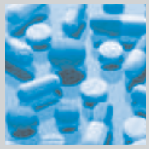
DENVER INSTRUMENT





Moisture Determination in Melamine

General

Nitrogen compounds, acid amides and weakly basic amines (heterocyclenes) give no problem. They can be titrated according to the standard procedures. Strongly basic amines alter the pH value and must be neutralised prior to the titration. Some amines lead to side reactions, the nature of which is yet unknown. In some cases, these side reactions are suppressed in methanol-free reagents, sometimes additional neutralisation with benzoic acid or salicylic acid helps. Small sample sizes improve the end-point stability. The substance dissolves with difficulty. A weakly acid solvent is the most suitable.

Reagent

Titrant: HYDRANAL-Titrant 2
Working medium: 40 ml
HYDRANAL-Solvent + 10 g
Salicylic acid
(Composite reagent is not suitable)

Primary Settings

Method ID:	Melamine
Use oven:	No
Auto start:	Yes
Blank: No	
Uncert. calc.:	Yes
Reproducibility:	0.1%

Parameters

Stirring speed:	600 rpm
Max. bur. speed:	150%/min
Min. titr. time:	02:00 (min:s)
Max. titr. time:	00:05 (h:min)
Max. volume:	10 ml

Sample

Sample ID:	Yes
Sample unit:	g
Advised amount:	1.000 g
Uncertainty:	0.001 g
Sample factor:	1
Result unit:	%
Number of digits:	6
Quality control:	No

Procedure

The working medium is placed in a thermostated titration cell, heated to 50°C and titrated to dryness with the titrant. 1 g of sample is administered with a powder funnel. Weigh by difference. When it has dissolved, it is titrated at 50°C to a stable end-point.

Comments

1 measurement can be made with the same solvent. If more measurements are performed, the mixture will solidify when it cools down and clog the tubing to the waste bottle. The solvent must be renewed after each analysis. In order to obtain accurate measurements, we recommend adjusting the sample size and choosing the titre so that the delivered titrant amount is at least 1 ml. However, the solubility of some samples limits the amount that can be injected into the KF cell. If the sample has a low moisture content, the small sample size will mean that the amount of titrant needed may consequently be lower than 1 ml. The uncertainty on the delivered volume is quite constant so the only consequence is to increase the relative uncertainty on the result. This is taken into account for the results in the uncertainty calculations.

Results

Mean: 0.069 ±0.010% H_2O
(K=2, 3 replicates)
K: coverage factor