

CHILLED MIRRORS FOR TRANSFER STANDARD

APPLICATION NOTE

Climate Chambers

Simulation of climatic conditions including temperature, humidity, pressure, vibration and light intensity.

Humidity / Temperature

Validation / Calibration

Fast And Precise

- Automotive component test chambers
- Pharmaceutical stability chambers
- Calibration test chambers
- Military electronics test

Climatic Chambers - used in many industries to simulate climatic conditions

Climatic chambers are widely used to simulate climatic conditions including temperature, humidity, pressure, vibration and light intensity. Product samples are placed into the chamber and subjected to conditions with fixed or variable set points, with conditions and profiles defined by test specifications. Examples include 40°C, 75 % RH for accelerated drug stability tests, and 85°C, 85 % RH for electronic component reliability testing. Validation of test conditions is a requirement for compliance with standards and regulations. This application note is focused on humidity and temperature measurement and control in climatic chambers.

Importance of Chamber Validation

Validation of chamber performance in generating test conditions must be carried out periodically, usually to meet compliance requirements. It can be necessary to meet the requirements of quality systems such as ISO9001 and ISO17025. A chamber may be tested with or without load, and during the test procedure the conditions are measured using calibrated instruments for both humidity and temperature. Reported results would typically include measured conditions, stability, average, maximum, minimum, uniformity and mean kinetic temperature. The instruments used to measure the chamber performance will have an influence on the uncertainty that is assigned to the chamber calibration, so calibration uncertainty, actual performance and long term stability are important.

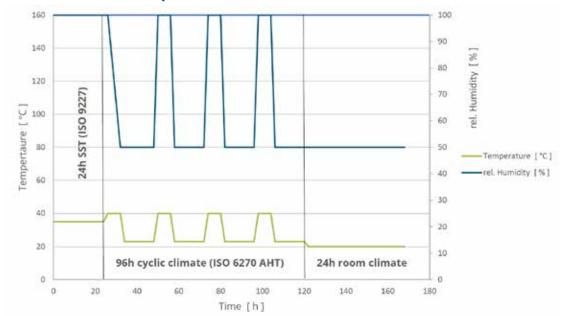
Industry Applications

- Chamber production
- Chamer Validation
- Pharmaceutical stability test
- Calibration systems
- Electronic reliability test
- Automotive test
- Plant growth chambers
- Aerospace/altitude test



Example test specifications:

Test type	Application	Typical Conditions
ICH Protocols (e.g. Q1A)	Drug stability	20, 30, 40 °C / 60, 75 % RH
MIL	Military equipment	85 °C / 85 % RH
Salt Fog DIN50021	Corrosion testing	Including saturation with respect to humidity



Climatic Chamber Example Test Profile

Technology Used for Chamber Measurement and Control:

Climatic chambers are controlled using either psychrometers (wet & dry bulb thermometers) or relative humidity sensors. These measurement technologies have some limitations that must be considered:

- Psychrometer measurement performance is user and maintenance dependent
- Psychrometers evaporate water vapour into the working volume
- Psychrometers have less accuracy at low temperatures and relative humidity
- RH probes having variable performance characteristics
- RH sensors drift over time, when exposed to high humidity and temperature conditions
- RH probe measurement performance is temperature dependent

In both cases, skilled users and effective procedures, including regular calibration, can produce adequate results, but periodic comparison to a reference standard is an ongoing need for validation, and to maintain traceability and performance. Any calibration or reference standard used should be of a higher performance capability than the unit under test.

