Determination of Formaldehyde using Classical Kjeldahl Instrumentation for Steam Distillation.

S.Diezi, T.Ziolko, B.Haag
Büchi Labortechnik AG, Postfach , 9230 Flawil, Switzerland

Introduction

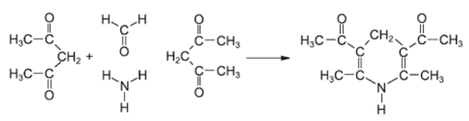
The AOAC method 964.21 describes the determination of formaldehyde in maple syrup. This official method was released in 1964 and specifies dedicated equipment, which is sorted out in today's laboratories (e.g. condenser with asbestos insulation). This work describes the formaldehyde determination following the relevant instructions of AOAC method 964.21 but using modern instrumentation (classical Kjeldahl instrumentation, spectrophotometer), which are simple to use, cost effective and available in today's laboratories.

Official regulations

- Determination of formaldehyde in emulsion paints
  (German Association of Lacquer Industry) [3]
- Determination of formaldehyde in maple sirup
  (Official AOAC Method 964.21) [4] .
- Determination of formaldehyde in textiles
  (European Norm ISO 14184-1) [5] .
- Determination of formaldehyde in household products
  (Official test method in Japan Law 112).
- Determination of formaldehyde in water
  (US National Environmental Methods Index , NEMI D6303). [6]

Methodology

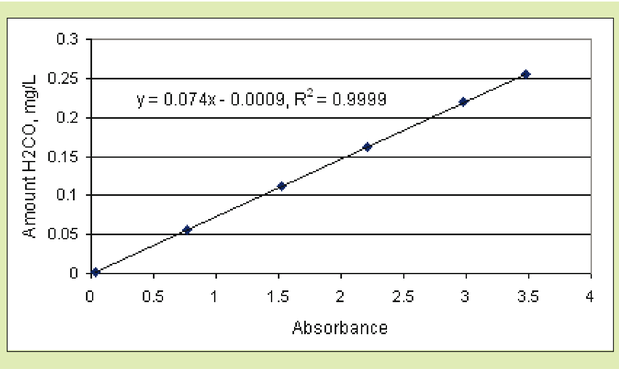
This method describes a procedure for the quantitative determination of formaldehyde in a diluted formalin solution in concentration range of > 0.220 mg/L. The determination of formaldehyde is done by steam distillation, followed by spectrophotometry. Formaldehyde reacts with acetylacetone in the presence of ammonium acetate to form 3,5-diacetyl-1,4-dihydrolutidine. (Hantsch-Reaction [1]) The absorption of this compound is measured at 412 nm with a spectrophotometer.



Spectrometric Determination and Calibration Procedure

Determination of the formaldehyde content of a standard solution Pipette 20.0 mL of the standard solution (3 g of formaldehyde solution (≥36.5% in H2O) diluted with deionized water to 1000ml) into a beaker, add 25.0 mL of a 0.05 mol standard iodine solution and 10.0 mL of 1 mol sodium hydroxide solution. After 5 min acidify with 11.0 mL of 1 mol hydrochloric acid and determine the excess iodine by titration with a 0.1 mol standard sodium thiosulphate solution.

From each of the 10 mL volumetric flasks from the dilution series transfer 2.5 g of the solution to a 25 mL volumetric flask. Dilute this solution with 10 mL of the acetylactone reagent and fill up to the 25 mL with deionized water. After 2 hours record the adsorption of the formaldehyde / acetylactone reagent complex at 412 nm using cells with an optical path length of 10 mm.



According to the DIN 32645 [2] the following characteristics can be evaluated:

Detection limit:	0.061 mg/L
Limit of determination:	0.123 mg/L
Quantification limit:	0.219 mg/L

Distillation of formaldehyde in samples

Distillation Unit:	Buchi K-355
Steam power:	100 %
Distillation time:	12 min
Distillation tube volume:	300 mL
Receiver vessel volume:	500 mL
Distillate volume approx.:	400 mL

1. Prepare the Buchi Distillation Unit K-355 by preheating the unit
2. Pipette the sample into the distillation tube
3. Add 50 mL deionized water and 20 mL H_2S0_4 (25%) by pumping the acid with the acid resistant pump
4. Add 30 ml deionized water to the receiving vessel
5. Attach the tube to the instrument and add 10 mL sulfuric acid 18 N by pumping the acid with the acid resistant pump.
6. Start the distillation

Spectrophotometrical measurement

- Transfer 2.5 g of the distillate to a 25 mL volumetric flask
- Add 10 mL of the acetylacetone reagent und fill up to the 25 mL with deionized water
- After 2 hours record the adsorption of the formaldehyde/acetlacetone reagent complex at 412 nm using cells with anoptical path length of 10 mm

Verification

For verification, the determination of formaldehyde has been confirmed by the analysis of a 0.25 mg/L formaldehyde solution.
Using the calibration curve the formaldehyde concentration can be calculated after measuring the extinction of the samples and thus the recoveries.

Sample No.	Recovery [%] of 0.25 mg/L sol.
1	99.18
2	98.54
3	98.78
Mean value	**98.83**
RSD [%]	**0.33**

Conclusion

The threefold determination of the formaldehyde-content of a 0.25 mg/L formaldehyde solution gives a recovery of 98.8% with a small RSD of 0.3%.

The use of steam distillation as a separation process is a proven and cost-effective method utilised in many areas of food production, animal feed manufacture, cosmetic industry and environmental analysis. The presented Buchi method for the determination of formaldehyde is based on a modification of the classical Hantsch-Reaction. The method is fast and shows comparable method performance.

References

[1] T. Nash, Biochem J., 55(3), 416-421, 1953.
[2] DIN 32645
[3] Verband der Lackindustrie, VdL-Richtlinie
Formaldehydbestimmung 03. Mai 1997
[4] AOAC Official Methode 964.21
[5] EN ISO 14184-1
[6] US National Environmental Methods Index , NEMI D6303

BUCHI Labortechnik AG
9230 Flawil / Switzerland
T+41 71 394 63 63
F+41 71 394 65 65
www.buchi.com

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