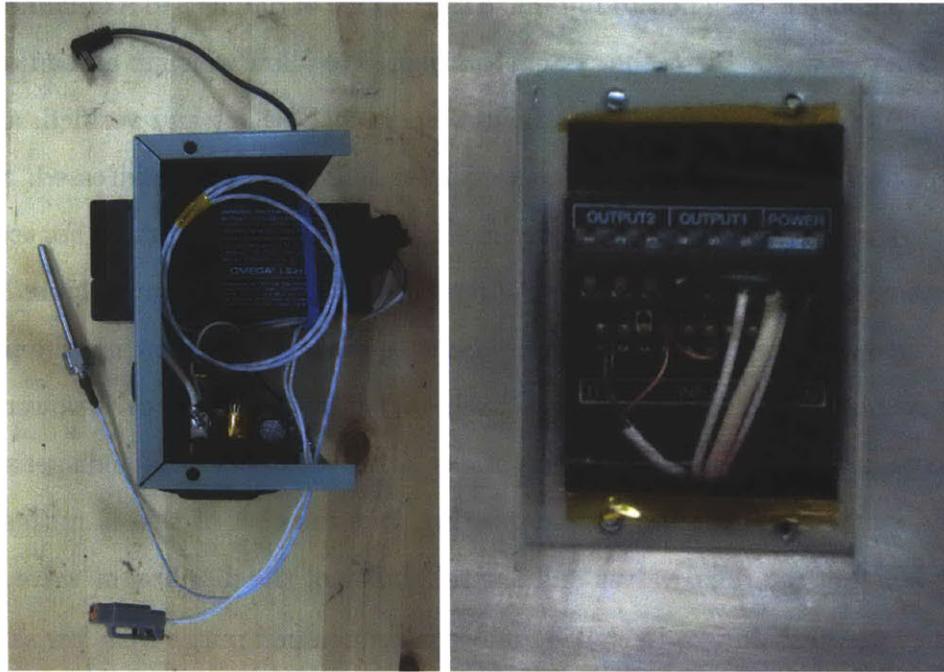


Figure 6-7: Moisturematic electronics.

Table 6.3: Moisturematic electronics BOM.

Description	P/N	Manufacturer	Supplier
Temperature controller	CNI16D24-C24	unknown	Omega
RTD	PRCTL-2-100-A-3/16-24-40	unknown	Omega
Heater blanket	35765K228	unknown	McMaster
enclosure (small)	1411D	Hammond	Digikey
enclosure (large)	1411L	Hammond	Digikey
Pressure controller power supply	MSM0724	ICC/Elpac	Digikey
connector (output)	50-37-5023	Molex	Digikey
connector (input)	50-37-5033	Molex	Digikey
connector pin (input/output)	08-70-1040	Molex	Digikey
fuseholder	03450621H	Littelfuse	Digikey
fuse	0234002.MXP	Littelfuse	Digikey
power entry module	PX0580/63	Bulgin	Digikey
power cord	232003-06	Qualtek	Digikey
Deutsch DTM 2 pin connector	various	Deutsch	Ladd Ind.

### 6.4.5 Safety

The Moisturematic is designed to minimize the chance of damage to a document; this is achieved in two ways. First, the system is designed to allow warmup and adjustment while bypassing the document. This allows setpoints to be set and verified, and also allows any effects from storage, such as a sticky relief valve, to be addressed. Second, the impact of most electrical and mechanical failure modes is minimal. For example, if the pressure controller valve fails in the open position, the minimum humidity will be set by the cylinder regulator, which need not be set much higher than the pressure controller setpoint. If a component fails such that flow is blocked or if power to the pressure controller fails, only the volume of gas in the saturator will be dispensed. An overpressure situation could be very damaging, but the risk of this is mitigated by the redundancy of the relief valves and the reliability of the diaphragm valve design.

By far the gravest danger to the document is a sustained purge with low saturator pressure, which would dispense gas with too high a humidity. This situation could arise if the cylinder is nearly empty or if the cylinder regulator is set too low. Assuming a four hour purge at 1.5 L/min, it would be good practice to never start a purge if the cylinder pressure is below 200 psi. Even though the Moisturematic is designed to be simple to operate, the operator presents a bigger danger to the document than the machine.

### 6.4.6 Operational Envelope and Accuracy

The operational envelope is a consequence of the component selection. Using the specifications of the components listed above, we can determine the limits of operation for the Moisturematic.

The saturator is not intended to operate at temperatures drastically different from ambient, so this analysis will assume  $T_c = T_s$ . Neglecting the effect of the enhancement factor and assuming a fixed (roughly atmospheric) encasement pressure, the range of humidities that can be generated can be directly calculated from the usable absolute pressure range of the saturator.

The maximum saturator pressure is constrained by the fitting and vessel selection and the selection of pressure controller. The lowest-rated component of the device is the pressure controller, which has a maximum pressure capability of 690 kPa. This gives a minimum RH of 6.7%. Note that pressure controllers with higher pressure ratings are available. These controllers, however, will have less accuracy when used at lower pressures, since accuracy is rated as a percentage of full scale. Such a compromise is not necessary unless low humidities need to be generated. If low humidities need to be generated, the pressure capability of the entire system must also be considered. If system pressure were increased, certain parts would need to be changed to ensure that ratings are not exceeded. These parts include the pressure controller (690 kPa), gas regulator (790 kPa), interconnecting tubing (1380 kPa), and rotameter (1480 kPa).

The minimum pressure is determined by the pressure drop through the components downstream of the saturator. The biggest contributors to pressure drop are the metering valve and the BPRs. To maintain a flow rate of 1.5 L/min, a pressure differential across the metering valve of approximately 2.2 kPa is required, as calculated using the methods of [28]. Combined with the minimum cracking pressure of 1.2 kPa (gauge) and an atmospheric pressure of 101.4 kPa, the minimum saturator pressure is approximately 105 kPa, giving a maximum RH of about 96%. Of course, at very high output humidities, the possibility of condensation after the saturator must be considered.

Also of interest is the error in the RH output as a result of inaccuracies in temperature and pressure readings, saturator efficiency, and enhancement factor. Table 6.4 shows the nominal setpoints corresponding to 40%RH @ 21.1 °C, estimated maximum errors, and the effects of those errors on output humidity. As shown in Figure 6-8, the largest contributors to the relative humidity error are the saturator temperature and encasement pressure.

An alternative way of determining errors is to compute the Jacobian of Eq. 6.2:

$$J_{RH}(T_s, T_c P_c, P_s) = \left[ \frac{\partial(RH)}{\partial T_s} \middle| \frac{\partial(RH)}{\partial T_c} \middle| \frac{\partial(RH)}{\partial P_c} \middle| \frac{\partial(RH)}{\partial P_s} \right]. \quad (6.9)$$

Computing the Jacobian requires either a numerical solution, or an approximate expression for  $e_w$ , which can be found in [22]:

$$e_w = 61.121 \exp \left( \left( 18.678 - \frac{T_s}{234.5} \right) \frac{T_s}{257.14 + T_s} \right). \quad (6.10)$$

Assuming the enhancement factors to be equal to unity, evaluating Equation 6.9 at the suggested operating point from Table 6.4 results in

$$J_{RH}(T_s, T_c, P_c, P_s) = [0.0239 \text{ }^\circ\text{C}^{-1} | -0.0247 \text{ }^\circ\text{C}^{-1} | 0.00390 \text{ kPa}^{-1} | -0.00123 \text{ kPa}^{-1}], \quad (6.11)$$

*i.e.*, a 1 °C rise in saturator temperature will raise the output humidity from 40 % to 42.4 %. The saturator temperature measurement contributes more error than either of the pressure measurements. The saturator temperature is measured with an RTD; these are widely used for precision temperature measurements, but have limited accuracy “out of the box.” The manufacturer offers a calibration service that would reduce this uncertainty. Similarly, factory calibration is available for the pressure controller.

One can see that the instrumentation selected approximately balances the error from each source. Since errors are often expressed in percentages of full scale ranges, it is important to select instrumentation that matches the expected operating range. For example, RH error is more sensitive to encasement pressure than saturator pressure. This occurs because the saturator operates at a higher pressure than the encasement. While the saturator pressure sensor has a larger pressure error than the encasement pressure sensor, the encasement pressure sensor introduces somewhat more relative humidity error. The encasement pressure error estimate is based on the use of MKS Baratron<sup>®</sup> 870B pressure sensors on the encasement, which have an accuracy of 1% of the reading.

Finally, saturator pressure error could be halved with factory calibration, but one can see from Figure 6-8 and from Equation 6.9 that reducing saturator temperature error and encasement pressure error are more effective than reducing saturator

variable	value
<b>nominal values</b>	
saturator pressure	324.8 kPa
saturator temperature	25.0 °C
encasement pressure	102.6 kPa
encasement temperature	21.1 °C
saturator efficiency	100 %
enhancement factor	1.000
<b>errors</b>	
saturator temperature	±0.2 °C
saturator pressure	±1.7 kPa
encasement pressure	±1.0 kPa
saturation efficiency	+0, -1 %
combined enhancement factors	+0.007, -0.000
<b>outputs</b>	
humidity (nominal)	40.0 %
humidity (maximum)	41.4 %
humidity (minimum)	38.6 %

Table 6.4: Effect of estimated errors on humidity of output gas.

pressure error.

## 6.4.7 Operation

Procedures for operation are in Appendix A.3.

## 6.4.8 Future work

The design of the Moisturematic was thought to be a success by all involved in the encasement project. Still, there is always room for improvement. Two areas, both related to human interaction with the machine, were thought to need the most improvement: usability of the electronics, and weight and handling of the machine. Also, although the accuracy of the machine was determined to be adequate for this encasement project, a few simple changes could further reduce error.