Test Uncertainty Ratio:

The comparison of the 'accuracy' of the Unit Under Test (UUT) and the accuracy of the reference standard is the Test Accuracy Ratio (TAR). A typical requirement within standards is a TAR of 4:1. This means that the reference standard should have a specification 4 times better than the UUT. With humidity measurement this is quite challenging, and clearly, calibrating a measurement device with a device of similar specifications is not likely to support an adequate TAR. However, TAR does not consider other potential sources of error in the calibration process, so Test. Uncertainty Ratio (TUR) is more commonly used. Users calculate measurement uncertainty by combining possible (error) contributions statistically and usually report the measurement result at a 95 % confidence level (k=2). A comparison of measurement uncertainty when using a dew point mirror and an RH probe in a test chamber (at 40 °C / 75 % RH) is shown below:

Measurement uncertainty budget using a dew point chilled mirror hygrometer

The measurement uncertainty budget is divided into 3 steps:

- 1.Calculation of the measurement uncertainty of the air temperature.
- 2. Calculation of the measurement uncertainty of the dew point temperature.
- 3. Calculation of the measurement uncertainty of the relative humidity from 1. and 2.



1. Overall uncertainty of temperature in chamber

Uncertainty source	Estimated value	Units	Sensitivity coefficient	Distribution	Divisor	Uncertainty contribution
Measured temperature	40.02	°C				
Uncertainty of the average value (measurement)	0.02	°C	1.00	normal	1.00	0.02
Temperature indicator calibration (PRT included)	0.03	°C	1.00	normal	2.00	0.02
Interpolation between calibration points / nonlinearity	0.01	°C	1.00	rectangular	1.73	0.01
PRT hysteresis (repeatability)	0.01	°C	1.00	rectangular	1.73	0.02
PRT drift	0.03	°C	1.00	rectangular	1.73	0.02
PRT self-heating	0.01	°C	1.00	rectangular	1.73	0.01
PRT heat dissipation	0.00	°C	1.00	rectangular	1.73	0.00
Resolution of inidcation	0.01	°C	1.00	rectangular	3.46	0.00
Temperature gradients in chamber	0.10	°C	1.00	rectangular	1.73	0.06
Temperature fluctuations in chamber	0.06	°C	1.00	rectangular	1.73	0.03
Radiation influence in chamber	0.08	°C	1.00	rectangular	1.73	0.05
		Standard uncertainty				0.09
		Expanded uncertainty (95 % confidence)				0.18 °C

2. Overall uncertainty of dew point temperature in chamber

Uncertainty source	Estimated value	Units	Sensitivity coefficient	Distribution	Divisor	Uncertainty contribution
Measured dew point temperature	34.71	°C dp				
Uncertainty of the average value (measurement)	0.05	°C dp	1.00	normal	1.00	0.05
Dew point mirror calibration	0.07	°C dp	1.00	normal	2.00	0.04
Dew point mirror repeatability	0.05	°C dp	1.00	rectangular	1.73	0.03
Dew point mirror drift	0.05		1.00	rectangular	1.73	0.03
Interpolation between calibration points / nonlinearity	0.05	°C dp	1.00	rectangular	1.73	0.03
Temperature dependence of the measuring head	0.01	°C dp	1.00	rectangular	1.73	0.01
Unknown condensation layer (frost / dew)	0.00	°C dp	1.00	rectangular	1.73	0.00
Resolution of inidcation	0.01	°C dp	1.00	rectangular	3.46	0.00
Pressure difference between chamber and mirror	0.10	hPa	7.00	rectangular	1.73	0.00
Dew point tempera ture fluctuations in chamber	0.13	°C dp	1.00	rectangular	1.73	0.08
Dew point inhomogeneity in the chamber	0.05	°C dp	1.00	rectangular	1.73	0.03
		Standar	0.11			
		Expanded uncertainty (95 % confidence)				0.23 °C

Calculation of relative humidity

Uncertainty source	Estimated value	Units	Sensitivity coefficient	Distribution	Divisor	Uncertainty contribution
Overall uncertainty of temperature in chamber (1.)	0.09	°C	4.00	normal	1.00	0.35
Overall uncertainty of dew point temeprature in chamber (2.)	0.11	°C dp	4.16	normal	1.00	0.47
Uncertainty of absolute pressure measurement	1.00	hPa	0.00	rectangular	1.73	0.00
Uncertainty of used formulas	0.02	% RH	1.00	rectangular	1.73	0.01
		Standard uncertainty				0.59
		Expanded uncertainty (95 % confidence)				1.17 %

