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 ($KMnO_4$, 0.02 mol/L) and 50 ml sulfuric acid (H_2SO_4, 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, 1mol/l). The excess of $KMnO_4$ reacts with Iodide to Iodine, which is titrated with sodium thiosulfate ($Na_2S_2O_3$, 0.2 mol/L).

Chemical equations:

The weight is adjusted to a consumption of about 50% of the MnO_4^- .

Lignin + MnO_4^- + 4 H^+ → oxidised Lignin + MnO_4^- (excess) + MnO_2 + 2 H_2O

The reaction is stopped by adding KI.

2 MnO_4^- + 10 I^- + 16 H^+ → 2 Mn^{2+} + 5 I_2 + 8 H_2O

The formed Iodine is titrated with $Na_2S_2O_3$.

2 $S_2O_3^{2-}$ + I_2 → S_4O_6 + 2 I^-

Calculation of the Kappa number:

1. Calculation of the consumed volume V_a of $KMnO_4$:

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

with

V_1 = Consumption of $Na_2S_2O_3$ during blank titration
V_2 = Consumption of $Na_2S_2O_3$ during sample titration
c = Concentration of $Na_2S_2O_3$
0.1= numerical Factor, calculated from the molarity of the permanganate and the stoichiometric factor f of the reaction: $c_{KMnO_4} \cdot f = 0.02 \cdot 5$

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

Titrator	TL 7000 with 20 ml interchangeable unit
Electrode	Pt 6980
Cable	L 1 A
Bürettes	T 500 with 50 ml interchangeable unit
	T 300 with 50 ml interchangeable unit
	T 300 with 20 ml interchangeable unit
Sample changer	TW alpha plus with 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.