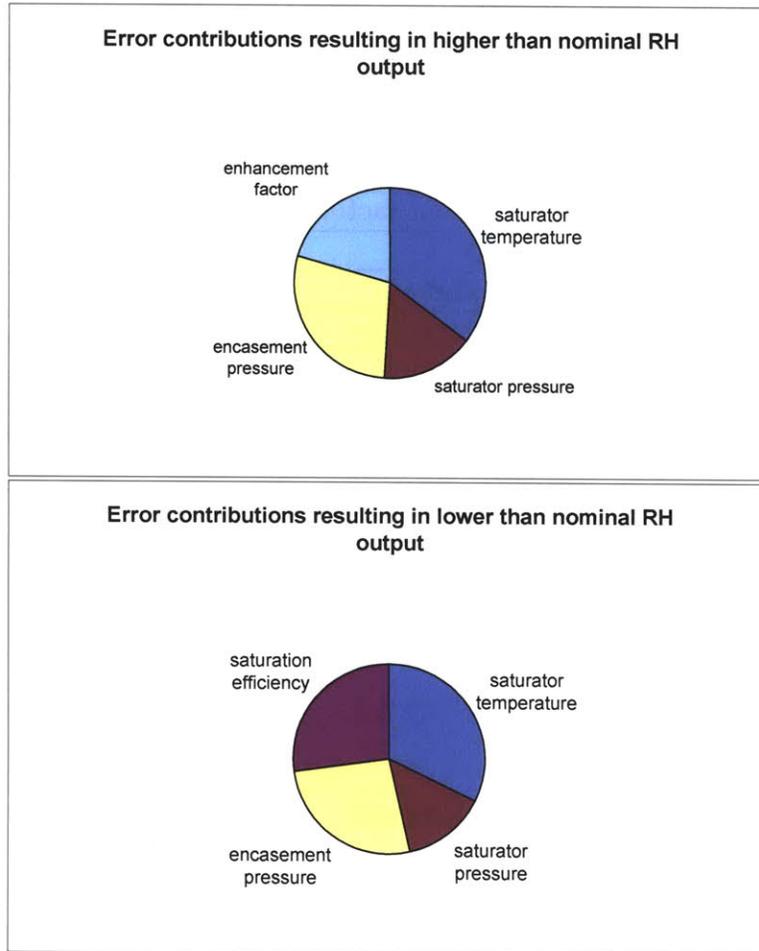


Figure 6-8: Error contributions from various sources.

## **Electronics**

Both the pressure controller and temperature controller are capable of being controlled by a computer through a serial port. Enabling this functionality would improve the usability of the machine for a few reasons:

- The electronic controls are low on the machine, and therefore hard to reach.
- The controller interfaces are somewhat complicated (adjustment is not as simple as pressing up/down buttons).
- Adjustment would be automated, reducing the chance of an input error.

## **Weight and handling**

Overall weight reduction and a reduction in the center of gravity height would both contribute to better handling of the machine. The 200 cubic foot cylinder could be replaced with a smaller cylinder (reducing both weight and CG), and the reduction in capacity would probably be of little concern. At a flow rate of 1.5 L/min, an 80 cubic foot cylinder would last around 25 hours. Downsizing of the argon cylinder also paves the way for tighter integration of the helium cylinder, which was not in the original Moisturematic design. Two 80 cubic foot cylinders would easily fit side by side on the hand truck. Another advantage of the smaller bottles is that they are usually available for purchase, while the larger bottles must usually be leased. As a result, Massachusetts Archives will have to lease cylinders when the time comes to helium leak test or flush the encasements.

## **Accuracy improvements**

As mentioned in section 6.4.2, the saturator would likely benefit from insulation and a heated metering valve. Further accuracy improvements could be had by regulating pressure and temperature better. Both controllers can be calibrated at the factory (at additional cost) to reduce the error.



# Appendix A

## Procedures

This chapter is the “owner’s manual” for the Massachusetts Archives encasements. Here, we describe procedures for cleaning, assembly, and testing.

### A.1 Cleaning

Cleanliness is very important prior to encasement assembly, both for the safety of the document and the integrity of the seal. Cleanliness is also important to maintain the aesthetics of the museum display.

#### A.1.1 Glass

The glass is the only component that will need regular cleaning. The glass should be cleaned with an ethanol/water solution and clean room wipe. Never use a metal tool on the anti-reflective coating, or it will be permanently damaged. Low light makes any lint on the surface of the glass very visible; getting the glass clean can take several tries.

#### A.1.2 Small metal parts

Most of the smaller parts fit into an ultrasonic cleaner bath. Examples of these parts include manifolds, brackets, deflectors, fasteners, and platen mount components,

These parts were cleaned with a Luminox<sup>®</sup> solution, rinsed with deionized water, dried with compressed air, and bagged until ready for use. Luminox<sup>®</sup> will not corrode aluminum components as some stronger detergents will. The Luminox<sup>®</sup> should be diluted 25:1 with warm water before use.

### **A.1.3 Tub and clamp**

The tub, clamp, and platen were too large, too delicate, and too heavy to wash in a sink. Instead, these parts were wiped with several rounds of cleanroom rags soaked in ethanol. After cleaning, these were stored in large plastic bags. These components were wiped again immediately before use.

### **A.1.4 Platens**

The platens are the most difficult component to clean, because each one has thousands of holes. The platens were cleaned in a large darkroom sink at Massachusetts Archives. The platens were sprayed with Luminox<sup>®</sup> solution, scrubbed, rinsed with warm tap water, and finally rinsed with warm distilled water. After the platens dried, they were stored in plastic bags until ready for use.

### **A.1.5 Melinex<sup>®</sup> document clips**

The document clips are the only material that contacts the document, other than the cardboard backing, which was supplied by the Archives. After bending, the clips were wiped with a cleanroom rag soaked in ethanol and stored in a plastic bag until ready for use. After cleaning, they were handled with gloves to prevent possible contamination, as well as prevent smudges and fingerprints, which could be visible in the museum display.

### **A.1.6 O-rings**

The original set of o-rings were wiped with a cleanroom wipe dampened with isopropanol, a common practice with Viton<sup>®</sup> o-rings. However, Viton<sup>®</sup> is incompatible

Table A.1: Fastener torque specifications.

Fastener	Size	Torque
platen posts	1/2"-13	50 N – m (37 lb – ft)
clamp plate	3/8"-16	40 N – m (30 lb – ft)
valve brackets, manifold brackets, platen clips	1/4"-20	5.7 N – m (50 lb – in)
fitting blocks, deflectors	#10-32	3.4 N – m (30 lb – in)
valve handles	#6-32	1.4 N – m (12 lb – in)
cross clamp	#4-40	0.8 N – m (7 lb – in)

for long term contact with any polar solvents, including water. Even “light” hydrocarbons (which would evaporate quickly) are incompatible, leaving little that can be done to clean an o-ring before use. Since it was felt that contact with isopropanol may have compromised the original set of seals, the replacement o-rings were only wiped with a dry lint-free rag.

## A.2 Encasement Assembly

This section details the assembly procedure of the Massachusetts Archives encasements. This information is also expected to help if an encasement component should need servicing or replacement. Before beginning assembly work, all components should be cleaned using the techniques described in Section A.1.

### A.2.1 Fasteners and fittings

All fasteners should be torqued to the proper specification, as shown in Table A.1. The aluminum threads on the encasement can be damaged by overtightening fasteners. Undertightened fasteners may become loose, possibly causing leaks.

Swagelok VCR<sup>®</sup> fittings are face seal fittings that utilize a metal gasket. A new gasket must be used each time the fitting is made up. Nickel side loading gaskets were used; the side loading design retains the gasket during assembly. No sealants or lubricants should be used on any part of the fitting. Be careful to not scratch

the sealing surfaces during installation, or the fitting will leak. All Swagelok VCR® fittings should be tightened by “turn of nut” instead of to a torque specification. Because of the short (and therefore relatively stiff) tubing runs, angular misalignment in the fittings means that “finger tight” might not be an adequate starting point. In this situation, as per Swagelok instructions, fittings should be snugged with a wrench until an increase in resistance is felt, indicating that the faces of the fitting are fully in contact with the seal. They should then be tightened an 1/8 of a turn more. Placing a mark across both fittings before final tightening helps to indicate when the nut has been turned 1/8 of a turn, as shown in Figure A-1. It is important to note that some VCR® fittings do not have a rotatable gland; examples include the cross and tee. This means the non-rotatable gland must be held stationary while the fitting is tightened. In all cases, a backup wrench should be used to avoid putting stress on the plumbing. For more information on installing VCR fittings, refer to Swagelok’s Publication MS-13-150 [29].

Also note in this figure that the cross is held in place with stainless #4-40 screws, screwed into a stainless bracket. Stainless-on-stainless threads are prone to galling. These screws must have a small amount of anti-seize applied. Similarly, the bolts that attach the valves to their brackets and the screws that hold the valve handles on must also be coated with anti-seize before assembly.

## **A.2.2 Base assembly**

### **Valve preparation**

The first assembly step is to remove the original valve handles, which are too large to be used. The replacement valve handles can be installed at this time; anti-seize should be applied to the valve handle fasteners to prevent galling.

