A Two-Pressure Humidity Generator

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A device for precisely humidifying a process gas is presented, with particular application to document preservation. The machine is designed to minimize the risk of damage from overpressure by incorporating multiple safety mechanisms and a simple user interface. The design theory is presented, discussed, and verified. This device is capable of generating humidified gases with relative humidities between 7% and 96% to high accuracy. At conditions suitable for document preservation, the machine is accurate to within ±3.5%, *generating a relative humidity of* (40 ± 1.4)%. [DOI: 10.1115/1.4007302]

1 Introduction

To display a historic document while minimizing decay, it must be enclosed in a sealed encasement that controls the temperature and composition of the atmosphere within the encasement. The atmosphere is typically a humidified inert gas such as argon. Several commercially available machines produce a humidified gas; however, many suffer from a lack of accuracy and/or portability, and none of them protect from overpressure. Document encasements often have a large glass front, the area of which makes the encasement sensitive to differential pressure. A suitably designed filling apparatus must be unable to overpressure an encasement—10 kPa above atmospheric pressure is a conservative limit for most encasement designs.

We present a device capable of generating humidified gas using a method often employed for calibrating humidity measurement instruments. Our device has simple controls and is designed to be "inherently safe," in that multiple operational and mechanical failures must occur for an overpressure situation to occur. In addition, our device is compact and portable. This device was developed and employed for filling encasements designed and built for the Massachusetts Archives.

These basic design parameters are not unique to document preservation. Laboratory settings often contain experiments constructed of glassware, where overpressure would create a dangerous situation. Such a humidity-generating device could thus find applications in many industrial and research environments.

2 Humidity Generation Methods

A common method of generating a gas with a known relative humidity (RH) is to saturate a stream of gas and then expanding or heating the stream to reduce the relative humidity. Oftentimes, humidity-generating equipment will assume pressure or temperature to remain constant; most equipment lacks control over both temperature and pressure. While this simplifies both apparatus and analysis, precision applications demand that both temperature and pressure be controlled. The "two-temperature/two-pressure" method [1] accounts for all variables. Using this method, the relative humidity of a gas can be calculated as

$$\mathrm{RH} = \frac{f(P_s, T_s)}{f(P_c, T_c)} \times \frac{e_w(T_s)}{e_w(T_c)} \times \frac{P_c}{P_s} \tag{1}$$

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. 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 [2] were used. The enhancement factor f relates the partial pressure of a saturated gas to the saturation pressure of water alone. A literature review 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. Over a wide range of temperatures and pressures, the value of f is close to unity [3]. 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. We expect the effect to be smaller for argon, as seen in the data presented in Refs. [4,5]. We conclude that ignoring the enhancement factor will not result in significant error. Though we will assume $f = 1$ for calculating setpoints, the enhancement factor will be included in an error analysis in Sec. 4.4.

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 $T_c = 21.1\,^{\circ}$C.

Table 1 summarizes parameters suitable for humidifying a gas to a relative humidity of 40% at the desired temperature and pressure. Note that the pressure is slightly higher than atmospheric due to the use of a back pressure regulator in the humidity generator circuit.

3 Purge Parameters

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

$$\dot{V}_w = \dot{V}_d \frac{P_s}{(P_s - e_w(T_s))} \tag{2}$$

At room temperature, $\dot{V}_w$ and $\dot{V}_d$ are nearly equal. As saturator temperature increases, $e_w(T_s)$ 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

$$\frac{V}{\dot{V}_w}\frac{dP_{O_2}}{dt} + P_{O_2} = 0 \tag{3}$$

We then expect the oxygen concentration in the encasement to follow

$$P_{O_2}(t) = P_{O_2}(t_0) \cdot \left(1 - e^{-t(V/\dot{V}_w)}\right) \tag{4}$$

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 min. Time constants of 8.4 are needed to drive the oxygen concentration inside the encasement

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Table 1 Parameters suitable for generating gas with 40% RH

