

## BEST PRACTICE FOR DETERMINING LOW FREE FORMALDEHYDE LEVELS IN PHENOL FORMALDEHYDE RESIN (RESOLES) USING EN 11402:2004-MODIFIED

Based on the results of a Round Robin (April 2016) and analytical tests performed by members of the EPRA – RSC, the following recommendations are given to anyone performing a determination of low level free formaldehyde (< 0.2 wt%) according to UNI EN ISO 11402:2004 in Phenol Formaldehyde Resole Resins (Resole in water or methanol/ water solvent).

For determination of low free formaldehyde (< 0.2 wt%) in Resole Resin, ensure that your procedure fully complies with UNI EN ISO 11402:2004 its modifications as described below.

### *Test temperature*

Carry out the test 23 +/- 1 °C

### *Test portion*

Weight, to the nearest 0.1 mg, into a 250 ml beaker, a test portion of mass in order to reach a titration difference of  $V_1 - V_0$  of more than 3 ml, if less than 3 ml the weight sample portion should be increased.

### *Determination*

Add 50 ml solvent<sup>\*1</sup> (Methanol or IPA/water) to the content of the beaker with the sampling portion test, switch ON the magnetic stirrer and stir until the Resin has dissolved and the temperature has stabilized at 23 +/- 1 °C.

<sup>\*1</sup>Solvent recommendation:

Methanol - free of aldehyde and ketenes.

IPA/water - 3 volumes of propan-2-ol (free of aldehyde and ketenes) and 1 volume of water.

The accuracy of the formaldehyde determination depends on accurate pH measurement and therefore a minimum water content of 10 % wt is required in the system. Default solvent for this method is Methanol, however for Resole Resins with a low water content it is highly recommended to use IPA/water as solvent.

Introduced the electrodes of the pH-meter in to the solution and, using 0.1 mol/L solution of hydrochloric acid for neutralized resin or the 1 mol/L solution for highly alkaline resins, then adjusted the pH at 3.5.



Pipette into the solution approx. 25 ml of hydroxylamine hydrochloride solution at 23 +/- 1 °C (previously adjusted at pH 3.5). Stir for 10 +/- 1 min. Titrate rapidly, using the 0.1 mol/L, until the pH 3.5, as the starting point. ( $V_1$ )

#### Blank test

Conduct a blank test in parallel with the determination, by the same procedure, using the same reagents as in the determination but omitting the test portion. ( $V_0$ )

#### Expression of result

Calculate the free formaldehyde content expressed as a percentage by mass using the equation:

$$\text{Free Fd} = 0.3002 * (V_1 - V_0) / \text{mass}$$

$V_0$  = volume in ml of the solution of sodium hydroxide utilized for the blank test

$V_1$  = volume in ml of the solution of sodium hydroxide utilized for the determination

Mass = mass of the test portion in grams

Each determination of free formaldehyde should be based on at least a double determination.

Check for systematic errors (e.g. volumetric devices, analytical balance etc.).

The procedure should be carried out quickly and continuously without unnecessary interruptions

#### Analytical Sensibility:

Minimum analytical quantity measurable with the specific analytical technique:

0.029 wt % (as free Formaldehyde)

#### Analytical repeatability:

The analytical repeatability is expressed as value with a level of confidence of 95 % (2x STD)

0.007 wt % (as Free Formaldehyde) in water

0.021 wt % (as Free Formaldehyde) in resole

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