Due to limited access, the valves must be screwed to the brackets before the brackets are attached to the encasement, but the screws should not yet be tightened. The valve and bracket assembly can then be screwed to the case, but also should not be tightened. The idea is to let components “float” during assembly, minimizing residual

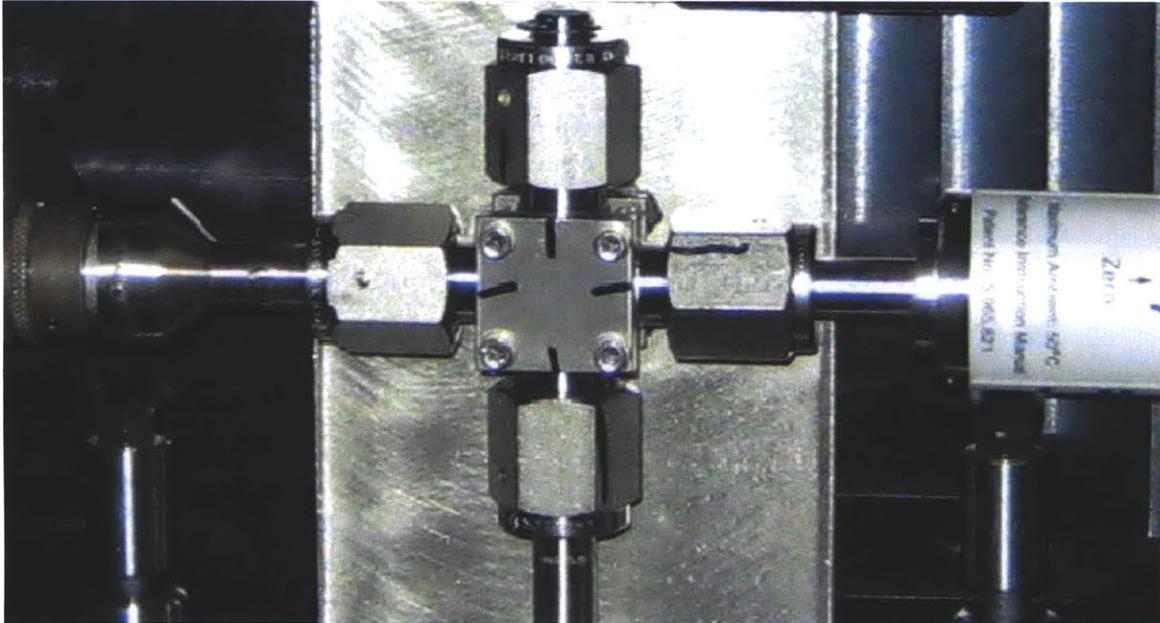


Figure A-1: VCR<sup>®</sup> fittings with marks indicating tightening 1/8 of a turn beyond initial seal contact.

stresses in plumbing components. Figure A-2 shows this assembly step.

### **Sensing groove plumbing**

The bracket for the outer manifolds must then be installed, followed by the outside manifolds. Use a new VCR<sup>®</sup> gasket on each connection, and tighten according to the instructions above. Where manifolds connect to the tub, the connection must be sealed with an o-ring.

The sensors should then be installed. Since the pressure sensors have an adjustment screw; the sensor should be oriented so the screw is accessible. Any unused ports must be closed with VCR<sup>®</sup> caps.

### **Center popup plumbing**

Installation of components in the center popup is similar to the components of the outside manifolds. Screws holding the valves to their brackets and the brackets to the tub should be left loose to allow components to float. The tee, cross, union, and sensors can then be installed, leaving the cross clamp loose until all fittings have

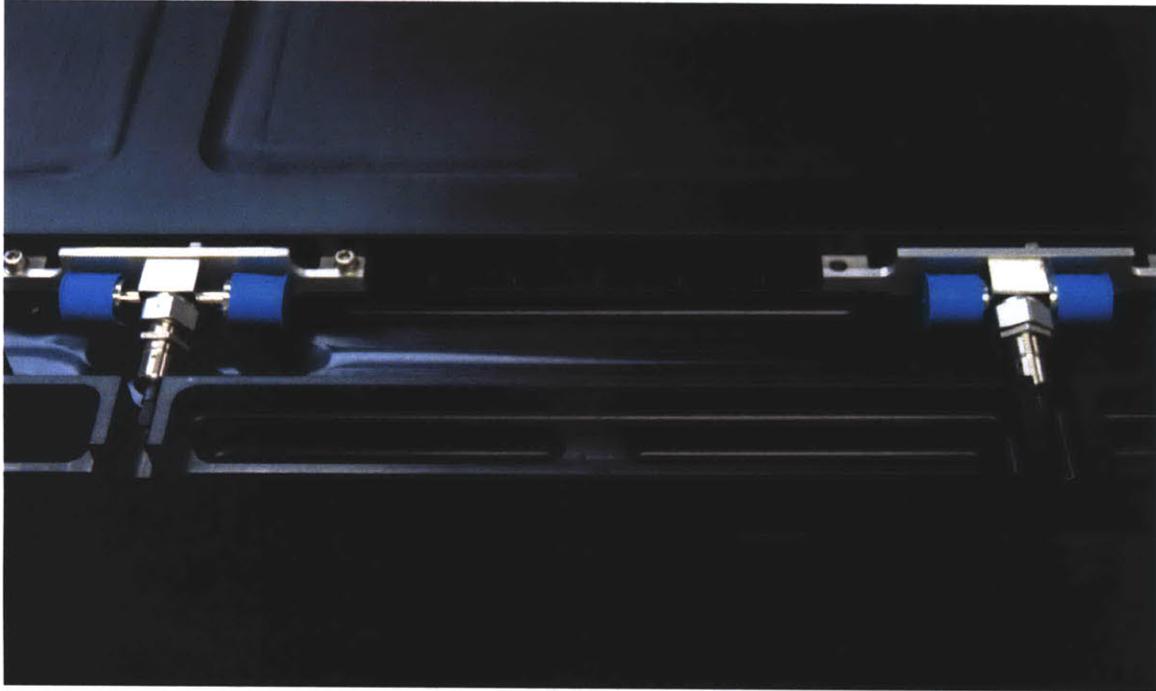


Figure A-2: The first assembly step is to install the valves and brackets, but the screws should not be tightened at this time.

been tightened. Finally, tighten the remaining screws to secure the plumbing. When everything is installed properly, the components in the center will look as shown in Figure A-3.

### **A.2.3 Helium leak checking the base assembly**

VCR<sup>®</sup> fittings, o-rings, welds, valve bellows, sensors, and feedthroughs should be helium leak checked after assembly. It is best to check backside plumbing before final assembly, while it is still easy to fix problems. Because the leak detector needs a vacuum to function, the ports that connect the backside plumbing to the sensing grooves and encasement interior must be plugged. A test plug was constructed as shown in Figure A-4. Tightening the nut (finger tight is enough) squeezes the o-rings, forming a seal between the grip of the screw and the port.

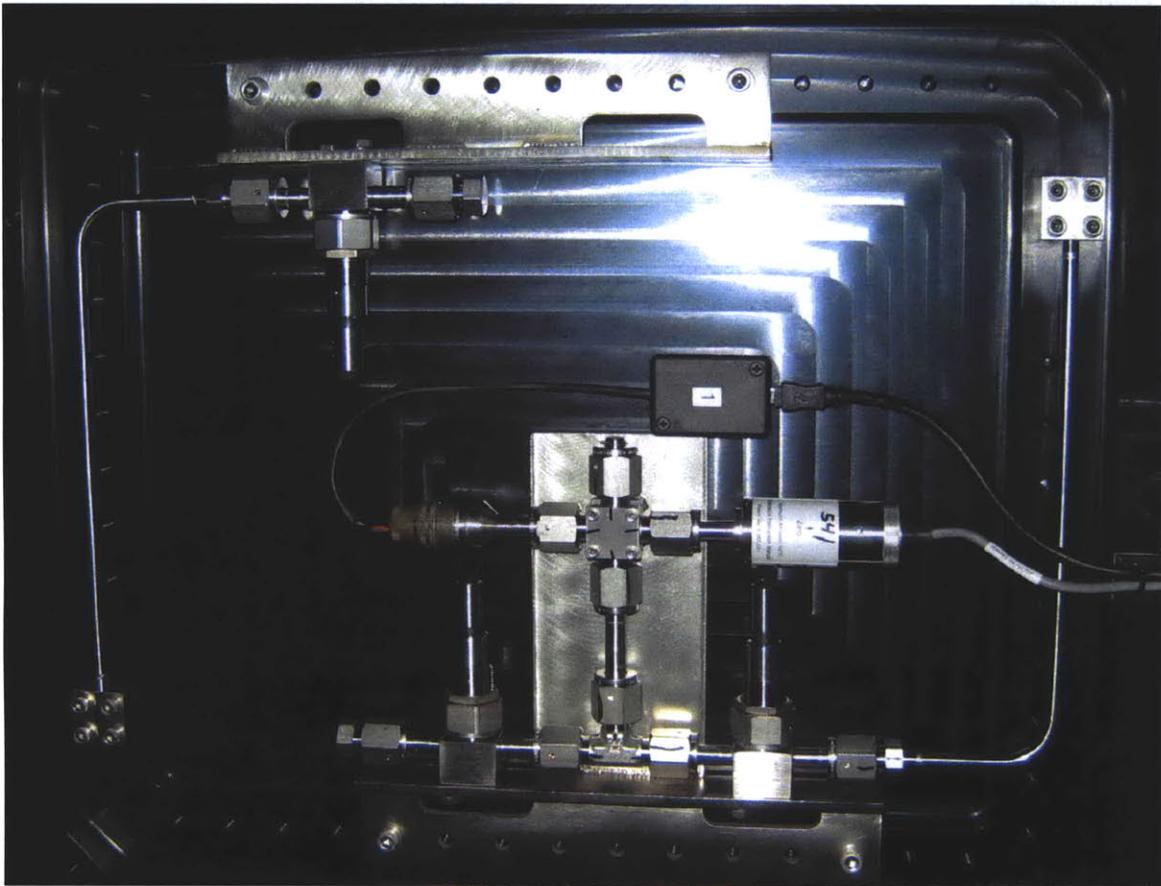
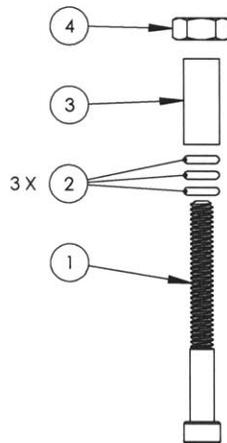


Figure A-3: Plumbing and instrumentation in the center popup.



