

Automated Kappa number determination of pulp

Description

The Kappa number describes the Lignin content of pulp which gives information about the bleaching process of the pulp. The longer the pulp was cooked and bleached the lower the Kappa number is.

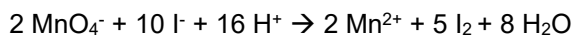
For the determination of the Kappa number a sample weight adjusted to the expected result is chopped and inserted into a beaker, then 400 ml of water is added. The sample/water mixture is stirred for 10 minutes. Afterwards 50 ml of potassium permanganate (KMnO₄, 0.02 mol/L) and 50 ml sulfuric acid (H₂SO₄, 2 mol/L) are added simultaneously. The mixture is stirred for another 10 minutes. The reaction is stopped by adding 10 ml potassium iodide (KI, 1 mol/l). The excess of KMnO₄ reacts with iodide to iodine, which is titrated with sodium thiosulfate (Na₂S₂O₃, 0.2 mol/L).

Chemical equations:

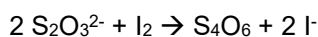
The weight is adjusted to a consumption of about 50% of the MnO₄⁻.



The reaction is stopped by adding KI.



The formed Iodine is titrated with Na₂S₂O₃.



Calculation of the Kappa number:

1. Calculation of the consumed volume V_a of KMnO₄:

$$V_a = \frac{(V_1 - V_2)c}{0.1}$$

with

V_1 = Consumption of Na₂S₂O₃ during blank titration

V_2 = Consumption of Na₂S₂O₃ during sample titration

c = Concentration of Na₂S₂O₃

0.1 = numerical Factor, calculated from the molarity of the permanganate and the stoichiometric factor f of the reaction: $c_{\text{KMnO}_4} \cdot f = 0.02 \cdot 5$

2. Calculation of a correction factor d , which corrects the consumption of permanganate depending on V_a to a consumption of 50%.

$$d = 10^{0.00093(2V_a - 50)}$$

$$= e^{\ln(10) \cdot (0.00093(2V_a - 50))}$$

3. Calculation of the Kappa number corrected to 25 °C.

$$K = \frac{V_a \cdot d}{m} \cdot (1 + 0.013(25 - T))$$

with

T = actual temperature, measured during the titration

m = sample weight in g

Instrumentation

Titratör	TL 7000 or higher
Exchangeable head	WA 20
Electrode	Pt 6980
Cable	L 1 A
Bürettes	T 500 with 50 ml exchangeable head
	T 300 with 50 ml dosing unit
	T 300 with 20 ml dosing unit
Sample changer	TW alpha plus with 12 Position sample tray TZ 1453
Stirrer	Rod stirrer TZ 1844 with propeller blade TZ 1863
Pump	Membrane pump MP 25
Miscellaneous	TitriSoft, Thermometer W 5780 NN
Laboratory glassware	600 ml beakers, high form, without spout

A complete list of all required components incl. order numbers is available on request.



