



Water determination in ground coffee

HYDRANAL™ Laboratory Report L 061 + L 062

Roasted coffee releases the water it contains extremely slowly; residual moisture is tightly bound at cellular level so the results of Karl Fischer (KF) titration at room temperature are too low.

Two series of tests were carried out as a result of this experience:

1. Extraction of the water and subsequent titration of an aliquot.
2. Titration in boiling methanol.

1. Extraction of the water

Approximately 2 g of coffee is precisely weighed into a 100 mL flask with ground stopper, displaced with 50 mL of Hydranal-Methanol dry and the total weight calculated. At 55°C to 60°C, the water is extracted under gentle agitation in 30 minutes. Before the sample is taken, the mixture must be cooled to room temperature.

In order to determine the blank value, 50 mL of methanol is treated in the same way with the methanol always being dosed from a dry pipette or an appropriate dosing device in order to best protect it from humidity.

Procedure for volumetric one-component titration:

Place 30 mL of Hydranal-Methanol dry or Hydranal-Methanol Rapid in the titration vessel and titrate to dryness with Hydranal-Composite 5. Precisely weigh in an aliquot of around 25 mL, determined by differential

weighing, and titrate the water content with Hydranal-Composite 5.

The blank value of the methanol must be taken into account in the calculation.

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Procedure for volumetric two-component titration:

Place 30 mL of Hydranal-Solvent in the titration vessel and titrate to dryness with Hydranal-Titrant 5. Precisely weigh in an aliquot of around 25 mL, determined by

differential weighing, and titrate the water content with Hydranal-Titrant 5.

The blank value of the methanol must be taken into account in the calculation.

2. Determining the water content in boiling methanol

The extraction process as described under point 1 was carried out in boiling methanol for 30 minutes. To evaluate the results, the drying loss at 130°C was also determined after 2 hours, 4 hours, and 6 hours. After drying, the water content was determined

according to KF – it was around 0.4% H₂O. The coffee was also dried with a higher drying loss for 20 hours. Surprisingly, however, the residual water content remained almost unchanged: a doubtful result.

Results

In order to ensure that residual water was actually titrated with the KF titration, and not an ingredient which causes a side reaction with a component of the KF reagent, the residual water was titrated using two different methods. In one, the water was extracted according to the process already described and an aliquot determined using volumetric titration. The second procedure used the indirect method with a KF oven and the sample was heated at 120°C. The water released was then transferred into the coulometry cell with a carrier gas. Comparable values were found with both methods. From this it can be determined that

the dried samples actually contained around 0.4% water. The increasing weight loss can clearly be traced back to a deterioration of the coffee.

We have documented the titration progression at 24°C and 50°C using curves. It could be seen that the titration was not concluded even after 30 minutes. As a result, we used a different method and titrated the roasted coffee directly in boiling methanol at 65°C. After just 16 minutes a very stable 20 sec. end point was achieved; after 30 minutes the end point was stable for 60 seconds.

Drying loss [%] after			Residual water by KF titration [%]	Drying loss [%] after	Residual water by KF titration [%]
4 h	5 h	6 h		20 h	
4.30	4.32	4.39	0.48	4.60	0.41
4.31	4.35	4.43	0.42	4.63	0.44
4.33	4.37	4.43	0.46	4.63	0.48

Titration results at 65°C:

4.59%, 4.61%, 4.56%, 4.57%, 4.60% H₂O

Mean 4.58%, standard deviation 0.02%

Direct titration at 65°C seems to be the method of choice.



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Procedure for titration at 65°C:

An all-glass titration vessel with double wall is provided with a reflux cooler and connected to a thermostat heated to 70°C. Alternatively, a 3-neck flask is used, which is heated with a hemispheric heating mantle.

Prior to heating, 50 mL of Hydranal-Methanol dry is placed in the titration vessel and dry titrated with Hydranal-Composite 5 during the boiling. In this way, the apparatus is kept completely dry and determination of the blank value is omitted.

After gentle cooling, around 1 g of sample is precisely weighed in, the sample is boiled at the reflux, and simultaneously the water content is titrated with Hydranal-Composite 5.

Hydranal-Water Standard 10.0, Hydranal-Water Standard 1.0, and Hydranal-Standard Sodium Tartrate Dihydrate are suitable for determination of the titer or control of the volumetric determination.

Summary

Roasted coffee releases the water only slowly and incompletely during extraction with methanol, and in the subsequent KF titration of the extract, the water contents found are too low. KF titration in boiling methanol gives the best extraction and reliable results.

VOLUMETRIC REAGENTS

34805	HYDRANAL-Composite 5	34801	HYDRANAL-Titrant 5
34741	HYDRANAL-Methanol dry	34800	HYDRANAL-Solvent
37817	HYDRANAL-Methanol Rapid		

WATER STANDARDS

34849	HYDRANAL-Water Standard 10.0	34696	HYDRANAL-Standard Sodium Tartrate Dihydrate
34425	HYDRANAL-CRM Water Standard 10.0		
34828	HYDRANAL-Water Standard 1.0	34424	HYDRANAL-CRM Sodium Tartrate Dihydrate
34426	HYDRANAL-CRM Water Standard 1.0		

AUXILIARIES

34241	HYDRANAL-Molecular Sieve 0.3 nm	34788	HYDRANAL-Humidity Absorber
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