Item	Description	McMaster P/N
1	screw, #4-40 × 1.125" (partially threaded)	92196A117
2	o-ring, 2.5 mm ID × 4.5 mm OD × 1 mm CS	9262K511
3	aluminum sleeve, 0.120" ID × 0.180" OD × 0.44" L	N/A
4	nut, #4-40, nylon	94812A112

Figure A-4: Test plug for backside plumbing

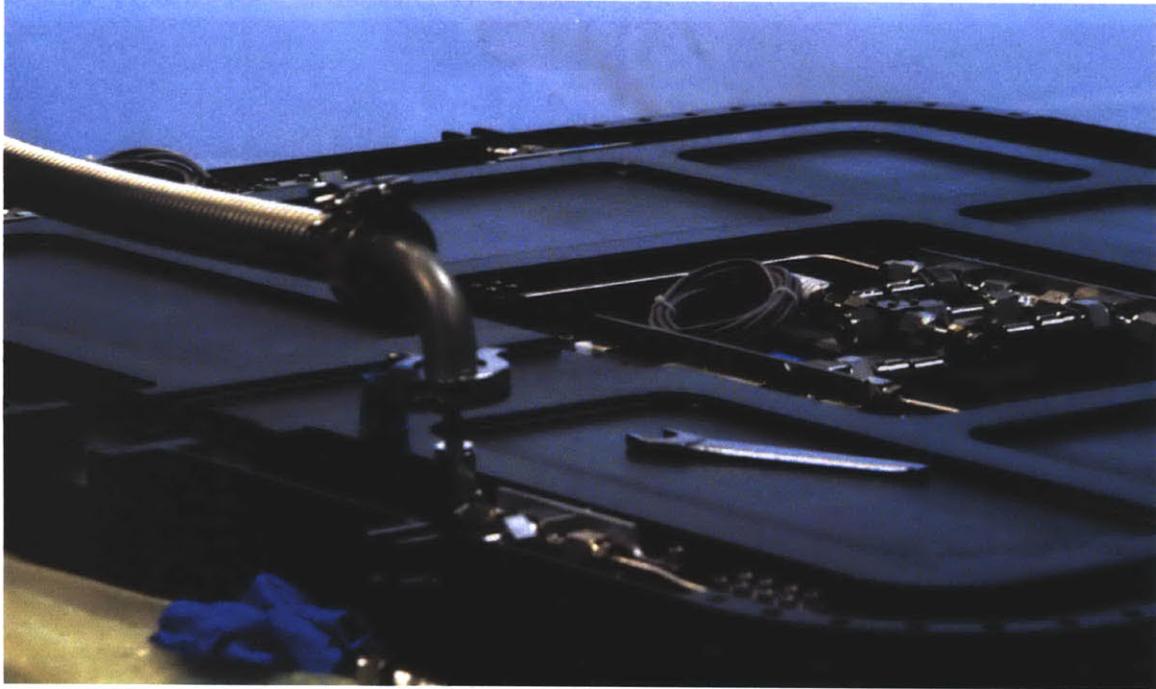


Figure A-5: Encasement upside down on the work bench. The large hose is for helium leak testing the assembled manifold.

The helium leak detector should then be connected to each inlet/exhaust port, as shown in Figure A-5. The helium leak detector should be warmed up and calibrated as per the manufacturer's instructions before looking for leaks. Every suspected leak path should then be exposed to helium for several seconds while watching the helium leak detector for signs of a leak. Helium flow rate should be limited to less than 1 L per minute. Too much helium in the atmosphere can increase background, making real leaks more difficult to detect. Figure A-6 shows the dispensing equipment used in this project. The needle allows helium to be directed into tight spaces, while the valve allows the flow to be shut off when not in use. The flow rate should be set to about 1 L/min, which assures helium surrounds the seal being tested without significantly raising the helium background in the room, which can lead to incorrect readings. The flow rate can be set by pointing the needle at one's tongue; only enough flow is needed to feel a cooling sensation.

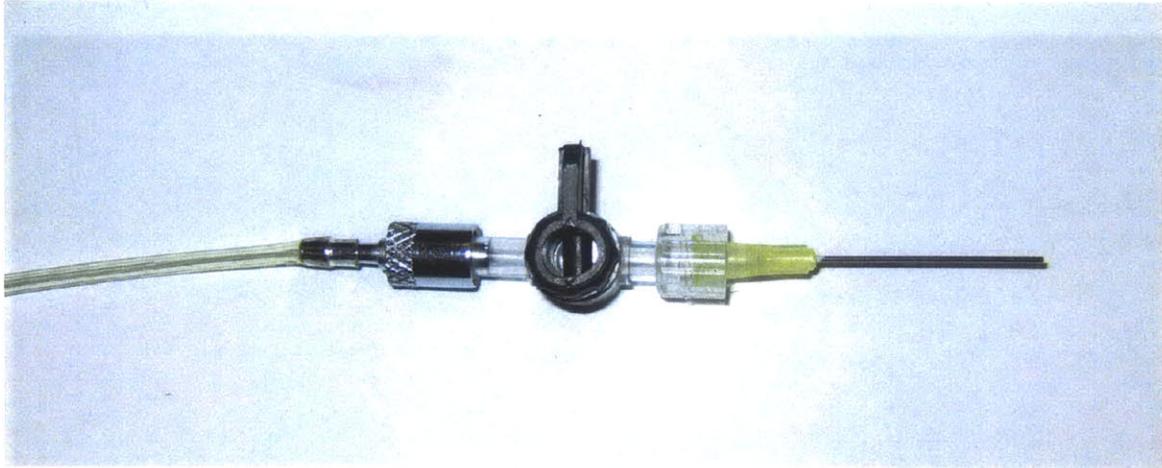


Figure A-6: The helium dispensing probe with needle and valve.

Figure A-7 shows a small scratch in a sealing surface of one of the tubs. This defect caused an unacceptably high helium leak rate, requiring the removal of the manifold, repair of the defect, and replacement of the manifold. The defect was repaired by sanding with 1000 grit sandpaper. After the scratch was removed, the sandpaper was rotated about the axis of the center hole to prevent radial scratches from causing leaks. The leak was undetectable after repair (the helium leak detector range stop was set at  $0.1 \times 10^{-10}$  atm – cc/sec).

#### A.2.4 Topside assembly

The next assembly step is to install components on the top side of the tub, which include only the argon deflectors and the platen posts. Because the washers under the platen posts are thin, a special tool (Figure A-8) was created to hold the washers and posts concentric while tightening. The screws and posts should be properly torqued, as specified in Table A.1. Figure A-9 shows the installation of argon deflectors; the platen posts are already installed.

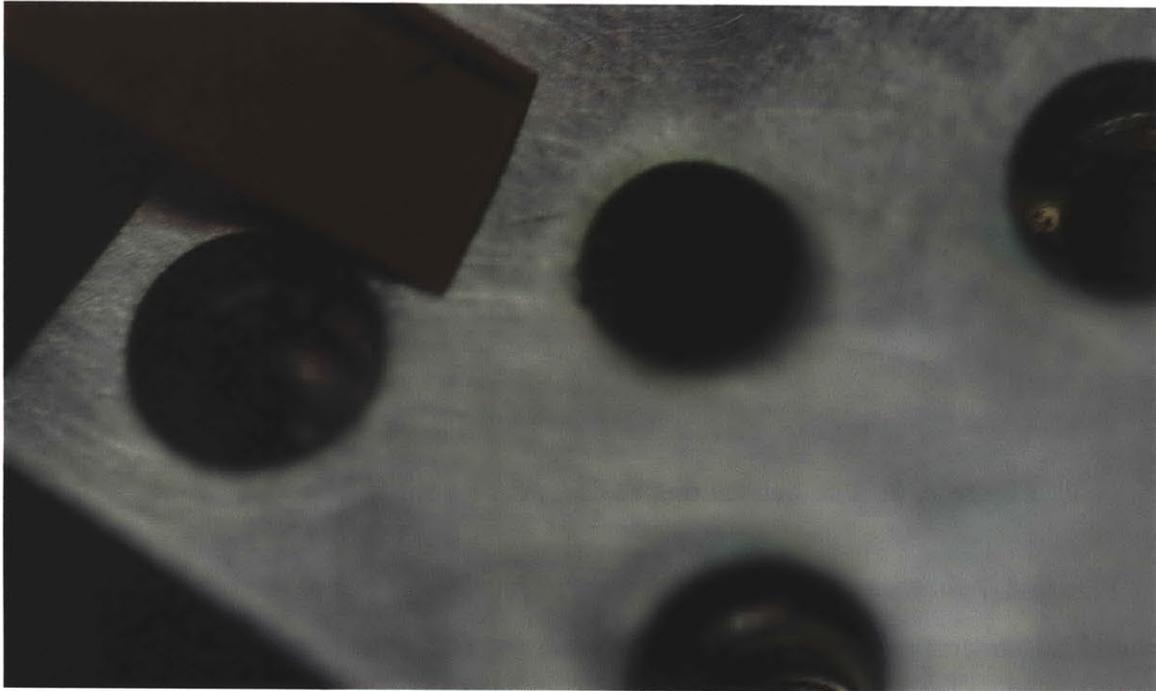


Figure A-7: A scratch in a sealing surface that required polishing to seal.

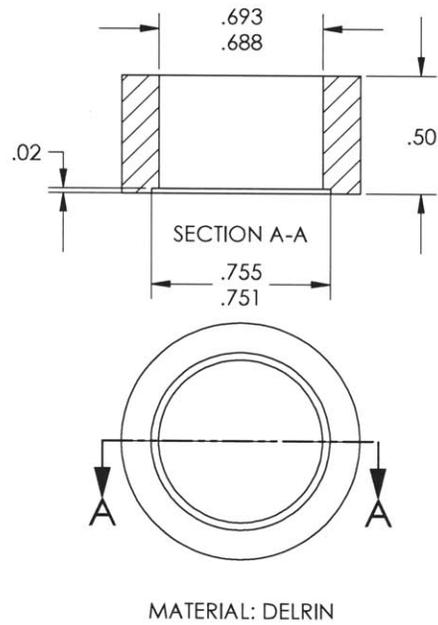


Figure A-8: Platen post washer centering tool.