Variable	Symbol	Value
Desired temperature	T_c	21.1 °C
Desired pressure (abs)	P_c	102.6 kPa
Saturator temperature	T_s	25.0 °C
Saturator p.p. H_2O	$e_w(T_s)$	3.170 kPa
Encasement p.p. H_2O	$e_w(T_c) \cdot \text{RH}$	1.001 kPa
Saturator pressure (abs)	P_s	324.8 kPa

below 0.005%, 2 orders of magnitude less than the 0.5% maximum oxygen concentration specified by conservators. A purge time of 4 h is therefore 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 IAPWS-IF97 formulations. The mass of water consumed in a purge is

$$m_w = \rho_v(T_s) \cdot \text{RH} \cdot \dot{V}_w \cdot t \tag{5}$$

and gas consumption is, of course

$$V_t = \dot{V}_d \cdot t \tag{6}$$

4 Humidity Generator Design

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 machine is shown in Fig. 1, the detailed design of which is discussed in the subsequent sections.

4.1 Schematic and Description of Components. To begin the design, a list of functional requirements was generated:

(1) The machine should accurately humidify argon (or other inert gas).
(2) The machine should regulate the flow rate 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.

The functional requirements 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 Fig. 2.

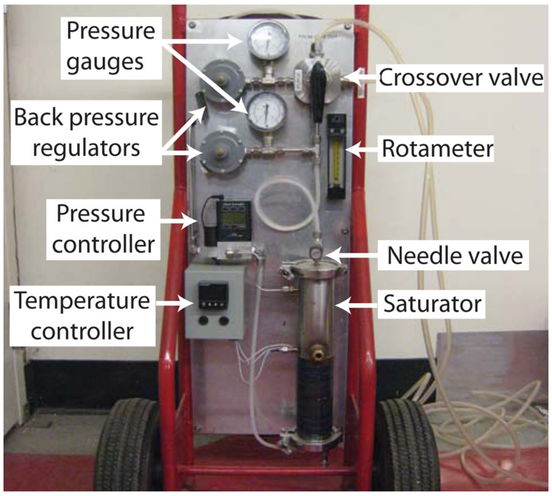

Fig. 1 Completed moisturematic

4.1.1 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 $200\ \text{ft}^3$ cylinder of argon and an $80\ \text{ft}^3$ cylinder of helium. The regulators used have a maximum outlet pressure of 690 kPa.

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

4.1.3 Rotameter. An Omega FL-3651 G-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 provides measurements from 0.19 l/min to 1.9 l/min of dry argon at 410 kPa.

4.1.4 Pressure Controller. An Alicat Scientific PC3-100PSIA-D digital pressure controller 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 690 kPa (absolute). The pressure controller requires 24 VDC for operation, which is supplied by an ICC/Elpac MSM0724 power supply.

4.1.5 Temperature Controller. An Omega CNI16D24-C24 digital 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.

4.1.6 Saturator. The custom built saturator is described in Sec. 4.3.

4.1.7 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.

4.1.8 Diaphragm Pressure Gauges. Two Omega PGL-25 L-35 gauges measure pressures in the inlet and outlet lines of the encasement. These gauges measure from 0 to 8.6 kPa.

4.1.9 Back Pressure Regulators (BPRs). Two Emerson 289U-4 BPRs, adjustable from 1.2 to 6.2 kPa, act as relief valves to limit pressure on both the inlet and outlet hoses. The outlet BPR creates a slight positive pressure in the encasement while purging. The inlet BPR prevents overpressure of the encasement in case of mechanical or operational failure.

4.1.10 Crossover Valve. A Swagelok SS-45YF4-1466 four-port crossover valve allows purging to be started or stopped with a 1/4 turn of a single lever. When the purge is stopped the encasement is isolated, but gas will still flow through the saturator and out of the BPRs, allowing warmup and adjustment.

