

Methanol in Softdrinks

Test of cold sterilisation with DMDC

Application APP-PHM-0004

General

Depending on national laws and regulations, dimethyl dicarbonate (DMDC, E242, INS 242) can be used for the cold sterilisation of a variety of beverages. After it has been in a drink only a short time, DMDC breaks down completely (hydrolyses) into carbon dioxide and methanol (MeOH). From 250 parts DMDC, 120 parts MeOH are produced in this process (factor 0,48). This application describes the photometric determination of the methanol produced from DMDC in drinks. As part of this process, possible interferences with the test due to ingredients in the drinks (oxidation and reduction agents, e.g. ascorbic acid) are taken into account.

Method

Determination of methanol concentration in soft drinks: The enzyme alcohol oxidase catalyses the reaction of alcohol with water and acid to produce acetaldehyde and hydrogen peroxide. The hydrogen peroxide produced combines with aminophenazone and a derivative of benzoic acid, catalyzed by a peroxidase, to form a red, quinoid colorant. This is measured in the photometer.

The pH value of the sample must be between 2 and 6.

Material

LPV422.99.00001	Spektrophotometer DR 2800 or
LPV424.99.00001	Spektrophotometer DR 3800 or
LPV408.99.00001	Spektrophotometer DR 5000 or
LPV440.99.00011	Spektrophotometer DR 3900 or
LPV441.99.00011	Spektrophotometer DR 6000
LCK300	Cuvette test Alcohol
LCX300	Methanol (6g/l) or
	Methanol p.A. 99.8%
LTV073	Thermostat LT20 or water bath
	and/or temperature chamber 20°C

When using the DR 2800 / DR 3800 / DR 5000 / DR 3900 / DR 6000 for the first time:

Download the additional evaluation Methanol in Softdrinks APP-PHM-0004 as an application from the Internet.

- Go to **www.hach-lange.com** and search under **LCK300** and **Documents and Software** the application **Methanol in Softdrinks** and save it on your PC.
- Open the zipped file with a double-click and save the folder used for your photometer to a USB stick:
 - DR 2800 / DR3800 dbhlc
 - DR 5000 dbhl
 - DR 3900 dbhlm
 - DR 6000 dbhlh
- Take the USB stick and upload the application to your photometer.
- In the PDF file you will find the application note with detailed description.

For further information, please see the operating instructions for your photometer.

Determination of alcohol content

Samples

Required:

- Sample 1: 50 ml drink formulation with DMDC not yet added
- Sample 2: 50 ml drink formulation that has undergone quarantine period
- Sample 3: 10 ml of sample 1 (prior to addition of DMDC) with addition of 120 mg/l methanol

Sample preparation

- Degas samples containing carbonic acid (for 1 minute, stirring continuously)
- Add 10 ml of sample 1 to 0,2 ml methanol parent solution (6 g/l) (= sample 3; formulation and testing of the parent solution, see appendix)
- Dilute samples 1, 2 and 3 at a ratio of 1:5 with distilled water (1 ml sample + 4 ml distilled water). The measuring range there is 10–150 mg/l methanol (originally produced from a dosage of 1,6–25 ml/hl DMDC).
- If the samples become turbid, filter 10 ml of each with membrane filtration set LCW916.

Analysis

- Set water bath or thermostat/temperature chamber to 20°C, switch on and leave running for approx. 30 minutes.
- Remove three cuvettes from LCK300 packaging, label them S1, S2 and S3

- Remove screw cap, discard and screw on Dosicap.
- Release the contents of the Dosicap by tipping it up and down 4–5 times. Do not shake it violently; the formulation foams up easily!
- Unscrew the Dosicap again and add 0,2 ml to each prepared sample using pipette. Close cuvette again using the Dosicap.
- Mix the contents of the cuvette by tipping it up and down 4–5 times. Again, do not shake; the formulation foams up easily!
- Immediately place the cuvettes in the water bath/thermostat/temperature chamber.
- Set timer to 45 minutes and then start it.
- On the measurement instrument, select user test DMDC
- After 45 minutes, immediately before the measurement, set to zero with the zero cuvette from the packaging.
- Remove test cuvettes from the water bath/thermostat/temperature chamber, clean thoroughly and measure in the order P1, P2, P3.

Evaluation

Following the measurement of P3 the results are shown immediately in ml/hl DMDC.

The additional display % yield is only to be used for assessing the applicability of the process to the specific drink sample and should be between 80% and 120%.



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Appendix:

Methanol parent solution, 6 g/l

A parent solution consisting of 99,8% methanol is created in distilled water.

- Fill a 100 ml volumetric flask with approx. 80 ml distilled water, place on weighing scales and tare to 0.
- Add exactly 0,600 g methanol (99,8%) using a pipette.
- Then fill the volumetric flask with distilled water to 100 ml.

Test of the methanol parent solution

- Add 0,200 ml of the methanol parent solution (6 g/l) to a 50 ml volumetric flask and fill up to the mark with distilled water.
- Carry out cuvette test LCK300 with the help of the work instructions, with 0,2 ml from the dilution as a sample.
- After the reaction time of 45 minutes (20°C), select the Methanol user test on the photometer.
- Set the instrument to zero with the zero cuvette and then measure the test cuvette with the diluted parent solution. The result must always be 24 ± 2 mg/l.

Example from practical application

An apple spritzer, a forest fruits spritzer and a health drink were tested.

Calculation DMDC MeOH	Apple spritzer	Apple/forest fruits	Health drink
Fruit juice content	60%	55%	3%
Additives	Lemon juice Carbonic acid Natural flavorings	Lemon juice Carbonic acid Natural flavorings	Lemon juice Carbonic acid Natural flavorings Various kinds of sugar Yerba mate extract Citric acid Ascorbic acid Green tea extract Vitamin mix
Color	yellow	red	light yellow
MeOH recovery:	100,5%	93,7%	-1,4%
MeOH concentration in the sample determined by DMDC conditioned on	113,5 mg/l	113,3 mg/l	not applicable
C additive for filling:	20,6 ml/hl	20,34 ml/hl	22,55 ml/hl
Theoretical MeOH concentration in the sample determined by DMDC conditioned on	ca. 120 mg/l	ca. 120 mg/l	ca. 130 mg/l
Analyses comparable samples by means of headspace gas chromatography produced the following results: (MeOH mean value)	110 mg/l	99 mg/l	not identified

Results

- This method for determining methanol Concentration can be used for the fruit juice spritzers but not for the health drink, because the additives in the health drink interfere with the analysis.
- The methanol concentration in the fruit juice spritzers recorded using this method corresponds within tolerable levels (+/- 10%) to the values from the headspace gas chromatography.
- The methanol concentration determined by the DMDC in the fruit juice spritzers calculated using this method corresponds within tolerable levels (+/- 10%) to the concentrations that were predicted.

Disposal information

Waste disposal must be carried out in compliance with regional and national regulations.



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