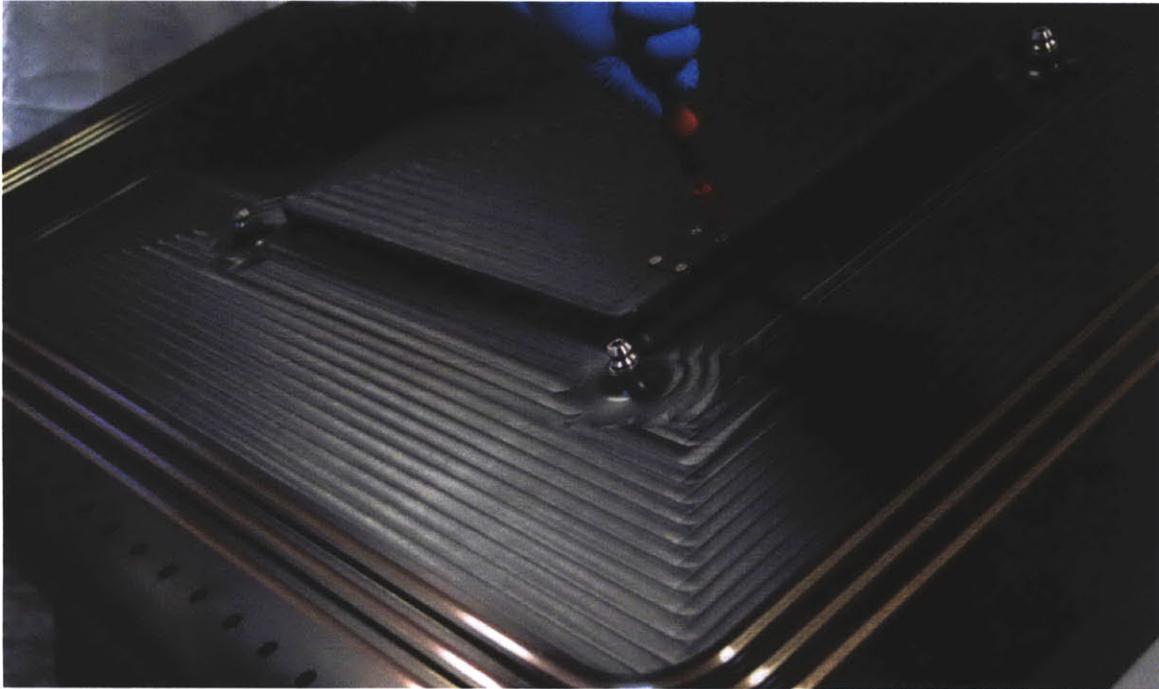


Figure A-9: Argon deflectors and platen posts are installed.

### **A.2.5 Glass and clamp preparation**

Vacuum cups are used to handle the glass. Figure A-10 shows the encasement glass being lowered into the clamp plate, which already has o-rings installed. The glass is then held to the clamp plate during assembly with vacuum cups modified modified to capture the clamp plate. These allow the clamp plate and glass to be installed on the encasement as an assembly, while retaining the o-rings in the clamp plate.

Any streaks or dust left on the inside of the glass will be visible and difficult to fix. The glass should be given a final wipe with a cleanroom wipe soaked with a 50/50 solution of deionized water and ethanol, as shown in Figure A-11.

### **A.2.6 Platen preparation**

If a backing board will be used between the document and the platen, these should be cut to shape before the platen is assembled. To cut the backing board, the platen should be placed on top of the backing board. The outline can then be trimmed with a razor, which will give the visible edge a finished appearance.



Figure A-10: The glass is lowered onto the clamp plate, after the o-rings are installed in the clamp plate.



Figure A-11: Cleaning glass just before final assembly.

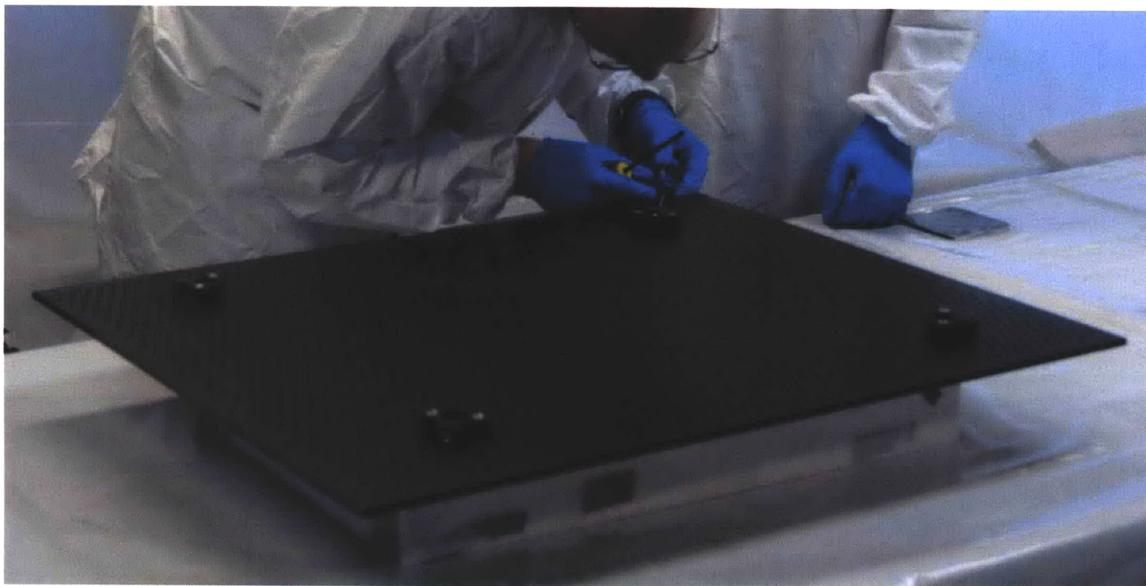


Figure A-12: The platen clips are adjusted.

Next, the platen clips should be installed and their fasteners torqued. The ball plungers should then be adjusted until the platen attaches with a “satisfying click”, but can be removed without undue force. Figure A-12 shows the platen clips being adjusted. The hollow-lock fasteners can then be tightened to secure the ball plunger position.

The backing and document are then be placed on the platen. As shown in Figure A-13, small weights were used to hold the document in place while the document clips were installed. Also note that the platen is resting on a stand to allow access to the bottom of the platen, allowing document clips to be installed.

The lead conservator wanted the document to be allowed to assume its final shape on its own, so no attempt was made to flatten the document with the clip placement. After the conservator was satisfied with the clip placement, the document clip retainers were pushed in and tightened.

### **A.2.7 Final assembly**

To achieve low helium leak rates, cleanliness at the assembly level is critical. A temporary “cleanroom” was set up at the Massachusetts Archives using heavy plastic sheeting. This room had a plastic floor, ceiling, and walls, and seams were duct taped

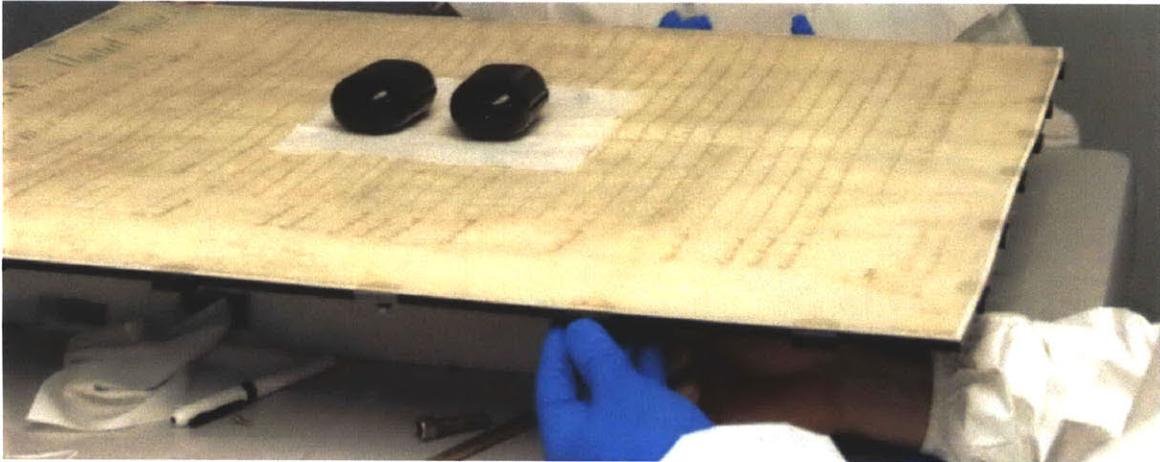


Figure A-13: Installation of document clips onto the Bill of Rights.

closed. An air filter and a humidifier or dehumidifier (depending on the season) were placed in the cleanroom to condition the air.

Final preparation work for the platen and glass was performed in this cleanroom. This included final cleanings of the tub, platen, and glass/clamp plate assembly, as well as installation of the document onto the platen and installation of the o-rings into the tub.

Dust control measures included using compressed argon and/or “air in a can” to blow dust off of sealing surfaces and carefully executing a preplanned assembly order. Because the encasement is at an angle, putting a bolt in the top row can drop dust into the sealing grooves. Instead, a “zipper” approach was employed, starting with the middle of the bottom row of bolts, where any dust generated would fall away from the seals. Two crews then installed bolts, working from bottom to top, installing bolts on both sides and snugging them enough to ensure contact between the seal and glass. After all the bolts were installed and made hand-tight, they were then torqued in increments of 10 N – m to the final torque of 40 N – m. With all of the bolts torqued, the encasement assembly can be purged and helium leak tested.



Figure A-14: The platen is installed in the tub.



