固体中の水分測定　Determination of Water in Solids(C.28.1.)

Introduction:

The concentration of water in solids is an important parameter in many fields - e.g. in the determination of the content of synthetic peptides where the results are based on the amino acid analysis, the counter-ion determination, the content of residual organic solvents and the water content. Both the synthesis strategy and the final dosage are dependent upon these values.

Method:

In contrast to the indirect methods of determination of water whereby products of reaction with water are quantified, our analytical method measures water directly. After thermal desorption at 140°C, water is separated from other volatile components and detected quantitatively. Quantitation is done by the external standard method.

Advantage:

- The determination of water is not interfered with by other hydroxyl-containing components.
- Other volatile components do not falsify results.
- The amount of sample required is approx. 1 mg
- The relative standard deviation of the analysis is 8% in case of 10% absolute water content.
- The limit of detection is approx. 0,1 %.

System Suitability Test (SST):

Injection without sample is done to determine the blind value. It should <0.8%. The peak should have a tailing factor of <3.7.
A sample with known concentration of water is determined within the acceptance criteria.

Chromatography:

Column:	PORAPAK T 4m*2.8mm
Detector:	TCD (Thermal Conductivity Detector)
Injector:	Injector for solids
Injection:	about 0.4mg to 1.5mg
Carrier gas:	Hydrogen
Flows:	Carrier gas 0.6bar, Split 0ml/min
Oven temperature:	Injector: 140°C, Detector 140°C
	Oven: isotherm for 15min, 10°C/min to 150°C
	(Parameters are allowed to change to optimize the separation.)

The peaks are identified by their retention times.
Quantitation is done by external standardization.

Apparatus:

Gas chromatograph:	Agilent 7890 or equivalent
Integrator:	ChromPerfect Spirit or equivalent

Calculation:

$$RF_i = \frac{Area\ External\ Standard}{Amount\ of\ External\ Standard}$$

$$Concentration_i = \frac{Area_i}{RF_i}$$

Asparagine with known amount of water as the external standard
外部標準は水分量既知の
アスパラギンを用いる

Sample preparation:

The sample is weighted in a special, dry micro vessel. This vessel is transferred into the injector. Desorption is achieved at 140°C for 15min.