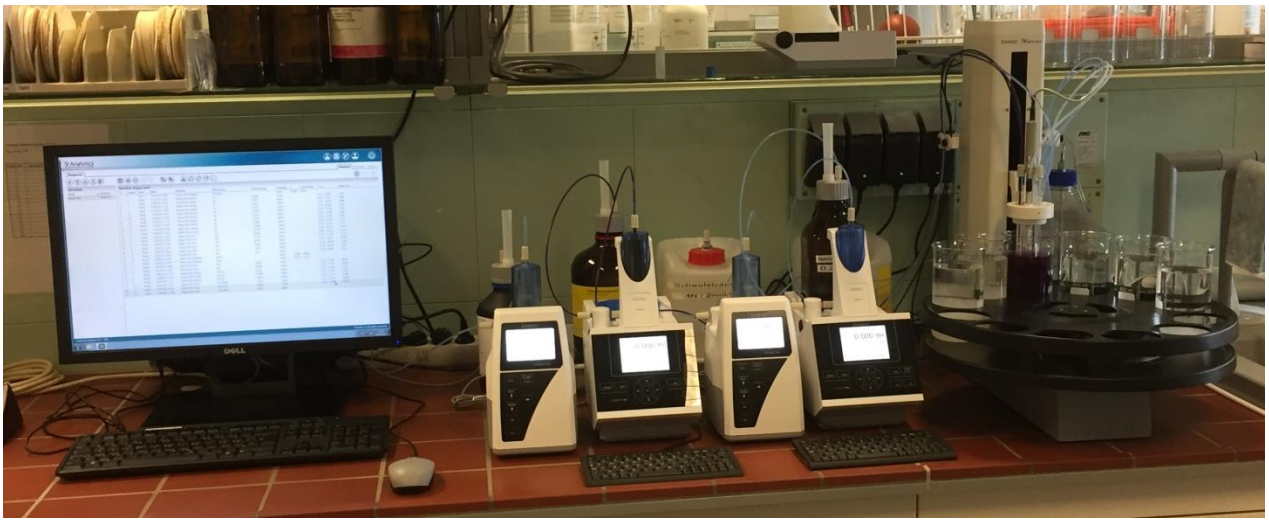


Figure 1: Complete set-up for an automated Kappa number determination

Figure 2: Titration head, view 1

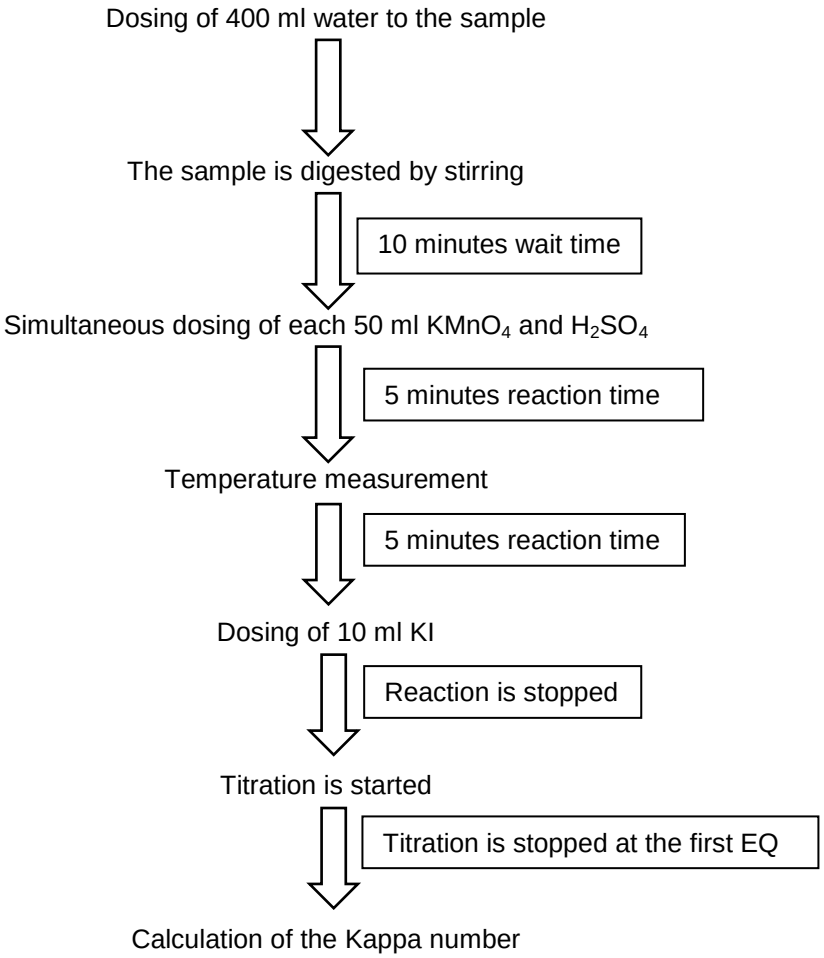
Figure 3: Titration head, view 2

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.

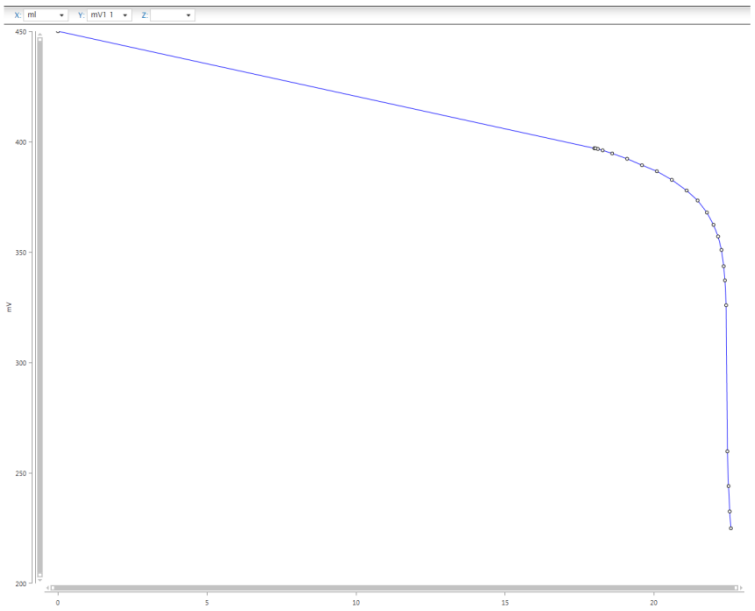


The performance of the blank determination is identical with the sample titration. The consumption at EQ is evaluated and stored as global variable.