Figure A-15: The glass and clamp assembly is installed on the tub.

Table A.2: Rotameter correlation data for argon and helium, using approximate correction factors.

Rotameter reading (mm)	Air 0 psig, 70 °F (cc/min)	Argon 60 psig, 70 °F (cc/min)	Helium 60 psig, 70 °F (cc/min)
65	986	1890	5990
60	913	1750	5550
55	836	1610	5080
50	757	1450	4600
45	679	1300	4130
40	596	1150	3620
35	513	985	3120
30	427	820	2600
25	336	645	2040
20	241	463	1470
15	156	300	948
10	78	150	474
5	23	44	140

### A.3 Moisturematic operation

#### Determining setpoints

The setpoints to generate the correct humidity can be determined from the equations in Chapter 6. To generate argon humidified to 40 %RH, the saturator temperature and pressure were set to 25 °C (77 °F) and 324.8 kPa (47.1 psia) respectively.

It is also useful to determine the target rotameter readings ahead of time. Reference scale rotameters are supplied with a table correlating a float position to a flow rate of air or water. Table A.2 shows the table supplied with the Moisturematic rotameter for room-temperature, atmospheric-pressure air, along with correlations for argon and helium at 60 psig, using the methods shown in [30]. These correction factors are approximate.

#### Initializing

1. Open the saturator by loosening the top clamp. Check that water level covers sightglass. Add distilled water if necessary. Check that diffuser is present.

2. Reassemble saturator and check that both clamps are hand tight.
3. Connect hoses to encasement. Observe flow direction.
4. Verify that encasement valves are open.
5. Check position of switchover valve.
6. Make sure crossover valve is set to "idle".
7. Open gas cylinder and set regulator output pressure (60 psig is suitable for down to about 35 %RH, but needs to be set higher for lower humidities).
8. Plug in unit. Controls should automatically initialize.
9. Check/set saturator temperature and pressure setpoints.
10. Set metering valve to desired flow rate as measured with the rotameter.
11. Check adjustment of back pressure regulators (BPRs). Cover each outlet port with a finger and watch the diaphragm gauges. Make adjustments on the other BPR. The upper BPR sets the back pressure, and the bottom BPR sets the overpressure relief. It is recommended that the top BPR should be set to 5" W.C. (water column), and the bottom BPR set to 10" W.C.
12. When temperature and pressure has stabilized, the machine is ready. Flow rate may be reduced if machine is to idle for a while, but ensure flow rate is set and readings are steady before beginning purge.

### **Purging**

1. Ensure the encasement valves are open.
2. Rotate the crossover valve to the "purge" position.
3. Verify that gas is flowing through the loop. If the diaphragm gauges both show the same back pressure (set previously) then gas should be flowing. If the lower

BPR shows a significantly higher pressure, an encasement valve may be closed or other restriction may be present.

4. Leave the purge running for the required length of time (4 hours is typical).

### **Pausing the purge**

1. Simply turn the crossover valve to “idle.” Continue on to helium leak checking if appropriate, or restart the purge by turning the valve to “purge”

### **Performing the helium leak test**

Refer to Section A.4.

### **Shutdown (temporarily—up to a few weeks)**

1. Close cylinder valves and back off cylinder regulators.
2. Unplug unit.

### **Shutdown and cleanup (for storage)**

1. Open drain valve on saturator (have a container ready). Let run for several minutes.
2. Close cylinder valves and back off cylinder regulators.
3. Unplug unit.
4. Open the top and bottom of the saturator.
5. Thoroughly dry with a sponge, compressed air, hair dryer, etc. The air stone is difficult to dry, and can be removed instead.
6. Reassemble saturator to keep it clean until the next use.

## A.4 Helium leak testing

Since there are two main seals to be tested, helium leak testing is a two step process. First, the inner seal is tested, followed by the outer seal. The helium leak detector can remain connected to the inner sensing groove for both steps.

### Preparation

First, the helium leak detector and encasement should be prepared:

1. Make sure helium leak detector is warmed up.
2. Calibrate helium leak detector (follow manufacturer's instructions).
3. Connect the helium leak detector to the inside sensing groove.
4. Make sure sensing groove valves are open.
5. Press "test" on the helium leak detector.
6. Ensure background helium levels are below  $1 \times 10^{-8}$  atm – cc/sec.
7. If background helium levels remain high for a long time, "cleanup" may be accelerated by using a tee fitting to evacuate both sensing grooves simultaneously. Such an attachment is shown in Figure A-16.

### Inner Seal

Next, the helium leak rate of the inner seal should be measured:

1. Isolate getter bottle by turning off getter bottle valve.
2. Make sure Moisturematic is set to "idle"
3. Ensure helium regulator is set and cylinder valve is open.
4. Toggle switchover valve.
5. Wait for helium to displace argon in Moisturematic (~5 minutes).

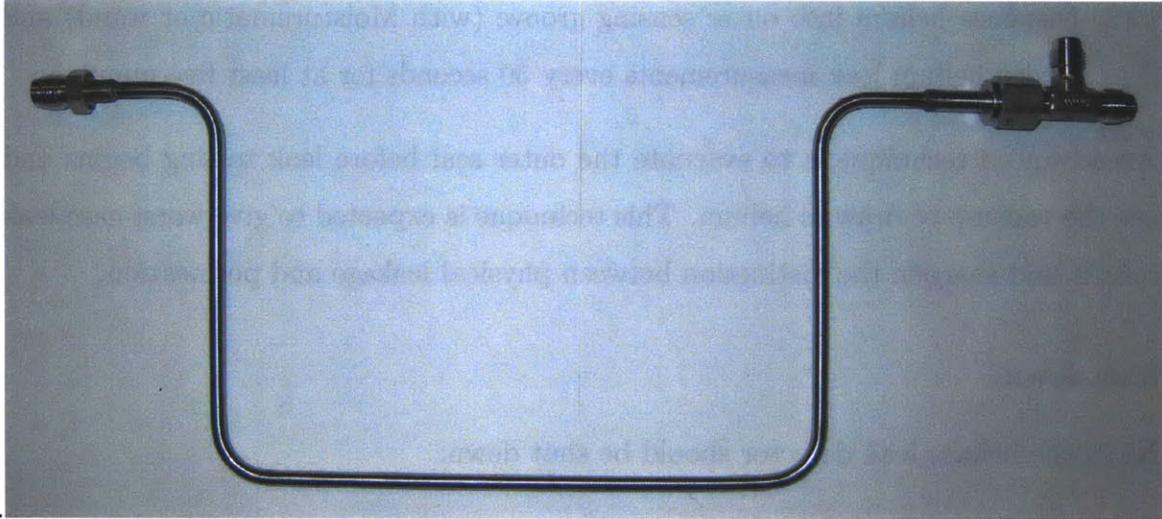


Figure A-16: Tee fitting for evacuating sensing grooves simultaneously, used for fast reduction of helium background levels.

6. Set flow rate (15.5 mm on rotameter for 1 L/min of helium). Note that the rotameter correlations are different for argon and helium.
7. Check saturator temperature. The temperature may be upset by switching the gas. Wait if necessary.
8. Set Moisturematic to “purge”, wait a predetermined amount of time (*e.g.*, one minute), and set back to “idle”. This will dispense a measured volume of helium.
9. Close encasement valves (“finger tight”).
10. Record helium leak measurements every 30 seconds for at least five minutes.
11. Close encasement valves with wrench (“snug”).
12. Install VCR<sup>®</sup> plugs (with new gaskets) on the encasement inlet and outlet.

### Outer seal

Next, the helium leak rate of the outer seal should be measured:

1. Before measuring the helium leak rate of the outer seal, the leak rate for the inner seal must stabilize. Helium can be purged from the encasement with more argon if necessary.

2. Dispense helium into outer sensing groove (with Moisturematic or wand) and record helium leak measurements every 30 seconds for at least five minutes.

An advanced technique is to evacuate the outer seal before leak testing begins and use the vacuum to draw in helium. This technique is expected to give worst case leak results and sharpen the distinction between physical leakage and permeation.

### **Shutdown**

Next, the helium leak detector should be shut down:

1. Vent helium leak detector.
2. Turn off helium leak detector.
3. Disconnect the hose at both ends.
4. Cap inlet of helium leak detector.

### **Purge sensing grooves**

The final step is to purge the inner and outer sensing grooves:

1. Set the Moisturematic to dispense argon.
2. Connect the Moisturematic to each of the sensing grooves and let purge for five minutes. Open the getter bottle valve slowly while purging, maintaining a positive pressure in the system.
3. When done with each groove, shut the encasement valves, then disconnect the Moisturematic.
4. Plug the open ends of the encasement plumbing with VCR<sup>®</sup> plugs (with new gaskets).
5. Continue with Moisturematic shutdown instructions.

