



METER

MÉTODOS E PRINCÍPIOS DE MEDIDA DE ATIVIDADE DE ÁGUA

METER Group Latam

Tânia M. M. Shibata

MEDIDORES DE ATIVIDADE DE ÁGUA

- Higrômetros com polímeros ou capilar
- Ponto de congelamento
- Equilíbrio isopiéstico
- Higrômetros eletrônicos
- Espectrofotômetro de Infravermelho
- Sensor por ponto de orvalho
- Laser



PRINCÍPIOS DE MEDIÇÃO



Higrômetro capilar



Ponto de congelamento
(Freezing point depression)

Equilíbrio isopiéstico

(processo de aquecimento de um gás a pressão constante, com variação de volume)



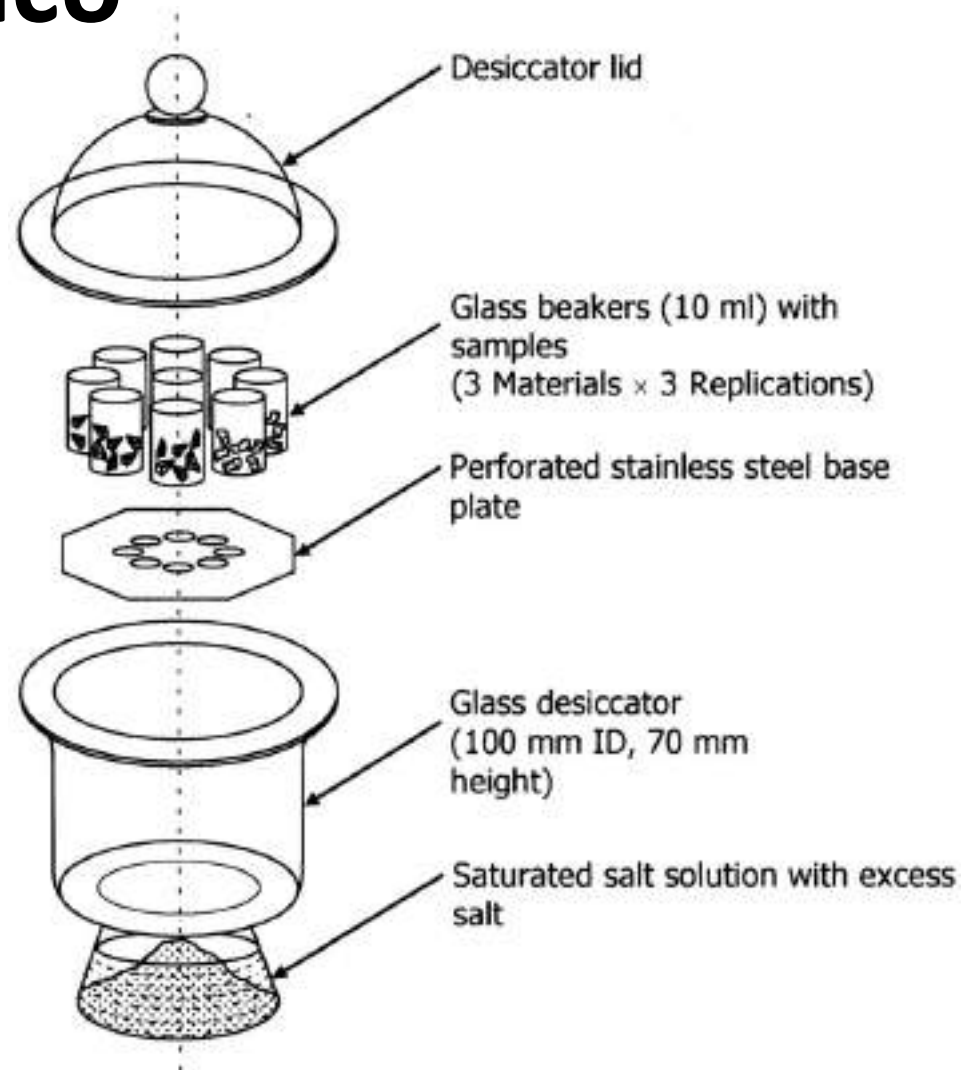
Table 9—The a_w of selected saturated salt solutions between 15°C and 37°C