Figure 1: Complete set-up for an automated Kappa number determination



Figure 2: Titration head, view 1



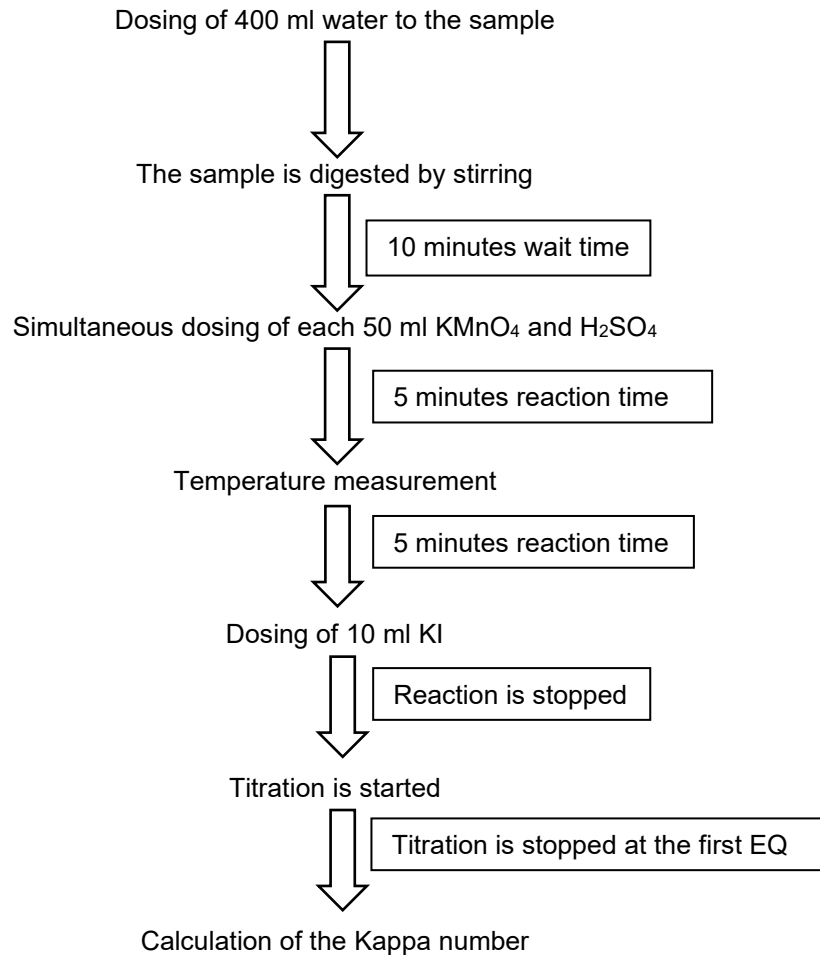
Figure 3: Titration head, view 2

Reagents

1	Deionized water
2	Potassium permanganate, 0.02 mol/L
3	Sulfuric acid, 2 mol/l
4	Potassium iodide, 1 mol/L
5	Sodium thiosulfate, 0.2 mol/l
All reagents should be of analytical grade or better.	

Titration performance

All instruments are controlled and the titration performed by the titration software TitriSoft. The principle structure is shown below. All steps are performed automatically by TitriSoft.

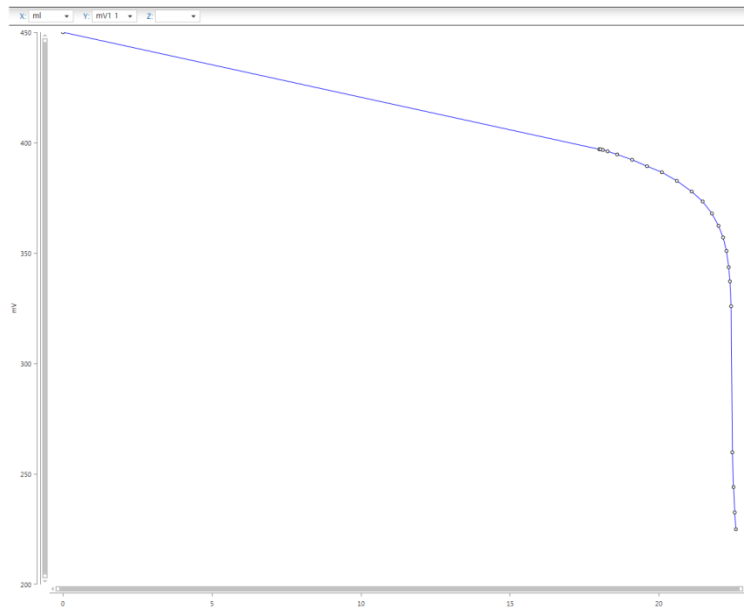


The performance of the blank determination is identical with the sample titration. The consumption at EQ is evaluated and stored as global variable.