# Appendix B

## Humidity Sensor Code

```
1 #include <p18F2410.h>
  #include <stdio.h>
3 #include <delays.h>
  #include "usart.h"
5 // #define DEBUG
  #define RELEASE
7
  #ifdef DEBUG
9 #define SCK LATBbits.LATB1
  #define SDA TRISBbits.TRISB0
11 #define SDA_read PORTBbits.RB0
  #define TRISBmask 0b00000001
13 #endif

  #ifdef RELEASE
15 #define SCK LATBbits.LATB7
17 #define SDA TRISBbits.TRISB6
  #define SDA_read PORTBbits.RB6
19 #define TRISBmask 0b01000000
  #endif
21
  #define LED_ON LATAbits.LATA4=0;
23 #define LED_OFF LATAbits.LATA4=1;

25 #define CHECK_TEMP 0b00000011
  #define CHECK_HUMD 0b00000101
27 #define CHECK_STAT 0b00000111
  #define WRITE_STAT 0b00000110
29 #define RESET      0b00011110

31 void init(void);
  void main(void);
33 void InterruptHandlerHigh(void);
  void error_handler(void);
35 void RX_handler(void);
  void sht7x_start(void);
37 void sht7x_reset(void);
  unsigned int sht7x_read(unsigned char mode);
```

```

39 void output(unsigned int temperature, unsigned int humidity, unsigned
    char mode);
    unsigned int sht7x_read_byte16(void);
41 enum {TEMP,HUMD, MACH, RAW};
    char RXbuf;
43
44 union {
45     struct {
46         unsigned Binary:1;
47         unsigned Raw:1;
48         unsigned degF:1;
49         unsigned Error:1;
50         unsigned tempcorr:1;
51         unsigned verbose:1;
52         unsigned none:3;
53     } Bit;
54     unsigned char Byte;
55 } Flags;

57 void main () {
    unsigned int temperature, humidity;
59 init();
    LED_ON;
61 while (1) {
    TOCON &= ~0x80;
63     Flags.Bit.Error=0;
    RXbuf=0;
65     LED_ON;
    while(RXbuf==0);
67     LED_OFF;
    TMR0H = 0;
69     TMR0L = 0;
    TOCON |= 0x80;
71     switch (RXbuf) {
        case('m'):
73         temperature=sht7x_read(TEMP);
            humidity=sht7x_read(HUMD);
75         output(temperature, humidity, MACH);
            break;
77         case('r'):
            temperature=sht7x_read(TEMP);
79             humidity=sht7x_read(HUMD);
            output(temperature, humidity, RAW);
81             break;
            case('h'):
83             temperature=sht7x_read(TEMP);
                humidity=sht7x_read(HUMD);
85             output(temperature, humidity, HUMD);
                break;
87             case('s'):
                output(6000, 1200, MACH);
89                 break;
            case('t'):
91             temperature=sht7x_read(TEMP);

```

```

    output(temperature, humidity, TEMP);
93     break;
    case('R'):
95     sht7x_reset();
        printf("connection reset\r\n");
97     break;
    case('C'):
99     Flags.Bit.degF=0;
        printf("deg C set\r\n");
101    break;
    case('F'):
103    Flags.Bit.degF=1;
        printf("deg F set\r\n");
105    break;
    }
107 }
}
109
#pragma code InterruptVectorHigh = 0x08
111 void InterruptVectorHigh (void) {
    _asm
113 goto InterruptHandlerHigh //jump to interrupt routine
    _endasm
115 }

117 #pragma code
#pragma interrupt InterruptHandlerHigh
119
void InterruptHandlerHigh () {
121 if (INTCONbits.TMR0IF) {
    INTCONbits.TMR0IF = 0;
123     error_handler();
    }
125 if(PIR1bits.RCIF)
    RX_handler();
127 }

129 void RX_handler(void) {
    RXbuf = ReadUSART();
131 }

133 void error_handler(void) {
    Flags.Bit.Error=1;
135 }

137 void sht7x_start(void) {
    SCK = 1;
139     Delay10TCYx(1);
    SDA = 0;
141     Delay10TCYx(1);
    SCK = 0;
143     Delay10TCYx(2);
    SCK = 1;
145     Delay10TCYx(1);

```

```

    SDA = 1;
147   Delay10TCYx(1);
    SCK = 0;
149   Delay10TCYx(1);
}
151
void sht7x_send_byte(unsigned char sht7x_command) {
153 static unsigned command;
unsigned char i;
155 command=sht7x_command;
for(i = 0 ; i < 8 ; i++) {
157   Rlcf(command,1,1);
    SCK = 0;
159   Delay10TCYx(1);
    SDA = STATUSbits.C;
161   LATAbits.LATA4=STATUSbits.C;
    Delay10TCYx(1);
163   SCK = 1;
    Delay10TCYx(1);
165   }
    SCK = 0;
167   SDA = 1;
    Delay10TCYx(1);
169   while (SDA.read == 1 && !Flags.Bit.Error);
    SCK = 1;
171   Delay10TCYx(2);
    SCK = 0;
173   Delay10TCYx(1);
    while (SDA.read == 0 && !Flags.Bit.Error);
175   while (SDA.read == 1 && !Flags.Bit.Error);
}
177
unsigned int sht7x_read(unsigned char mode) {
179 sht7x_start();
switch(mode){
181   case TEMP : sht7x_send_byte(CHECKTEMP); break;
    case HUMD : sht7x_send_byte(CHECKHUMD); break;
183   default : break;
}
185 return (sht7x_read_byte16());
}
187
void output(unsigned int temperature, unsigned int humidity, unsigned
char mode) {
189 float humidity_f, temperature_f;
unsigned char i, i2;
191 if (Flags.Bit.Error) {
    printf(" error!\r\n");
193   sht7x_reset();
    TOCON &= ~0x80;
195   return;
}
197 if (mode==RAW) {
    printf("%i%%i\r\n", humidity, temperature);
}

```

```

199     return;
    }
201 if (mode==HUMD || mode==MACH){
    humidity_f = -2.0468+.0367*humidity-0.0000015955*humidity*humidity
        +.05;
203     temperature_f = -40.1+temperature*.01+.05;
    humidity_f += (temperature_f-25)*(0.01*0.00008*humidity);
205     i = (char) humidity_f;
    i2 = (char) ((humidity_f-i)*10);
207 }
    if (mode==HUMD)
209         printf("humidity is %d.%01d%%\r\n", i, i2);
    if (mode==MACH)
211         printf("%d.%01d%%", i, i2);
    if (mode==TEMP && !Flags.Bit.degF)
213         temperature_f = -40.1+temperature*.01+.05;
    if (Flags.Bit.degF)
215         temperature_f = -40.2+temperature*.018+.05;
    i = (int) temperature_f;
217 i2 = (int) ((temperature_f-i)*10);
    if (mode==HUMD || mode==TEMP)
219         if (!Flags.Bit.degF)
            printf("temperature is %d.%01d C\r\n", i, i2);
221         else
            printf("temperature is %d.%01d F\r\n", i, i2);
223 if (mode==MACH)
    printf("%d.%01d\r\n", i, i2);
225 }

227 unsigned int sht7x_read_byte16(void) {
    unsigned char j;
229 unsigned int in_byte=0;
    for(j = 0 ; j < 8 ; j++) {
231         SCK = 0;
            Delay10TCYx(2);
233         SCK = 1;
            in_byte=in_byte<<1;
235         in_byte += SDA_read;
            }
237 SCK=0;
    Delay10TCYx(1);
239 SDA=0;
    Delay10TCYx(1);
241 SCK=1;
    Delay10TCYx(2);
243 SCK=0;
    SDA=1;
245 Delay10TCYx(1);
    for(j = 0 ; j < 8 ; j++) {
247         SCK = 0;
            Delay10TCYx(2);
249         SCK = 1;
            in_byte=in_byte<<1;
251         in_byte += SDA_read;

```

```

    }
253 SCK=0;
    Delay10TCYx(1);
255 SCK=1;
    Delay10TCYx(1);
257 SCK=0;
    return(in_byte);
259 }

261 void sht7x_reset()
    {
263 unsigned char i;
    SDA = 1;
265 Delay10TCYx(1);
    for(i = 0 ; i < 8 ; i++) {
267     SCK = 0;
        Delay10TCYx(1);
269     SCK = 1;
        Delay10TCYx(1);
271     }
    SCK = 0;
273 }

275 void init()
    {
277 SCK=0;
    TRISB = TRISBmask;
279 Flags.Byte = 0;
    INTCON = 0x20;
281 INTCON2 = 0x04;
    RCONbits.IPEN = 1;
283 TMR0H = 0;
    TMR0L = 0;
285 TOCON = 0x06;
    TRISA = 0;
287 LATB = 0;
    ADCON1=0x0F;
289 IPR1bits.RCIP = 1;
    OpenUSART(USART_TX_INT.OFF &
291     USART_RX_INT.ON &
        USART_ASYNC.MODE &
293     USART_EIGHT_BIT &
        USART_CONTRX &
295     USART_BRGH.HIGH, 77);
    INTCONbits.GIEH = 1;
297 Flags.Bit.degF=1;
    Flags.Bit.tempcorr=1;
299 }

```

main.c

# Appendix C

## Supplier Information

A&N Corporation  
707 SW 19th Avenue  
Williston, FL 32696  
(800) 352-6431  
[www.ancorp.com](http://www.ancorp.com)

Advanced Circuits  
21101 E. 32nd Parkway  
Aurora, CO 80011  
(800) 979-4722  
[www.4pcb.com](http://www.4pcb.com)

Alconox, Inc.  
30 Glenn St. Suite 309  
White Plains, NY 10603  
(914) 948-4040  
[www.alconox.com](http://www.alconox.com)

Alexandria Metal Finishers  
9418 Gunston Cove Rd  
Lorton, VA 22079  
(703) 643-1636  
[www.alexandriametalfinishers.com](http://www.alexandriametalfinishers.com)

Alicat Scientific, Inc.  
7641 N. Business Park Drive  
Tucson, AZ 85743  
(520) 290-6060  
[www.alicatscientific.com](http://www.alicatscientific.com)

Apple Steel Rule Die Co., Inc.  
7817 W. Clinton Avenue  
Milwaukee, WI. 53223  
(800) 227-3437  
[www.appledie.com](http://www.appledie.com)

Aquatic Eco-Systems Inc.  
2395 Apopka Blvd. Suite 100  
Apopka, FL 32703  
(877) 347-4788  
[www.aquaticeco.com](http://www.aquaticeco.com)

Astrodyne Corporation  
35 Hampden Road  
Mansfield, MA 02048  
(800) 823-8082  
[www.astrodyne.com](http://www.astrodyne.com)

The Bechdon Company, Inc.  
300 Commerce Drive  
Upper Marlboro, MD 20774  
(301) 249-0900  
[www.bechdon.com](http://www.bechdon.com)

Cambridge Valve and Fitting, Inc.  
50 Manning Rd  
Billerica, MA 01821  
(781) 272-8270  
<http://www.swagelok.com/cambridge>

