

A Guide to Microclimate Leakage Testing

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INTRODUCTION:

Sealed exhibition cases, storage enclosures, and packing crates are used within museums to create stable microclimates for sensitive materials and/or objects, which require specific environmental conditions. They play an important role in maintaining a safe relative humidity, as well as protecting objects from gaseous pollutants and dust within the air.^[1] To successfully maintain a distinct environment from the surrounding air, these cases must remain airtight. This requirement is difficult to verify, as a system for testing leakage rates accurately and cost-effectively is still not established in most institutions.

In 2007, a study by Watts et al. discussed the importance for leakage testing, while noting the challenges museums face. Case manufacturers often test the prototype, but not each individual case shipped to museums. To verify the seal of each individual case, museums can contract a specialist company or carry out in-house testing. Specialist companies (such as BSRIA Ltd) cost at least 500 British pounds per case tested, or over 600 U.S. dollars. Watts notes that some museums will, as a result, select a few representative cases for testing, as opposed to doing a thorough survey. In comparison, when the museum opt for in-house testing, they lack a refined protocol for testing and, in the case of this study, failed to implement changes even once the leakage rate was calculate, lacking a process for locating the location of the leak.

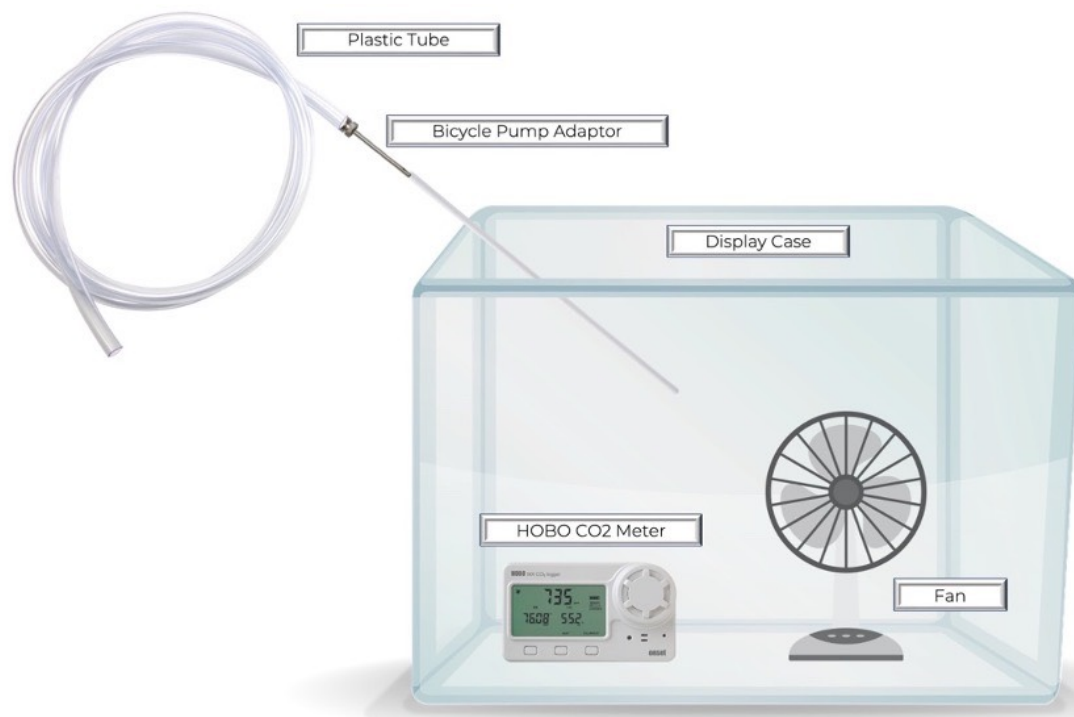
The purpose of this project is to provide a protocol for conservators or other museum employees seeking to quantify microclimate leakage rates, building upon past studies. Only few publications describe leakage testing, and those lack easily duplicatable procedures. Brimblecombe and Ramer describe leakage rate calculations in a very useful 1983 publication, but do not, however, provide a step-by-step guide. Calver et al. discuss leakage results, but likewise do not prioritize procedural details. In his 2019 American Institute for Conservation lecture and 2008 unpublished manuscript, Weintraub provides extensive descriptions of his calculations and available equipment. His work is foundational for this guide, which aims to condense past studies into a user-friendly format.

