Determination of Formaldehyde in aqueous solution

Description

The content of a formaldehyde solution can be determined by an acid/base titration. For this purpose, the reaction of formaldehyde with sodium sulphite is used, in which NaOH is released.
An excess of sodium sulphite (Na_2SO_3) is added to the sample. This produces the addition compound HCH(OH)-SO_3Na and NaOH. The released NaOH is titrated with 0.1 mol/L hydrochloric acid.
The formaldehyde content is calculated in %.

$$HCHO + Na_2SO_3 + H_2O \rightarrow HCH(OH)\text{-}SO_3Na + NaOH$$

Instruments

Titrator	TL 5000, TL 7000 or higher
Exchangeable head	WA 50
Electrode	N 62, A 162 2M DIN ID or A 7780
Cable	L1A (for electrodes with plug head)
Stirrer	Magnetic stirrer TM 235 / TM 50
Lab accessory	Glass beaker 150 mL
	Magnetic stirrer bar 30 mm

Reagents

1	Hydrochloric acid (HCl) 0.1 mol/L
2	Sodium Sulfite (Na_2SO_3)
3	Buffer pH 4.00
4	Buffer pH 7.00
5	KCl solution 3 mol/L
6	Distilled Water
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

HCl 0.1 mol/L

HCl is available as a ready-to-use solution.
The titer determination of the HCl is done as described in the application note "Titer determination of strong acids".

Na_2SO_3 0.1 mol/L

12,6 g of Na_2SO_3 are dissolved in dist. Water and made up to 1.0.

The Na_2SO_3 must not contain any carbonate or hydroxide. The exact content of the solution is not important.

Cleaning and storage of the electrode

The electrode is cleaned with distilled water. KCl solution 3 mol/l or electrolyte solution L911 is suitable for storage of the electrode.

The pH electrodes must be calibrated regularly (weekly). Two-point calibration with buffer pH 4.00 and pH 7.00 is recommended. If the slope is <95% the electrodes must be replaced.

Sample preparation

The sample is weighted into a 150mL beaker, 30 mL Na_2SO_3 solution 0.1 mol/L are added and made up up to 60 – 80 mL with dist. water. Water. The sample is titrated with hydrochloric acid 0.1 mol/L to one EQ.

The sample amount should be chosen so that about 5 - 50mg formaldehyde are contained. The required sample amount can be estimated according to this rule of thumb:

$$W(g) = \frac{35 * Titer\ [\frac{mol}{L}]}{expected\ HCHO - Content\ [\%]}$$

To check whether there is an excess of sulphite, it can be titrated to 2 EQs. The formaldehyde content is calculated from EQ1. The 2nd EQ only shows that there really was an excess of sulphite and corresponds to the reaction:

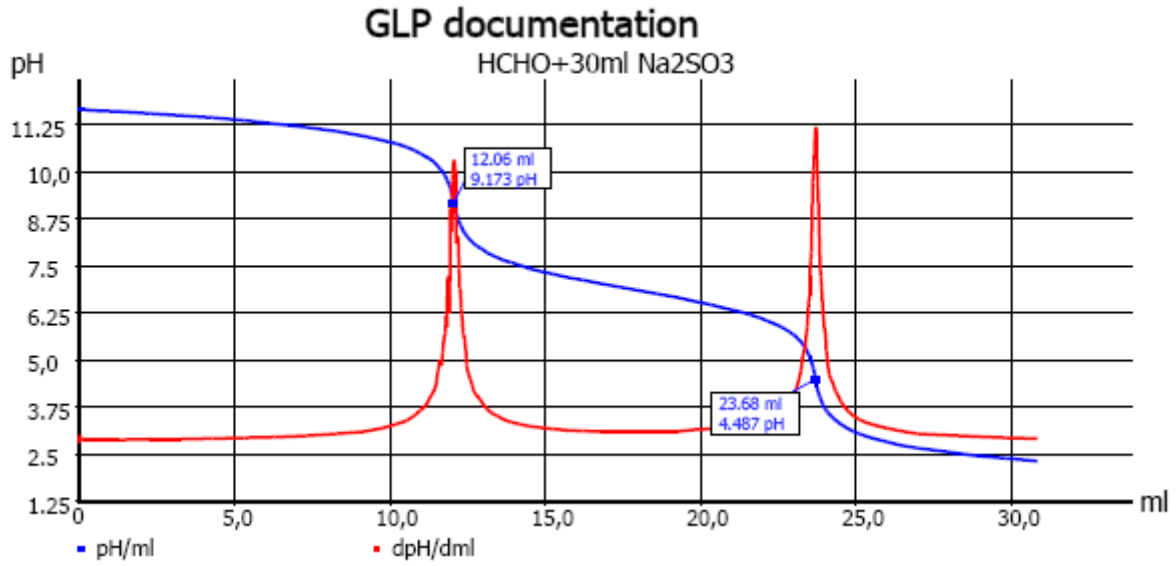
$$HSO_3^- + H^+ \rightarrow H_2SO_3$$

For samples with very low HCHO contents, the amount of Na_2SO_3 can be reduced.

For high HCHO contents, 1 molar solutions can be used instead of 0.1 molar solutions.

Titration parameter

Sample titration



Default method	---		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	normal	Minimum holding time	2 s
		Maximum holding time	15 s
		Measuring time	2 s
		Drift	20 mV/min
Initial waiting time	0 s		
Dynamic	average	Max step size	1.0 mL
		Slope max ml	10
		Min. step size	0.02 mL
		Slope min. ml	120
Damping	none	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (2)*	Slope value	300
Max. titration volume	30 ml		
Dosing speed	100%	Filling speed	30 s

* EQ 2 is only used to check whether there is an excess of sulphite, it is not needed to calculate the result. The final criterion EQ can also be set to 1 EQ to save titrant. It is not possible to determine a second EQ with a TL 5000.

Calculation:

The result is calculated as Formaldehyde (HCHO) in %:

$$HCHO\ \% = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

EQ 1		Consumption of the titrant on the first EQ [ml]
B	0	Blank value
T	WA	Actual concentration of the titrant [mol/L]
M	30,03	Molecular weight of Formaldehyde [g/mol]
W	m	Sample amount [g]
F1	0.1	Conversion factor
F2	1	Conversion factor

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