DataPro International Inc.  
1144 N.W. 52nd St.  
Seattle, WA 98107  
(206) 782-5259  
[www.datapro.net](http://www.datapro.net)

DLP Design, Inc.  
1605 Roma Lane  
Allen, TX 75013  
(469) 964-8027  
[www.dlpdesign.com](http://www.dlpdesign.com)

DuPont Teijin Films  
3600 Discovery Drive  
Chester, VA 23836  
(800) 635-4639  
[www.dupontteijinfilms.com](http://www.dupontteijinfilms.com)

Emerson Process Management  
Regulator Technologies Inc.  
McKinney, TX 75069  
(800) 558-5853  
[www.emerson.com](http://www.emerson.com)

Grainger W.W. Inc.  
100 Grainger Parkway  
Lake Forest, IL 60045  
(800) 323-0620  
[www.grainger.com](http://www.grainger.com)

Greene, Tweed & Co., Inc.  
2075 Detwiler Rd. P.O. Box 305  
Kulpsville, PA 19443  
(215) 256-9521  
[www.gtweed.com](http://www.gtweed.com)

Hammond Manufacturing Co. Inc.  
475 Cayuga Rd.  
Cheektowaga, NY 14225  
(716) 630-7030  
[www.hammfg.com](http://www.hammfg.com)

LADD Industries  
4849 Hempstead Station Drive  
Kettering, Ohio 45429  
(800) 223-1236  
[www.laddinc.com](http://www.laddinc.com)

Lantronix, Inc.  
167 Technology Drive  
Irvine, CA 92618  
(800) 526-8766  
[www.lantronix.com](http://www.lantronix.com)

McMaster-Carr Supply Company  
200 New Canton Way  
Robbinsville, NJ 08691  
(609) 689-3415  
[www.mcmaster.com](http://www.mcmaster.com)

Mill-Max Mfg. Corp.  
190 Pine Hollow Rd. P.O. Box 300  
Oyster Bay, NY 11771  
(516) 922-6000  
[www.mill-max.com/](http://www.mill-max.com/)  
MKS Instruments  
2 Tech Drive, Suite 201  
Andover, MA 01810  
(978) 645-5500  
[www.mksinst.com](http://www.mksinst.com)

National Instruments  
11500 N. Mopac Expwy  
Austin, TX 78759  
(888) 280-7645  
[www.ni.com](http://www.ni.com)

Omega Engineering, Inc.  
1 Omega Drive P.O. Box 4047  
Stamford, CT 06907  
(800) 848-4286  
[www.omega.com](http://www.omega.com)

Schott North America, Inc.  
555 Taxter Road  
Elmsford, NY 10523  
(914) 831-2200  
[www.us.schott.com](http://www.us.schott.com)

Sensirion AG  
Laubisruetistrasse 50  
8712 Staefa ZH  
Switzerland  
+41 44 306 40 00  
[www.sensiron.com](http://www.sensiron.com)

Solid Sealing Technology, Inc.  
44 Dalliba Avenue  
Watervliet, NY 12189  
(518) 874-3600  
[www.solidsealing.com](http://www.solidsealing.com)

University Products, Inc.  
517 Main Street  
Holyoke, MA 01040  
(800) 628-1912  
[www.universityproducts.com](http://www.universityproducts.com)

Swagelok Company  
31400 Aurora Road  
Solon, OH 44139  
(440) 349-5800  
[www.swagelok.com](http://www.swagelok.com)



# Bibliography

- [1] Eric Hansen. *Oxygen Free Museum Cases*, chapter Protection of Objects from Environmental Deterioration by Reducing Their Exposure to Oxygen, pages 7–16. J. Paul Getty Trust, 1998.
- [2] Nieves Valentín. *Oxygen Free Museum Cases*, chapter Preservation of Historic Materials by Using Inert Gases for Biodeterioration Control, pages 17–30. J. Paul Getty Trust, 1998.
- [3] Nathan Stolow. *Conservation and Exhibitions: Packing, Transport, Storage, and Environmental Considerations*. Butterworth-Heinemann, 1987.
- [4] NOVA. Saving the national treasures. [http://www.pbs.org/wgbh/nova/transcripts/3206\\_charters.html](http://www.pbs.org/wgbh/nova/transcripts/3206_charters.html). URL last checked 2011-1-14.
- [5] National Archives and Records Administration. Rubenstein magna carta: Conservation requirements for a new encasement. Private Communication, 2009.
- [6] Donald Etherington. Custom enclosures and encasements for historic artifacts. <http://www.archives.gov/preservation/conferences/2005/etherington.ppt>, September 2005. Proceedings of the 19th Annual Preservation Conference. URL last checked 2011-1-14.
- [7] Nasry Yousef Iskander. *Oxygen Free Museum Cases*, chapter Controlled-Environment Cases for the Royal Mummy Collection, pages 47–52. J. Paul Getty Trust, 1998.

- [8] Hari Kishan and Shin Maekawa. *Oxygen Free Museum Cases*, chapter Preservation of the Original Documents of the Constitution of India, pages 53–58. J. Paul Getty Trust, 1998.
- [9] Richard Rhorer. Encasement fabrication and testing: Processes and innovations. <http://www.archives.gov/preservation/conferences/2005/rhorer.ppt>, September 2005. Proceedings of the 19th Annual Preservation Conference. URL last checked 2011-1-14.
- [10] Orvis M. Knarr. *Industrial gaseous leak detection manual*. McGraw-Hill, 1998.
- [11] ERIKS Sealing Elements Technical Handbook O-rings. [http://o-ring.info/en/o-ring/Oring-Handbook/ERIKS\\_SealingElements\\_TechnicalHandbook\\_O-rings.pdf](http://o-ring.info/en/o-ring/Oring-Handbook/ERIKS_SealingElements_TechnicalHandbook_O-rings.pdf). URL last checked 2011-1-14.
- [12] Parker o-ring handbook. [http://o-ring.info/en/o-ring/Oring-Handbook/ERIKS\\_SealingElements\\_TechnicalHandbook\\_O-rings.pdf](http://o-ring.info/en/o-ring/Oring-Handbook/ERIKS_SealingElements_TechnicalHandbook_O-rings.pdf), 2007. Publication ORD 5700. URL last checked 2011-1-14.
- [13] R.N. Peacock. Practical selection of elastomer materials for vacuum seals. *J. Vac. Sci. Technol.*, 17(1):330–336, 1980.
- [14] L. Laurenson and N.T.M. Dennis. Permeability of common elastomers for gases over a range of temperatures. *J. Vac. Sci. Technol. A*, 3(3):1707–1710, 1985.
- [15] W.G. Perkins. Permeation and outgassing of vacuum materials. *J. Vac. Sci. Technol.*, 10(4):543–556, 1973.
- [16] T. Provost M.L. Johnson, D.M. Manos. Gas permeation and leakage. *J. Vac. Sci. Technol. A*, 15(3):763–767, 1997.
- [17] Richard Rhorer. Metal seals discussion. Private Communication, 2007.
- [18] Nishith Verma Stephen L. Gilbert Farhang Shadman Ce Ma, Eric Shero. Permeation of moisture and oxygen through polymeric o-rings. *Journal of the Institute of Environmental Sciences*, MARCH/APRIL:43–46, 1995.

- [19] Alexander H. Slocum. *Precision Machine Design*. Society of Manufacturing Engineers, 1992.
- [20] 3M Interconnect Solutions Mating Guide. <http://multimedia.3m.com/mws/mediawebserver?66666UuZjcFSLXTtmxTtnXTyEVuQEcuZgVs6EVs6E666666-->. Publication TS-2203-A. Last Modified 2007-10-10. URL last checked 2011-1-14.
- [21] S. Hasegawa and J.W. Little. The NBS two-pressure humidity generator, mark 2. *Journal of Research of the National Bureau of Standards- A. Physics and Chemistry*, 81A(1):81–88, 1977.
- [22] A.L. Buck. New equations for computing vapor pressure and enhancement factor. In *J. Appl. Meteor*, volume 20, pages 1527–1532. 1981.
- [23] Excel Engineering. X Steam 2.6. [http://www.x-eng.com/XSteam\\_Excel.htm](http://www.x-eng.com/XSteam_Excel.htm). URL last checked 2011-1-14.
- [24] M. Rigby and J. M. Prausnitz. Solubility of water in compressed nitrogen, argon, and methane. *The Journal of Physical Chemistry*, 72:330–334, 1968.
- [25] Gerald Koglbauer and Martin Wendland. Water vapor concentration enhancement in compressed humid nitrogen, argon, and carbon dioxide measured by fourier transform infrared spectroscopy. *J. Chem. Eng.*, 53:77–82, 2008.
- [26] H. Kitano, T. Niwa, N. Ochi, and C. Takahashi. Saturator efficiency and uncertainty of NMIJ two-pressure two-temperature humidity generator. *International Journal of Thermophysics*, 29(5):1615–1622, 2008.
- [27] Bruce Mayer, Craig C. Collins, and Mike Walton. Transient analysis of carrier gas saturation in liquid source vapor generators. *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films*, 19(1):329–344, 2001.
- [28] Swagelok Company. Valve sizing technical bulletin. <http://www.swagelok.com/downloads/webcatalogs/en/ms-06-84.pdf>, December 2007. Publication MS-06-84. URL last checked 2011-1-14.

- [29] Swagelok Company. An installer's pocket guide for VCR® metal gasket face seal fittings and micro-fit® weld fittings. <http://www.swagelok.com/downloads/webcatalogs/EN/MS-13-150.PDF>. Publication MS-13-150. URL last checked 2011-1-14.
- [30] Gilmont Instruments. Catalog. [http://www.grayledge.com/files/Gilmont\\_Instruments\\_Catalog.pdf](http://www.grayledge.com/files/Gilmont_Instruments_Catalog.pdf). URL last checked 2011-1-14.