Temp (°C)	NaCl	(NH ₄) ₂ SO ₄	KCl	BaCl ₂	K ₂ SO ₄
15	0.753	0.808	0.859	0.910	0.979
17	0.753	0.806	0.856	0.909	0.978
19	0.752	0.805	0.852	0.907	0.977
21	0.752	0.804	0.849	0.906	0.977
23	0.751	0.803	0.846	0.905	0.976
25	0.751	0.803	0.842	0.903	0.975
27	0.750	0.802	0.840	0.902	0.975
29	0.750	0.801	0.836	0.900	0.974
31	0.750	0.800	0.833	0.899	0.973
33	0.749	0.799	0.830	0.898	0.973
35	0.749	0.798	0.827	0.895	0.972
37	0.748	0.797	0.823	0.894	0.971

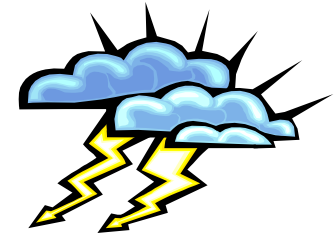
Theoretical Prediction of the Water Activity of Standard Saturated Salt Solutions at Various Temperatures

DORA KITIC, DENISE c. PEREIRA JARDIM, GUILLERMO J. FAVETIO, SILVIA L. RESNIK, and JORGE CHIRIFE

Equilíbrio isopiéstico

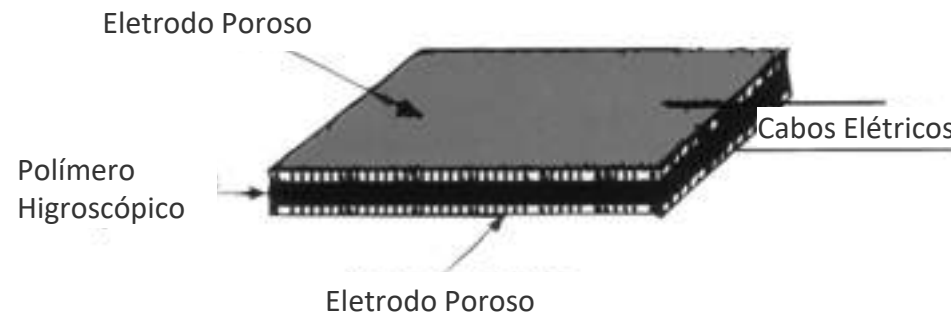


SENSORES DIELÉTRICOS



A umidade altera as propriedades elétricas de um material higroscópico em equilíbrio com o ar que está sobre a amostra.

Constante dielétrica ou Capacitância





AquaLab 4TEV (sensores de ponto de orvalho e capacitivo)

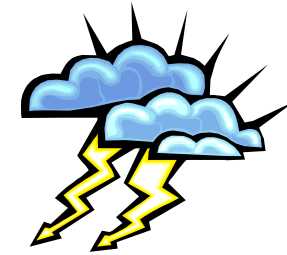


AquaLab PRE (sensor ponto de orvalho ou capacitivo)



Pawkit (sensor capacitivo)

SENSORES DIELÉTRICOS



Vantagens

Exatidão = $\pm 0,01a_w$

Relativamente insensível aos voláteis

Leitura de toda a faixa de a_w

Desvantagem

Necessita calibração (método secundário)