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:



Any questions? Please contact the application team:

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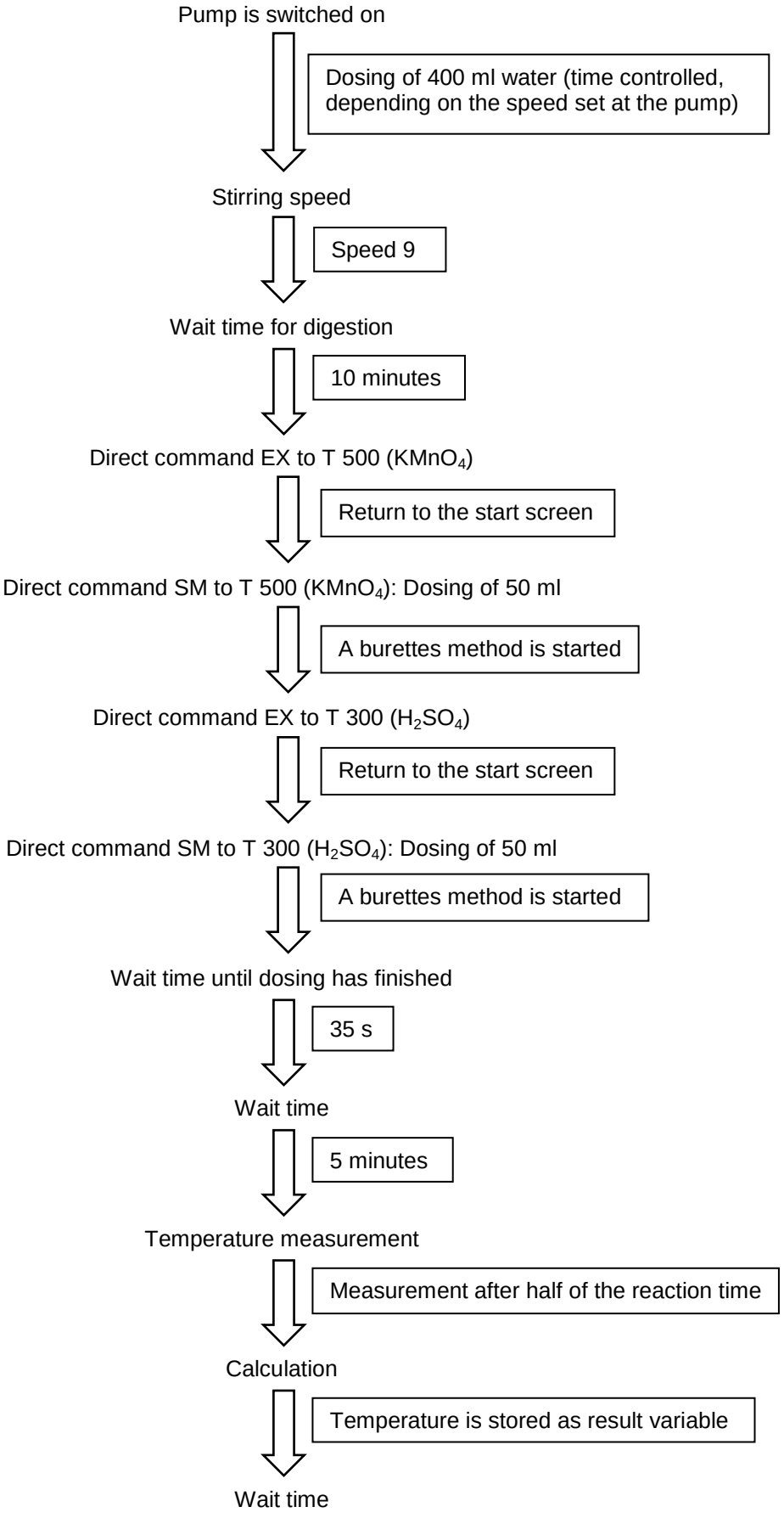
