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Determination of vapor pressure of chemical products - Thermogravimetry method

化工产品饱和蒸汽压的测定　热重法

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Determination of vapor pressure of chemical products - Thermogravimetry method

1 Scope

This Standard specifies the method for the determination of vapor pressure of chemical products using the thermogravimetry method.

This Standard applies to all solids and liquids of saturated vapor pressure within the range of 1^{-10} Pa ~ 1 Pa.

Impurities in chemical products have a certain influence on vapor pressure results during measurement.

2 Principle for test method

In this Standard, thermogravimetric analysis meter is used to determine the speed of loss in mass within a certain time under relatively high temperature and indoor pressure. Under a slow inert gas flow atmosphere and temperature T (in K), the loss in mass of sample is monitored within a certain time to obtain the speed of loss in mass v_T. Based on the linear relationships between vapor pressure logarithm and the speed of loss in mass, saturated vapor pressure p_T at temperature T can be calculated from v_T. If necessary, vapor pressure under 20°C and 25°C can be extrapolated from the regression between $1np_T$ and $1/T$.

3 Apparatus

3.1 Thermogravimetry analysis meter, which is equipped with the following devices:

3.1.1 Thermo-balance, including the following elements:

a) furnace chamber: capable of staying at a constant temperature and heating sample to 450°C at a constant speed of 5°C/min ~ 25°C/min;

b) temperature sensor: used to display the temperature of sample/furnace chamber, with a sensitivity of ± 0.1°C;

c) continuous record balance: with a sensitivity of ± 10 μg;

d) controlled inert gas atmosphere device: with a purity of inert carrier gas above 99.99%

and rate of flow (0 mL/min ± 5 mL/min) ~ (100 mL/min ± 5 mL/min).

3.1.2 Temperature controller: capable of executing specific temperature programs within temperature intervals selected, with a temperature change ratio of 0°C/min ~ 25°C/min and temperature fluctuation within ± 0.5°C.

3.1.3 Recording unit: capable of recording and displaying the mass signals of sample and the changes of noise signals to temperature and noise (the TGA Curve).

3.1.4 Crucibles: their area can be measured; they will not react with sample; they can remain stable under 450°C.

3.2 Graduated micropipettes: with a capacity of 20 μL ~ 40 μL and tolerance ± 1 μL.

4 Reagents and materials

4.1 Benzoic acid: with a purity not lower than 99.9%.

4.2 Nitrogen: with a purity of 99.99% (or other inert carrier gases). It is dried using molecular sieve or silica gel.

5 Analytical procedures

5.1 Sample pre-treatment

Apply liquid sample evenly on the surface of crucible (3.1.4). If sample is a solid, semi-solid or viscous liquid, it can be dissolved using an appropriate solvent and then infiltrated on the surface of crucible (3.1.4) using a solution.

5.2 Sample determination

5.2.1 Place crucible (3.1.4) applied with sample in the test chamber of thermogravimetric analysis meter; dry to a constant weight in a nitrogen (4.2) atmosphere. Adjust nitrogen (4.2) flow which shall not be too high, to ensure that gasified molecules can be taken away in time.

5.2.2 Set the temperature control procedure which consists of a series of isothermal sections. Maintain a temperature difference of 10 K between temperature sections. If another temperature is used, it shall be indicated in report. Maintain for a certain time before starting each temperature section; set 10 min or longer time of constant temperature. The increase of constant-temperature time is normally to be benefit of stable volatilization, but it will extend test time.

5.2.3 The lower limit of constant-temperature range is subjected to that there needs to

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