The procedures reviewed in this report will include: A) methods for dispensing CO₂ and monitoring its concentration levels, B) leakage rate and half-time calculations based on CO₂ measurements, and C) leakage detection equipment to find the point on the case where air is escaping.

As an introductory note, Carbon dioxide (CO₂) is the most frequently used tracer gas in leakage testing for several reasons. It is relatively inexpensive and easy to obtain, has a low concentration in the exhibition environment, and is generally safe gas to use. During this discussion, it is important not to confuse the leakage rate and half-time decay. As reciprocal values, an increase in case leakage results in an increase in k (leakage rate) and a decrease in $t_{1/2}$ (half-time decay rate). Also note that it is not necessary to know the specific half-time rate of decay rate ($t_{1/2}$) to determine the leakage rate.

^[1] Thomson, 1984.

PROCEDURE:



Part A: Dispensing and Monitoring CO₂

- 1) Measure CO₂ levels in the room where the case is held. Ideally, this could incorporate the average over several days.
 - 2) Place a CO₂ meter within the enclosure. One with Bluetooth capabilities (such as the battery-powered Hobo MX1102A CO₂ meter) is recommended for continuous monitoring, since it allows the collection of data in real time without opening the case.
 - 3) Adding a fan can speed up time it takes for CO₂ levels to reach equilibrium, especially within large cases. One that is remotely powered allows it to be easily incorporated without breaking the case seal. The fan should only operate until an equilibrium is achieved, because after this, it will exaggerate the leakage rate.^[2]
 - 4) Raise the concentration of CO₂ within the enclosure. Ensure that the concentration is significantly higher than surrounding levels, while also remaining aware of the upper range of the CO₂ monitor. The Hobo CO₂ meter for this study, for example, maxes out at 5,000 ppm, so the case levels were raised to around 4,000 ppm. For smaller cases, the CO₂ can rise simply by breathing through a straw. For larger enclosures, CO₂ dispensers or carbonation units can be attached to a plastic tube, which is fed into the case (see Appendix B for recommended equipment). Weintraub uses a bicycle pump adaptor to decrease the size of the plastic tube to fit tight edges or small holes in the case.
 - 5) Running the test for a few hours should be ample time for smaller cases. For larger cases and those with lower leakage rates, around 24 to 48 hours allows for a more significant change in CO₂ concentration to be observed.
 - 6) Once the data is collected, export the data for leakage rate calculations.
- ^[2] For more information, see research by Joy Bloser and Shannon Mulshine.

Part B: Leakage Rate and Half-time Calculations

There are two simple ways to calculate leakage rate, one by selecting two CO₂ values and plugging them into an equation, and the other using Excel to find the average rate of change over the series of datapoint. Both are based on the same principle (explored in Appendix B), and the steps are outlined below.

Method 1: Two Points

- 1) Select two CO₂ levels, one towards the beginning and the other towards the end of measurements, noting the time for each. Ensure these are not outliers from the dataset, in order not to skew results.
- 2) Subtract the measured average CO₂ concentration within the room from measured CO₂ levels within the case. This allows us to only consider the concentration of CO₂ within the case that is exponentially decreasing.
- 3) Use the following equation to calculate the leakage rate (k):

$$k = \frac{\ln(C_{t_1}) - \ln(C_{t_2})}{t_2 - t_1}$$

- 4) A start time of time=0 allows the equation to be simplified to:

$$k = \frac{\ln(C_0) - \ln(C_t)}{t}$$

Variables:

k= leakage rate constant

t= time

C_t= C_t-r

C₀=C₀-r

C_t = measured concentration of CO₂ at time t within the enclosure

C₀= initial measured concentration of CO₂ within the enclosure

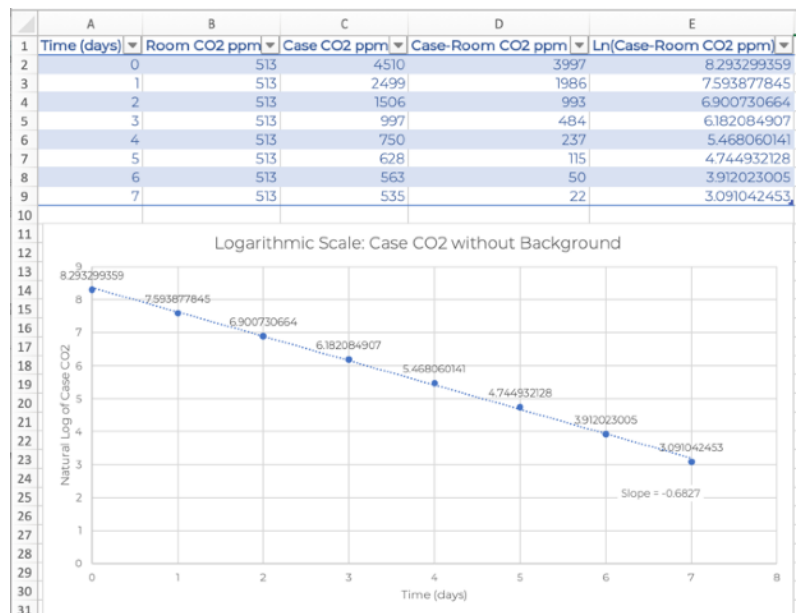
r= background concentration of CO₂ from the room

Example:

$$k = \frac{\ln(3997 \text{ ppm}) - \ln(1986 \text{ ppm})}{0.5 \text{ days}} = 1.4 \text{ days}^{-1}$$

Method 2: Excel

- 1) In an Excel column, insert measured values of CO₂ concentration in ppm from in the case over time (C_t).
- 2) Subtract the CO₂ concentration in ppm within the room from all of the values above (C_t).
- 3) Take the natural logarithm of the C_t values from step 2.
- 4) Insert a line graph with ln(C_t) from step 3 on the y-axis and time (t) on the x-axis.
- 5) Add a trendline, which will provide a linear, best fit line based on all the data points.^[3]
- 6) Add the slope of the trendline to the display. This value is the negative leakage rate (k) value.



^[3] Depending on the version of Excel, this can be found under "Add Chart Element."

Finally, once we have found the k value, we can solve for the half-time ($t_{1/2}$) or vice versa using the following equations:

$$k = \frac{\ln(0.5)}{t_{\frac{1}{2}}}$$
$$k \approx \frac{0.693}{t_{\frac{1}{2}}}$$
$$t_{\frac{1}{2}} \approx \frac{0.693}{k}$$

When referencing publications, be sure to note if they are referring to the half-time or leakage rate value (as noted in the introduction. Another common term for the leakage rate is Air Exchange Rate (AER), which is used in the AIC Wiki’s degree of case seal guidelines (see table below). Note that the units are days⁻¹. According to this guideline, degree III or above is required for microclimate control.

AIC Wiki (2020): Degree of Case Seal based on Air Exchange Rate:

Degree of Case Seal	Air Exchange Rate (based on CO ₂ or N ₂ O)
I Unsealed	> 1.0/day
II Moderately sealed	≤ 1.0/day
III Well-sealed	≤ 0.3/day
IV Very well-sealed	≤ 0.1/day
V Hermetically-sealed	No AE

Part C: Leakage Detection Equipment

Ultrasonic:

Ultrasonic leak detection equipment converts ultrasonic vibrations into audible sounds that vary with the strength of the ultrasonic signal. This equipment cannot provide quantitative proof of a leak but can help localize potential leakage points. Tones will travel through solids but will vary based on the density and depth of the material. Very thin portions of the case might produce a loud sound. Alternatively, if

air is escaping through a longer passage with sound-dampening materials, the ultrasonic signal might be weakened.^[4] As a general rule, try to keep the detector as close to the case as possible. Ultrasonic tone generators with an oscillating tone can help more easily detect changes.