Sensor requer compensação da temperatura

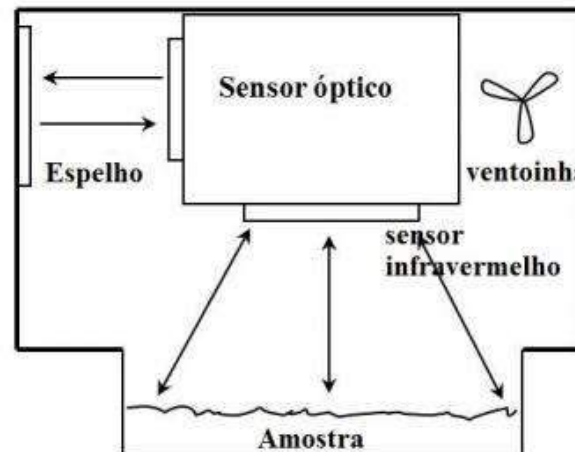
Sensor pode sofrer histerese



PONTO DE ORVALHO



O espelho se resfria até que se forme o orvalho. Célula fotoelétrica detecta o ponto exato da primeira condensação no espelho. Um termopar grava a temperatura na qual ocorreu a condensação. AquaLab então emite um sinal sonoro e apresenta os valores de atividade de água final e temperatura.



PONTO DE ORVALHO



AquaLab 3



PONTO DE ORVALHO



Vantagens

Método primário de medida de pressão de vapor (não necessita calibração)

Alta exatidão $\pm 0,003a_w$

Medição rápida <5min.

Faixa de medida a_w (0,03 – 1,00)

Alta confiança

Desvantagens

Pode precisar de limpeza do espelho

Leituras podem ser afetadas por alta concentração de álcool, propilenoglicol, ácido acético.



USP 922



TYPES OF WATER ACTIVITY INSTRUMENTS

Dew Point Temperature—Chilled Mirror

At the dew point temperature, the relative humidity will be 100%, the evaporation and condensation rates will be equal, and there will not be any net condensation. When the temperature of a surface drops below the dew point temperature, water will condense on it. The dew point temperature can be determined by cooling a mirror in the sample chamber until condensation begins to occur and recording the mirror temperature at that point. The condensation on the chilled mirror is detected by an optical sensor. The difference between the dew point temperature and the temperature of the sample chamber is used to determine the relative humidity. The mirror must be kept clean and protected from contamination by dust or residue that may condense on the chilled mirror. Therefore, measurements on microleak patches may increase the likelihood of repeat calibration issues. The measurement of dew point temperature is a primary method of determining the vapor pressure of the headspace.

Electronic Hygrometers—Resistive (Electrolytic) Hygrometer

In this instrument, the relative humidity in the sample chamber is measured by a hygroscopic resistor. The resistor is a liquid electrolyte that absorbs or loses water to the air in the sample chamber, and the resulting resistance is proportional to the relative humidity. These resistors provide good precision and typically function over the a_w range of 0.04–1.00. Soluble substances, which are soluble in the liquid electrolyte, may affect the resistance and interfere with the relative humidity measurement. Chemical filters may be used to absorb volatiles and prevent interference. The response of these instruments is not necessarily linear, so sensors should be calibrated using multiple reference solutions over the entire operating range. Alternatively, a two-point linear calibration over a narrower range that brackets the sample may be used.

Electronic Hygrometers—Capacitive Hygrometer

In this instrument, the relative humidity in the sample chamber is measured by a hygroscopic capacitor. The capacitor consists of a hygroscopic polymer held between conductive plates. The polymer absorbs or loses water to the air in the sample chamber, and the resulting capacitance is proportional to the relative humidity. These sensors may become saturated at high a_w and may exhibit hysteresis after measurement of high a_w samples. Capacitive hygrometers are typically less affected by water vapors than resistive or dew point sensors; however, care should still be used when performing measurements in the presence of other volatile compounds. The response of these instruments is not necessarily linear, so sensors should be calibrated using multiple reference solutions over the entire operating range. Alternatively, a two-point linear calibration over a narrower range that brackets the sample may be used.