Measurement uncertainty using a relative humidity probe

Uncertainty source	Estimated value	Units	Sensitivity coefficient	Distribution	Divisor	Uncertainty contribution
Measured temperature	40.02	°C				
Uncertainty of the average value (measurement)	0.02	°C	1.00	normal	1.00	0.02
Temperature indicator calibration (PRT included)	0.10	°C	1.00	normal	2.00	0.05
Uncertainty of used formulas	0.02	% RH	1.00	rectangular	1.73	0.01
Interpolation between calibration points / nonlinearity	0.03	°C	1.00	rectangular	1.73	0.02
PRT hysteresis (repeatability)	0.01	°C	1.00	rectangular	1.73	0.01
PRT drift	0.07	°C	1.00	rectangular	1.73	0.04
PRT self-heating	0.03	°C	1.00	rectangular	1.73	0.02
PRT heat dissipation	0.00	°C	1.00	rectangular	1.73	0.00
Resolution of indication	0.01	°C	1.00	rectangular	3.46	0.00
Temperature gradients in chamber	0.10	°C	1.00	rectangular	1.73	0.06
Temperature fluctuations in chamber	0.06	°C	1.00	rectangular	1.73	0.03
Radiation influence in chamber	0.08	°C	1.00	rectangular	1.73	0.05
		Standard uncertainty				0.11
		Expanded uncertainty (95 % confidence)				0.22 °C

Overall uncertainty of temperature in chamber

Lower Measurement Uncertainty:

Using a dew point chilled mirror can significantly lower the measurement uncertainty when compared with other humidity measurement techniques. This is because of their fundamental precision, lower long-term drift, more precise temperature measurement, lower temperature coefficients, and lower calibration uncertainty.

Overall uncertainty of relative humidity in chamber

Uncertainty source	Estimated value	Units	Sensitivity coefficient	Distribution	Divisor	Uncertainty contribution
Uncertainty of the average value (measurement)	0.10	% RH	1.00	normal	1.00	0.10
Humidity probe calibration uncertainty	1.2 0	% RH	1.00	rectangular	2.00	0.60
Humidity probe repeatability / hyseresis	0.50	% RH	1.00	rectangular	1.73	0.29
Humidity probe drift	1.50	% RH	1.00	rectangular	1.73	0.87
Interpolation between calibration points / nonlinearity	0.50	% RH	1.00	rectangular	1.73	0.29
Humidity probe temperature dependence	1.50	% RH	1.00	rectangular	1.73	0.87
Resolution of the relative humidity probe	0.10	°C	1.00	rectangular	3.46	0.03
Humidity probe heat dissipation	0.00	°C	4.00	rectangular	1.73	0.00
Humidity probe self heating	0.05	°C	4.00	rectangular	1.73	0.12
Radiation influence in chambermirror	0.08	°C	4.00	rectangular	1.73	0.18
Humidity inhomogeneity in the chamber	0.45	% RH	1.00	rectangular	1.73	0.26
Humidity fluctuations in chamber	0.60	% RH	1.00	rectangular	1.73	0.35
		Standard uncertainty				1.51
		Expanded uncertainty (95 % confidence)				3.01 °C



Dew Point Chilled Mirrors for Chamber Validation:

Chilled mirror dew point[™] hygrometers are used as reference standards in national and accredited laboratories, and increasingly for industrial applications such as climatic chamber calibration and validation. There are different types available to measure dew/frost points over the entire water vapor range from -95 C frost point to +95 C dew point.