4.2 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 machine operation to be checked and temperature/pressure setpoints to be verified. 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 stopped, only the volume of gas in the saturator will be dispensed. The risk

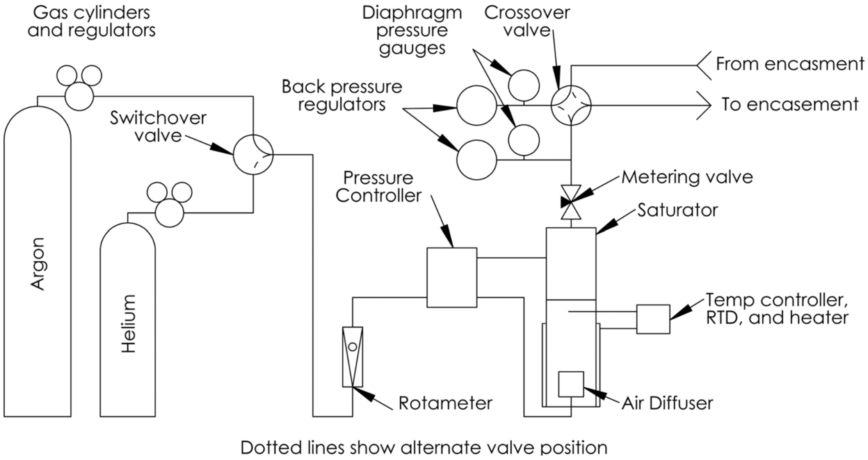

Fig. 2 Schematic of moisturematic

of an overpressure situation is mitigated by the redundancy of the relief valves and the reliability of the diaphragm valve design.

By far, the gravest danger to a document is a sustained purge with low saturator pressure, which would dispense gas with too high a humidity. This situation could arise if a purge was started without a high enough cylinder pressure. Given a 4 h long purge at 1.5 l/min, a purge should not be started if the cylinder pressure is below 1.4 MPa.

4.3 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: (1) a "bubbler" design where gas is bubbled through a column of water, or (2) 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 [1,6].

The bubbler saturator design was chosen for the moisturematic because data on saturation efficiency were readily available in Ref. [7]. 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 model is insensitive to changes in carrier gas, justifying the application of the results to a pressurized argon. 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. 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 saturator is constructed from stainless steel "Tri-Clamp®" fittings. These fittings provide a combination of features not available with other fittings; they can be quickly disassembled and reassembled without tools, they do not require sealants such as PTFE tape (which can plug small orifices), and they are available in relatively large diameters.

Fittings were welded on to the Tri-Clamp® caps for the inlet, outlet, and drain. The body of the saturator also has three additional ports for pressure sensing, temperature measurement, and a sightglass. The saturator is wrapped with a heating blanket for temperature regulation. The finished assembly is shown in Fig. 3. The saturator is filled such that the RTD is just under the surface of the water.

4.4 Operational Envelope and Accuracy. Using the specifications of the components listed above, we can determine the limits of operation for the moisturematic. Assuming $T_c = T_s$ and neglecting the enhancement factor, the range of humidities that can be generated can be directly calculated from the 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, but system accuracy would be affected. If lower humidities need to be generated, the pressure capability of the entire system must be considered.