Optical Hygrometer—Tunable Diode Laser

Water molecules in the gas phase absorb a product about 1/3 and 1/34 radiation. Strong and broad absorption bands exist that can be used to measure the vapor pressure of air, but many of these are near absorption bands of other volatiles that may interfere. The most specialized optical hygrometers are not used for water activity measurements. The absorption bands are composed of individual transitions and unique not only to a specific molecule but to a particular isotope of water. A diode laser can produce light at a specific wavelength, with a narrow bandwidth of ± 1 nm. The wavelength of a tunable diode laser (TDL) can be tuned to a specific, isolated absorption line in the water vapor spectrum by controlling the laser temperature and can be scanned across that line by modulating the laser current (and, therefore, the laser's temperature). This modulation provides both the absorption and unperturbed laser intensity in the headspace, from which the vapor pressure of the headspace can be calculated. Because the TDL is capable of measuring vapor pressure in the presence of even high concentrations of other volatiles, it is the only method that can correctly measure water activity in these situations. Electronic and dew point hygrometers are sometimes suitable in the presence of low concentrations of volatiles, but readings are always adversely affected by the volatiles. The TDL also can be used to measure water activity noninvasively in sealed and sealed containers.

Near-Infrared Spectroscopy

Near-infrared spectroscopy (NIR) data has traditionally been used to make the a_w measurements with the water content. The use of NIR to measure a_w is based on the generation of a predictive model for a particular official article (product or raw material) to correlate NIR absorbance spectra to the a_w measured with one of the previously mentioned direct methods. This predictive model may be used to rapidly predict the a_w of additional samples. When additional predictive models are available, NIR can be used for simultaneous determination of other compounds such as the content of active ingredients, additives, or impurities. The NIR method is best suited for rapid analysis of raw material, in-process materials, and end products.

Other Methods

Other methods for measurement of a_w may be useful but are not currently employed for routine measurements of pharmaceutical products, dietary supplements, and raw materials. These methods include gravimetry, lowing point depression, thermogravimetric analysis, and aqueous methods. The hygrometers may be used for approximate a_w measurements but not for routine work due to very high precision times and low accuracy. The freezing-point and thermocouple

ALGUNS PRINCÍPIOS DE MEDIÇÃO DE ATIVIDADE DE ÁGUA – USP 922

- Ponto de orvalho – espelho resfriado
- Higrômetro eletrônico
 - Capacitivo
 - Resistivo
- Higrômetro óptico – TDL
- Espectrofotômetro - IV Próximo
- Higrômetro capilar
- Depressão do Ponto de congelamento
- Isopiéstico



TDL TUNABLE DIODO LASER



SKALA

SISTEMA DE GERENCIAMENTO



SKALA

SISTEMA DE GERENCIAMENTO

Adicionar Ingredientes do Produto

▼ DETALHES DO PRODUTO

Descrição do produto	<input type="text" value="Raças para pelo"/>
Peso rotulado (g)	<input type="text" value="15000"/>
Instruções de armazenamento	<input type="text" value="Ao abrigo da luz"/>
Atividade de Água ideal	<input type="text" value="0,63"/>
Teor de Umidade Ideal	<input type="text" value="13"/>
Avg Pre-Cook Moisture Content	<input type="text" value="40"/>
Custo dos ingredientes por unidade (USD)	<input type="text" value="0,5"/>
Preço médio de venda por unidade (USD)	<input type="text" value="80"/>
PDF do produto	<input type="button" value="Carregar PDF"/>



SKALA

SISTEMA DE GERENCIAMENTO

Modelo de conteúdo úmido

Personalizada ⌵ ⊞

Atividade de Água – Water Activity

Adicionar automaticamente letras de unidade ao registrar a leitura da atividade de água (reversa para MZA)