The MODEL 473[™] is the most used dew point mirror for chamber applications. The cable mounted measuring heads work directly in the test space. The MODEL 473-SH2[™] and MODEL463-SHX[™] measuring heads feature flow modules that control sample flow over the dew point mirror, so that flow and pressure are stable during measurement. The flow module also ensures the sample gas is representative of the chamber working volume, so the measuring head can be positioned anywhere within the chamber. This gives the advantage that the waste heat of the measuring head does not affect the calibration. All 473 models include in a probe for measuring temperature, and this is used to calculate relative humidity. The dew point measuring heads have an additional internal temperature sensor so the user can make sure that the measuring head temperature is always above the dew point so that condensation does not occur.

For users who demand the very best measurement capability, dew point mirrors installed outside the chamber are used The MODEL 573[™] and MODEL 373[™] both feature temperaturecontrolled measuring heads and sample tubes so that measuring dew points higher than the ambient temperature are possible without condensation. This sampling configuration means that chambers can be calibrated without disturbing long-term stability tests. Another advantage is that the mirror can be cleaned without disturbing the chamber conditions.



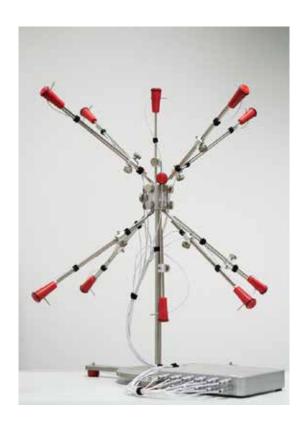
Temperature and Relative Humidity Uniformity

Dew point measurement data from chilled mirrors can be combined with temperature data from the T12 high precision multichannel thermometer. Data integration within Gecko R2 software means that spatial temperature and RH distribution can be measured, displayed and recorded for validation or calibration processes.

Measurement of Temperature Uniformity

Temperature uniformity is the most important measurement when calibrating or validating climatic chambers. It is also typically the largest uncertainty contribution.

The T12 thermometer is a high precision multi-channel thermometer that is suitable for measurement of temperature uniformity in climatic chambers. It is combined with specially performance Pt100 Ω platinum resistance thermometers (PRT) that are compact, precise and stable. Typical uncertainty of the T12/PRT combination is $\leq \pm 0.04$ °C (40mK), and the instruments can be delivered with ISO17025 accredited calibration certificate for the complete measurement system.



Also supplies a system for positioning the PRTs into climatic chambers, called the Spider (although it has more than 8 legs!). The Spider has been carefully designed to have a low thermal mass to minimize any influence on the chamber thermal performance, and includes Silcone isolating mounting points for each PRT. For engineers performing thermal mapping validation/calibration processes on multiple chambers, the Spider can save significant set up time. Consistent position of PRTs also supports better reproducibility of the calibration procedure.



Key Features of Dew Point Chilled Mirrors for Climatic Chamber Applications:

- Precise and stable measurement of humidity and temperature
- Dependable sampling at up to 99°C dew point
- Measuring heads with integrated flow modules
- Simultaneous measurement of temperature
- Determination of relative humidity
- Fully self-contained and easily transportable
- Integrated calibration verification function to allow on-site functionality tests
- Integration with multichannel temperature system for temperature and RH uniformity measurement
- Cable mounted dew point measuring heads that work effectively in the chamber working volume at up to 125 °C

For further information visit: www.process-insights.com



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Process Insights GmbH

Neukoellnische Allee 134 12057 Berlin Germany +49 30 2789580 www.process-insights.com info@emea.process-insights.com

Process Insights - EMEA

Process Insights GmbH Lyoner Strasse 15 60528 Frankfurt Germany +49 69 20436910 info@emea.process-insights.com

Process Insights - The Americas

4140 World Houston Parkway Suite 180 Houston, TX 77032 USA +1 713 947 9591 info@process-insights.com

Process Insights - APAC

Wujiang Economic and Technology Development Zone No. 258 Yi He Road, 215200 Suzhou Jiangsu Province China +86 400 0860196 info@emea.process-insights.com