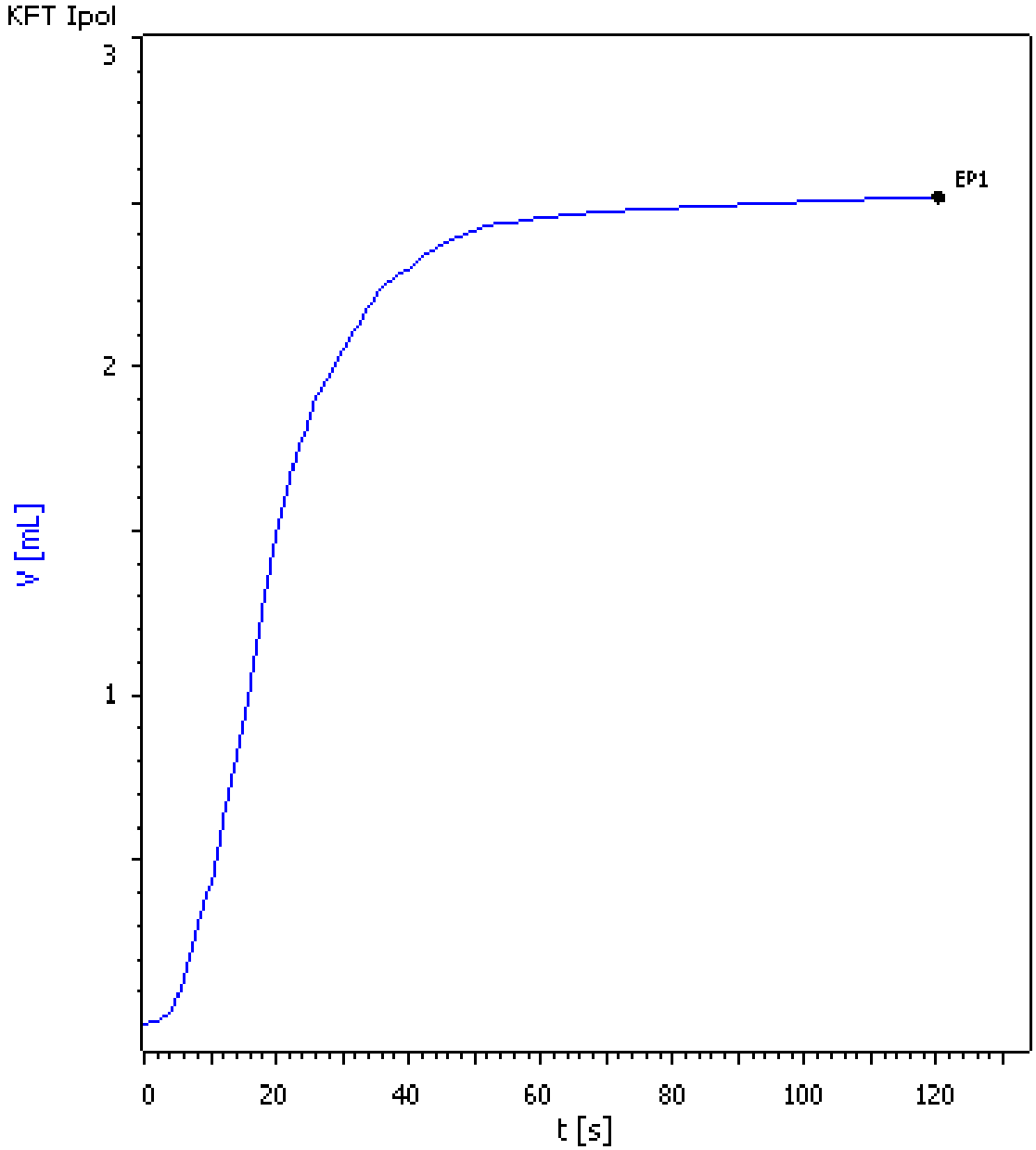
KF Application Note K-54

Determination of the water content in tablets



This Application Note describes the determination of the water content in tablets using automated volumetric Karl Fischer titration including sample preparation (MATi 11).

Metrohm

Method description

Sample

Tablets

- Pretuval® (cold medicine)
- Dafalgan® 500 mg and 1000 mg (analgetic)

Sample preparation

Pretuval® and Dafalgan® 1000 mg are divided in half. Both halves are added to the titration beaker. No sample preparation is necessary for Dafalgan® 500 mg.

Homogenization of the samples is carried out directly in the titration beaker using a Polytron PT 1300 D.

Electrodes

Double Pt electrode	6.9903.048

Reagents

HYDRANAL®-Water Standard 10.0	Fluka 34849
HYDRANAL®-Composite 5	Fluka 34805
HYDRANAL®-Methanol dry	Fluka 34741

Instrumentation

MATi 11 system

Analysis

System preparation

To prepare the system, a blank value is run and the result discarded. An empty beaker is closed with Al foil and holder and placed on the sample rack. After the titration to dryness, the titrant consumption during 300 s is recorded and the drift (mL/s) is stored as Common variable. This value is used for the drift correction of the blank, the titer as well as the sample determinations.

Blank determination

Three empty titration beakers are closed with Al foil and holder and placed on the sample rack. After addition of 50 mL methanol the blank values are determined. The mean value is saved as Common variable and subtracted from the endpoint volumes of all subsequent determinations (titer and sample).

Sample determination

For the sample determination, the samples are weighed in titration beakers and closed with Al foil and holder. Using Dosino and Dosing unit, 50 mL methanol are automatically added. After that, the samples are homogenized using the Polytron PT 1300 D and the water content determination is carried out.

Parameters

Except for the calculation, the parameters for all methods are identical.

The following parameters are changed compared to the default values.

Stop drift	40 µL/min
Extraction time	120 s
Stop volume	10 mL

Results

Drift for drift correction

Drift / [mL/s] (n = 6)	RSD / [%]
0.00054	10.88

Blank determination

Mean / [mL] (n = 6)	RSD / [%]
0.737	0.86

Sample determination

Pretuval®

Water content / [%] (n = 6)	RSD / [%]
3.49	0.70

Dafalgan® 500 mg

Water content / [%] (n = 6)	RSD / [%]
1.38	0.75

Dafalgan® 1000 mg

Water content / [%] (n = 6)	RSD / [%]
1.54	0.92