DLP

b0 b1

a0 a1

GAB

w0 4,76067 x 8,817116

e 12557,877197

BET

w0 e

MZA

c0 c1

c2 c3

Deslocamento linear 1,06110035400207

Atividade de água medida eq. 0,25

Teor de umidade medido eq. 12,34

Calcular ajuste



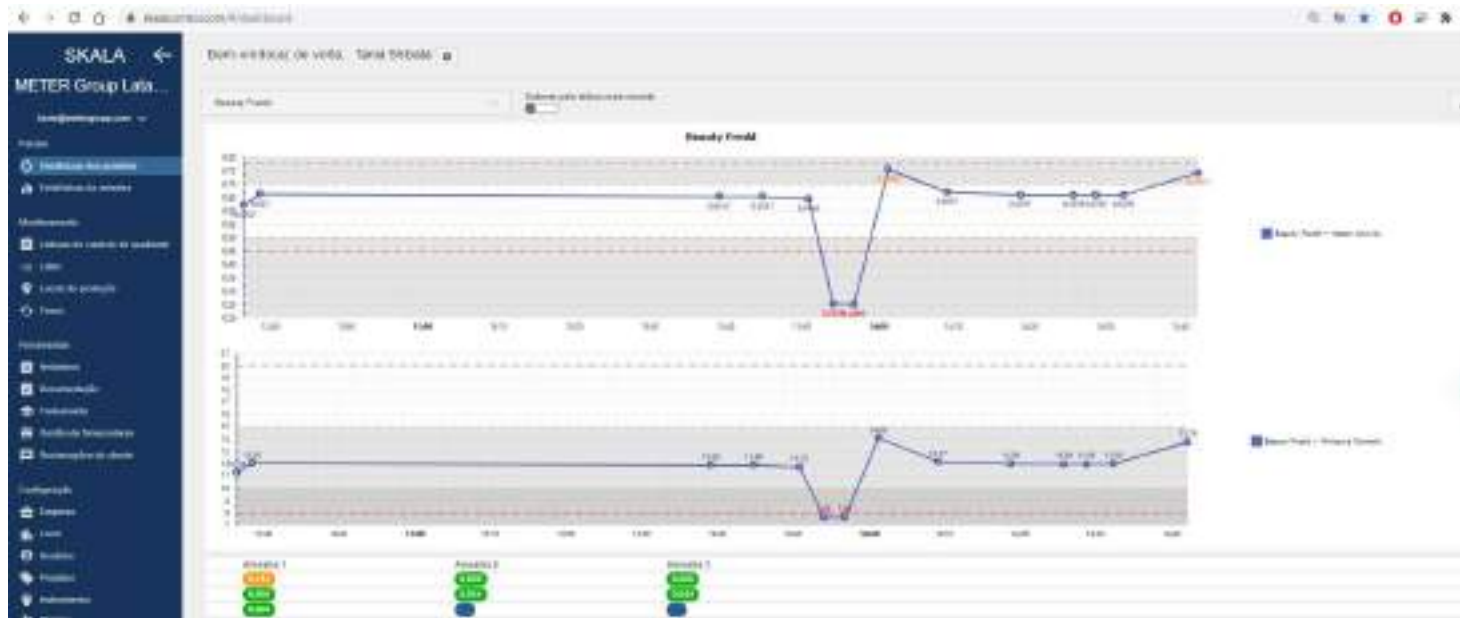
SKALA – SISTEMA DE GERENCIAMENTO

The screenshot displays the SKALA system interface for 'METER Group Ltda...'. The main heading is 'Leituras de controle de qualidade'. The interface includes a search bar with filters for 'Filtro: Todos os registros', 'Produto: Biscoito Pão de Mel', and 'Data: Todos os tempos'. A table lists 13 quality control readings with columns for 'ID', 'Código', 'Nome', 'Valor', 'Produto', 'Unidade de Medida', 'Valor', and 'Observação'. The 'Valor' column contains numerical values with green or orange status indicators. A sidebar on the left contains navigation options like 'Início', 'Relatório de qualidade', and 'Configurações'.