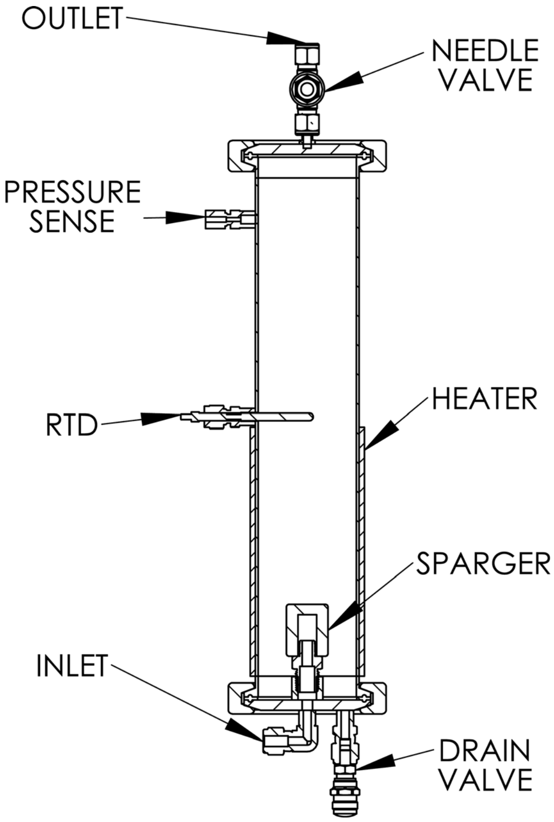

Fig. 3 Saturator assembly drawing

Table 2 Effect of estimated errors on humidity of output gas

Inputs	
Saturator pressure (T_s)	324.8 ± 1.7kPa
Saturator temperature (P_s)	25.0±0.2 °C
Encasement pressure (P_c)	102.61.0 kPa
Encasement temperature (T_c)	21.1 °C
Saturator efficiency	100^{+0}_{-1}
Enhancement factor (f)	$1.000^{+0.007}_{-0.000}$
Outputs	
RH (nominal)	40.0%
RH (maximum)	41.4%
RH (minimum)	38.6%

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. Combined with the minimum cracking pressure of 1.2 kPa and an atmospheric pressure of 101.4 kPa, the minimum saturator pressure is approximately 105 kPa, giving a maximum RH of about 96%.

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 2 shows the nominal setpoints corresponding to 40%RH @ 21.1 °C, estimated maximum errors, and the effects of those errors on output humidity as calculated using Eq. (1).

An alternative way to estimate the accuracy is to compute the Jacobian of Eq. (1)

$$J_{\mathrm{RH}}(T_{\mathrm{s}}, P_{\mathrm{c}}, P_{\mathrm{s}}) = \left[\frac{\partial(\mathrm{RH})}{\partial T_{\mathrm{s}}} \middle| \frac{\partial(\mathrm{RH})}{\partial P_{\mathrm{c}}} \middle| \frac{\partial(\mathrm{RH})}{\partial P_{\mathrm{s}}} \right] \tag{7}$$

Assuming the enhancement factors to be equal to 1, evaluating Eq. (7) at the suggested operating point from Table 2 results in

$$J_{\mathrm{RH}}(T_{\mathrm{s}}, P_{\mathrm{c}}, P_{\mathrm{s}}) = [0.0239\,^{\circ}\mathrm{C}^{-1} | 0.00390\,\mathrm{kPa}^{-1} | -0.00123\,\mathrm{kPa}^{-1}] \tag{8}$$

i.e., a 1 °C rise in saturator temperature will raise the output humidity from 40% to 42.4%. It can be seen that the uncertainties in saturator pressure and saturator temperature contribute approximately the same error.

5 Conclusion

The purpose of this project was to create a device for charging Massachusetts Archives encasements with humidified argon or helium. The safety and accuracy requirements are not unique to document preservation; the resulting design is generally applicable wherever a source of gas with known relative humidity or dewpoint is needed.

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Nomenclature

e_{w} = saturation pressure of water (kPa)
f = enhancement factor
m_{w} = mass of water used in one purge cycle
P = pressure (kPa)
P_{c} = encasement absolute pressure (kPa)
P_{s} = saturator absolute pressure (kPa)
$P_{\mathrm{O_2}}$ = partial pressure of oxygen in encasement (kPa)
RH = relative humidity
$\rho_{\mathrm{v}}(T_{\mathrm{s}})$ = density of saturated water vapor
t = length of purge
T = temperature (°C)
T_{c} = encasement temperature (°C)
T_{s} = saturator temperature (°C)
V = encasement volume
V_{t} = total volume of dry gas dispensed
$\dot{V}_{\mathrm{d}}$ = flow rate of dry gas
$\dot{V}_{\mathrm{w}}$ = flow rate of wet gas

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