Refrigerants:

While releasing refrigerants into the atmosphere is illegal, some small concentrations of non-chlorinated refrigerants is acceptable and found in products such as dust-removal spray cans. The refrigerant leak detector generates a beeping sound, which increases in frequency if the concentration of gas increases. Further research is required to verify whether it would negatively impact artworks housed in the cases.

Hydrogen gas:

Adding hydrogen gas to the case can also provide an effective method of locating the leak location. While hydrogen is highly flammable, a 5% concentration is safe to handle (the gas cylinder in Appendix 2, for example, contains 5% H₂ and 95% N₂). It is safe to use within cases housing objects, but is much more challenging to obtain. Hydrogen is a light gas, so in large gases, ensure it is well distributed and there is sufficient concentration in order to detect leaks towards the bottom of the enclosure.

Like ultrasonic lead detection, neither hydrogen gas nor refrigerant detection are quantitative. Unlike ultrasonic leak detection, which can be used while the enclosure remains open, these methods can only be carried out while the case is fully sealed and if the added gas is well-distributed throughout the space.

^[4]Weintraub, 2008

CONCLUSION:

Leakage rate testing of exhibition cases, storage enclosures, and packing crates is a critical measure for ensuring microclimates are serving their intended function. Using a tracer gas, taking measurements, calculating leakage rates, and locating leak locations might seem daunting, but creating a standardized procedure can significantly simplify the process.

While each museum will have differing equipment and case designs on hand, this protocol is intended to provide an initial guideline that can easily be applied to any specific set-up.

Cases and enclosures are a critical part of a museum's preventive conservation plan. More recent sustainability efforts within museums might lead to less strictly climate-controlled galleries, increasing the importance of controlled microclimates for sensitive objects. While suppliers promise airtightness based on prototypes, museums need to have a way to test their individual product, modifying leaking cases to improve air tightness, or in extreme cases, requesting a replacement from the manufacturer. In-house leakage testing is a relatively straight-forward and cost-effective procedure to ensure museum resources are being used effectively.

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APPENDIX A: GLOSSARY

Leakage Rate (k): the constant rate of CO₂ decay, calculated from the ln (natural logarithm) of the proportional change in concentration divided by the time over which that change in concentration occurred. The leakage rate is often referred to as air exchange rate (AER).

Microclimate: a contained space with an internal climate that remains distinct from its surroundings. Within a museum, microclimates commonly refer to sealed display cases or storage cabinets.

Leakage: the increase or decrease of a gas concentration from within an enclosure volume to its surrounding environment.

Diffusion: diffusion is a process that occurs when the random thermal motion of a gas causes it to move from an area of high to low concentration.

Permeability: diffusion through a solid material, which occurs at a much slower rate than diffusion through air.

Half-time decay ($t_{1/2}$): the time for a quantity of material to reduce to half of its original value (Note: half-time is used, as opposed to half-life decay, which more commonly refers to the decay of radioactive nuclei).

Air changed per day (acd⁻¹): the number of times in a day that the total air volume in a space is entirely replaced by surrounding air.

Graham's law of diffusion: this law states that the rate of diffusion of a gas is inversely proportional to the square root of its molar mass. If there is more than one air change per day, air exchange and not diffusion is occurring and Graham's law no longer applies.

Mean residence time: the reciprocal of the exchange rate constant.

APPENDIX B: BACKGROUND CALCULATIONS FOR LEAKAGE RATE EQUATIONS

To find the equation for leakage rate (k), we start with the traditional equation for exponential decay:

$$C_t = C_0 e^{-kt}$$

To find the value of the leakage rate constant, k, we take the natural logarithm of both sides, giving us the following equation:

$$\ln(C_t) = -kt + \ln(C_0)$$

This equation takes the slope intercept form ($y=mx+b$), which when plotted, forms a linear graph with a slope line of -k. This is the slope we can find using Excel in Method 2 of the procedure.