ID	Código	Nome	Valor	Produto	Unidade de Medida	Valor	Observação
2	1186-001 1820	Tampa Biscoito	0222212	Biscoito Pão de Mel	Conteúdo da Unidade	17,20	MARBOM, S/A
1	1186-001 1821	Tampa Biscoito	0200002	Biscoito Pão de Mel	Unidade de Esp. 1	6,30	AGULABE (1)
3	1186-001 1822	Tampa Biscoito	0219392	Biscoito Pão de Mel	Conteúdo da Unidade	17,20	AGULABE (1)
5	1186-001 1823	Tampa Biscoito	0241002	Biscoito Pão de Mel	Unidade de Esp. 1	6,30	AGULABE (1)
3	1186-001 1824	Tampa Biscoito	0241992	Biscoito Pão de Mel	Conteúdo da Unidade	16,20	AGULABE (1)
3	1186-001 1825	Tampa Biscoito	0233272	Biscoito Pão de Mel	Conteúdo da Unidade	17,20	MARBOM, S/A
3	1186-001 1826	Tampa Biscoito	0210002	Biscoito Pão de Mel	Unidade de Esp. 1	6,30	AGULABE (1)
3	1186-001 1827	Tampa Biscoito	0219392	Biscoito Pão de Mel	Conteúdo da Unidade	16,20	AGULABE (1)
3	1186-001 1828	Tampa Biscoito	0210002	Biscoito Pão de Mel	Unidade de Esp. 1	6,30	MARBOM, S/A
3	1186-001 1829	Tampa Biscoito	0210002	Biscoito Pão de Mel	Unidade de Esp. 1	6,30	AGULABE (1)



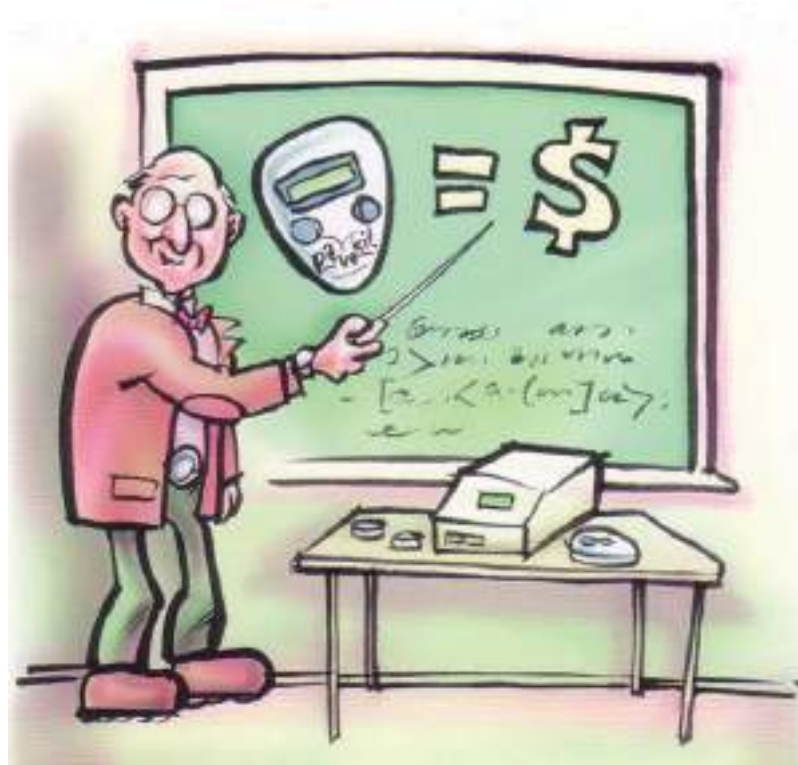
SKALA – SISTEMA DE GERENCIAMENTO



SKALA – SISTEMA DE GERENCIAMENTO



PERGUNTAS?



Muito obrigada pela sua participação

CONTATE-NOS

METER Group LatAm

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