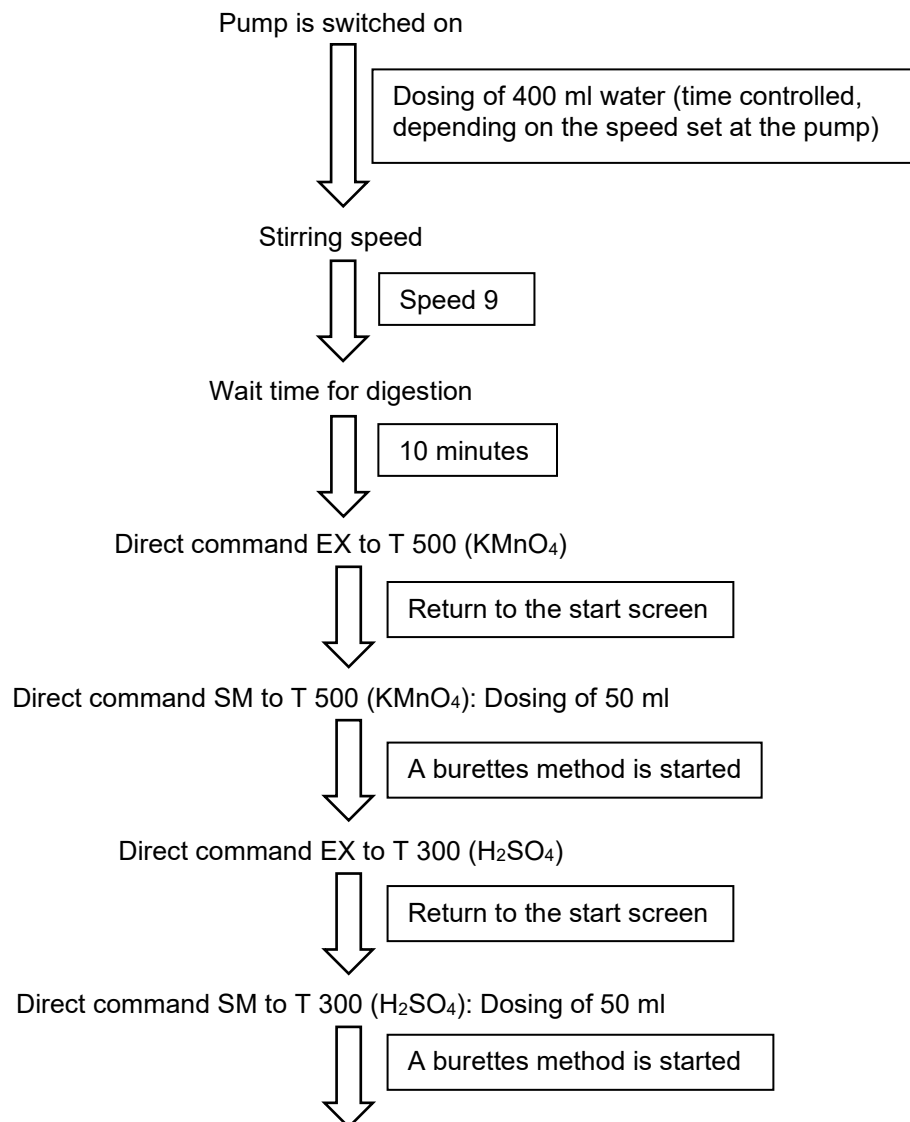
The complete structure of the titration commands is shown in the annex of this application report.