If we solve for the slope of this line using any two given points on the graph, we get the equation used in Method 1. Since slope is rise over run, we can take the vertical distance between two points on the line and divide by the horizontal distance.

$$-k = \frac{\ln(C_{t_2}) - \ln(C_{t_1})}{t_2 - t_1}$$

Converted to a positive k value:

$$k = \frac{\ln(C_0) - \ln(C_t)}{t}$$

APPENDIX C: EQUIPMENT

CO₂ Monitoring Equipment:

	Onset MX CO ₂ Logger	\$595	• 0 to 5,000 ppm, $\pm$ resolution • Accuracy: $\pm$5% of reading • 7.62 x 12.95 x 4.78 cm, 267.4 g • Data via Bluetooth or USB • https://www.onsetcomp.com/products/data-loggers/mx1102a/
	Extech CO ₂ Logger	\$340	• 0 to 9999ppm, 1ppm resolution • 117 x 102 x 102mm, 204g • Data via RS232 • http://www.extech.com/products/CO210
	Telaire CO ₂ Meter and Onset Logger	\$811	• 0 to 10,000 ppm • Accuracy: 1-2,000 ppm: $\pm$5% of reading, 2,000--10000, $\pm$10% of reading • https://www.transcat.com/onset-hobo-data-loggers-tel-7001-tel-7001-207325 • https://www.tequipment.net/HOBO-by-Onset/

CO₂ Dispensing Equipment:

	Clean DR Air Blaster and CO ₂ Dispenser	\$28	• 16 gram air cartridges • https://www.amazon.com/CleanDr-30103-AirBlaster-CO2-Actuator/dp/B00067EBKW/ref=cm_cr_ar_p_d_product_top?ie=UTF8
	Soda Siphon with CO ₂ chargers	\$39	• 8 gram air cartridges • https://impeccable-o.com/collections/soda-and-seltzer/products/ico-soda-siphon-1l
	CO ₂ canister and flow gauge	\$236	• 20 lb CO₂ canister (other sizes available online) • https://growershous.com/20-lb-co2-tank-regulator-with-valve-package-combo
	Sodastream Carbonation Unit	\$79	• 60L CO₂ cylinder • https://sodastream.com/products/terra

Ultrasonic Leak Detection Equipment

	Accutrak VPE-1000 Ultrasonic Leak Detector	\$1825	• Capable of detecting a 5 psi leak out of a 0.005" (5/1000 inch) hole, 20 to 30 feet away • Dedicated sensor for the touch probe, resulting in higher sensitivity detection • Comes with noise-cancelling headset https://superiorsignal.com/products/ultrasonic-instruments/accutrak-vpe-1000-ultrasonic-leak-detector
	Accutrak VPE Ultrasonic Leak Detector	\$630	• Capable of detecting a 5 psi leak out of a 0.005" (5/1000 inch) hole, 20 to 30 feet away https://superiorsignal.com/products/ultrasonic-instruments/accutrak-vpe-ultrasonic-leak-detector
	Accutrak VPESG2 Burst Tone Sound Generator	\$260	https://www.tequipment.net/AccuTrakVPESG2.html

Refrigerant Leak Detection Equipment

	Inficon D-Tek Select Leak Detector	\$538	https://www.trutechtools.com/Inficon-D-TEK-CO2-Leak-Detector
	Dust-Off Compressed Gas Duster (1,1- Difluoroethane, listed as R-152a)	\$16	https://www.homedepot.com/p/DUST-OFF-10-oz-Disposable-Compressed-Gas-Duster-DPSXL/302832922

Hydrogen Leak Detection Equipment

	TechnoTools D640 Hydrogen Leak Detector	\$390	• Detects leaks less than 5 ppm. https://www.autometer.com/refrigerant-leak-detector-autogage-6993.html
	Gasco H ₂ 5%, N ₂ 95% Gas Cylinder and Regulator (Non- Volatile Mixture)	\$105	https://www.calgasdirect.com/gasco-hydrogen-calibration-gas-5-h2-balance-nitrogen-in-a-34-liter-steel-disposable-cylinder-connection-type-cga-600/