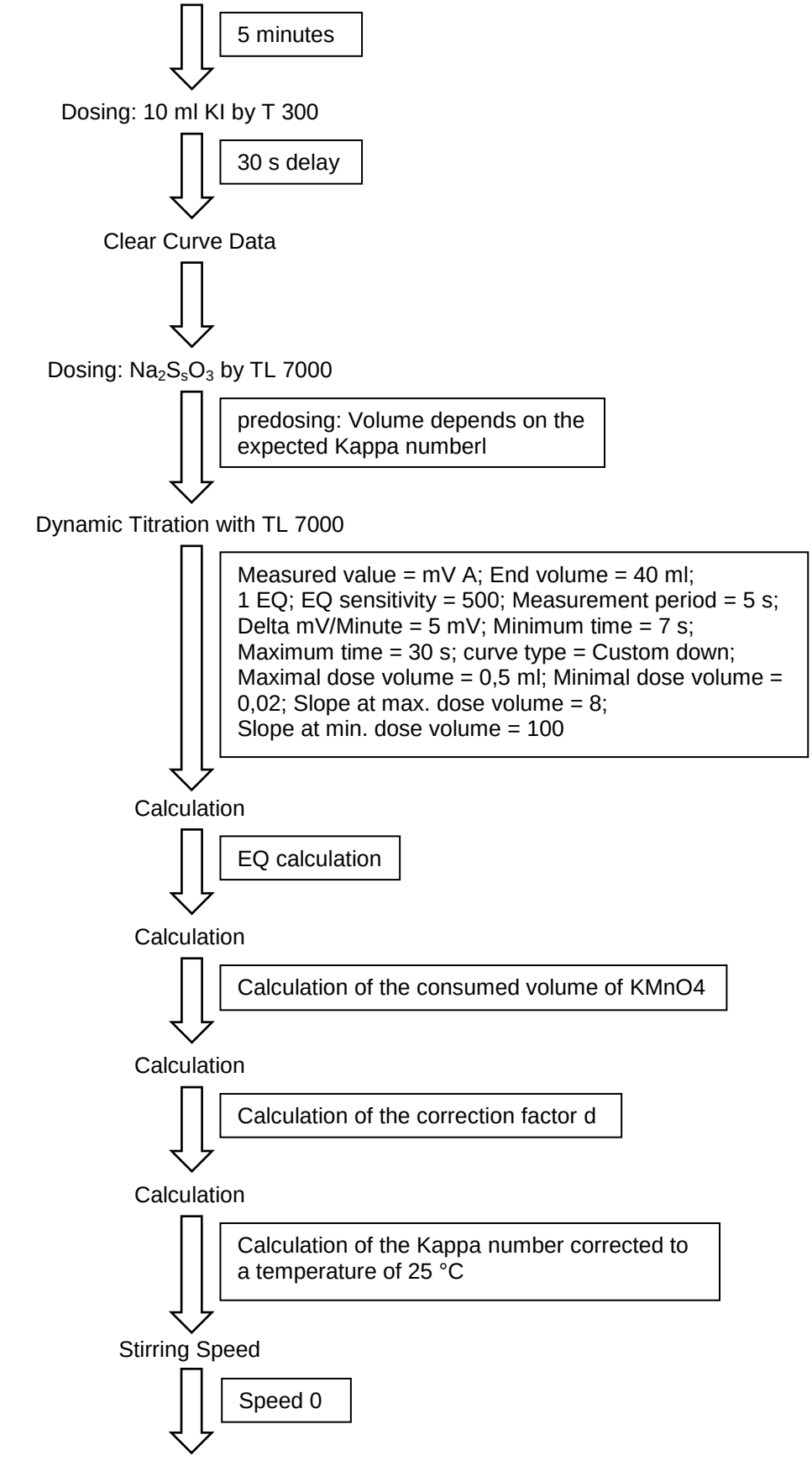
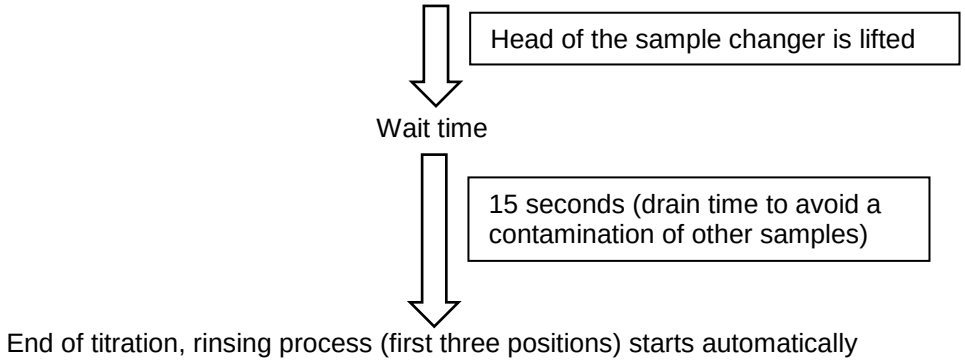
Annex: Flow chart of all required TitriSoft titration commands





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