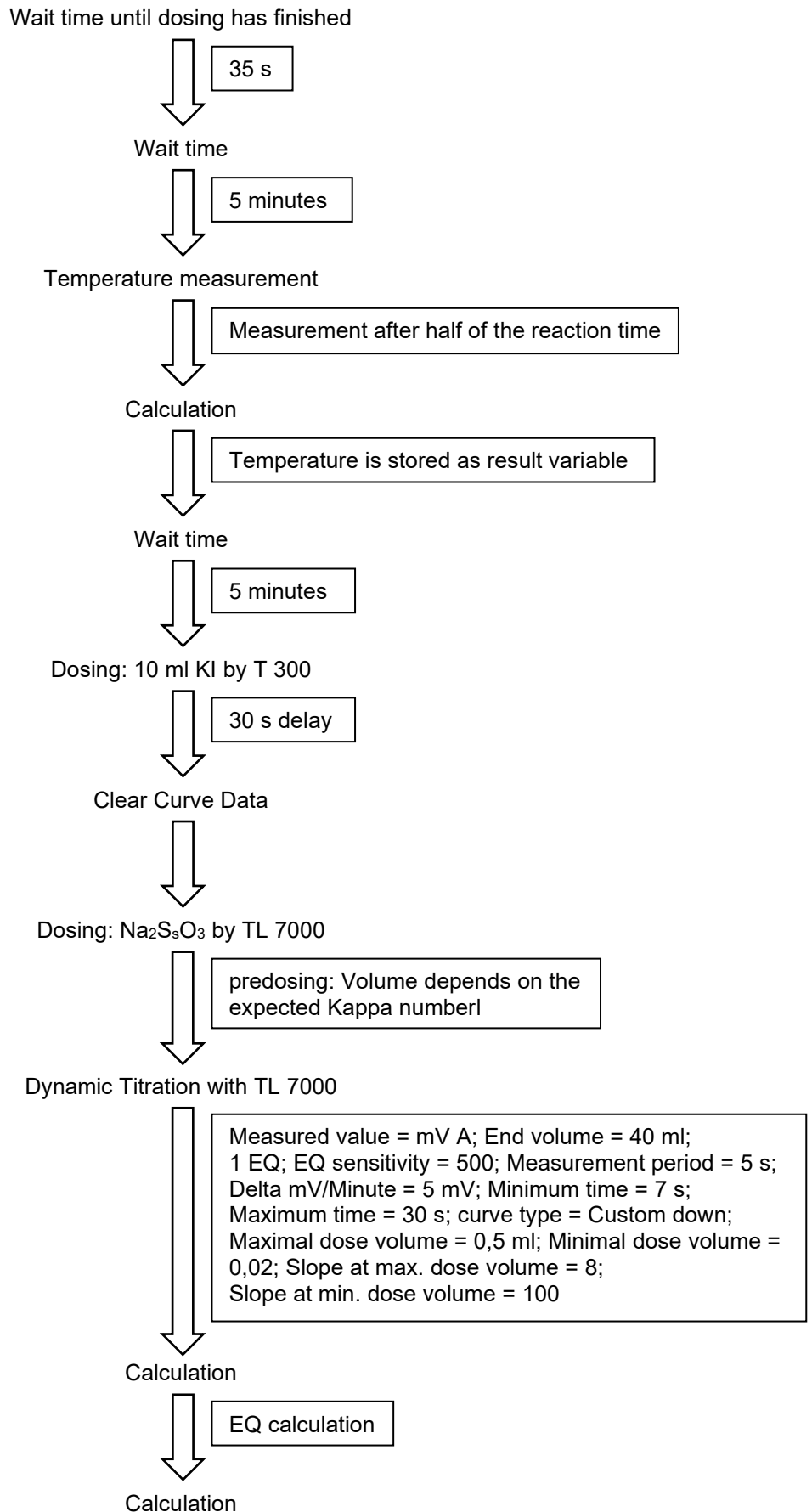
A configured database with all required methods and settings for the automated Kappa number determination is available on request.

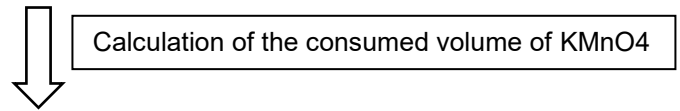
Example curve:



Annex: Flow chart of all required TitrSoft titration commands







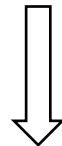
Calculation of the consumed volume of KMnO₄

Calculation



Calculation of the correction factor d

Calculation



Calculation of the Kappa number corrected to a temperature of 25 °C

Stirring Speed



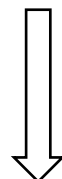
Speed 0

Direct command KH to TW alpha plus



Head of the sample changer is lifted

Wait time



15 seconds (drain time to avoid a contamination of other samples)

End of titration, rinsing process (first three positions) starts automatically