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EZ7252 Volatile Fatty Acids and Total Alkalinity analyser

Method and reagent sheets

10/2021, Edition 1.10

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1. Legal information

Manufacturer: AppliTek NV/SA

Distributor: Hach Lange GmbH

The translation of the manual is approved by the manufacturer.

2. Analytical specifications

Please refer also to the respective technical datasheet at Hach Support Online.

VFA, TA - All specifications			
Analysis method	Acid-base titration		
Parameter	Volatile Fatty Acids (VFA), Total Alkalinity (TA)		
Cycle time	Standard measurement cycle time: 15 minutes		
Limit of detection (LOD)	≤ 100 mg/L VFA; ≤ 250 mg/L TA		
Precision/Repeatability	Better than 3% full scale range for standard test solutions		
Cleaning	Automatic; frequency freely programmable		
Calibration	Automatic; frequency freely programmable		
Validation	Automatic; frequency freely programmable		
Interferences	Phosphates and similar dissociating ions and non-fatty acids which on acidification from undissociated acids may cause interference. Sulphide [$(S)^{2-}$] may deteriorate some types of pH electrodes. Fats, oil, proteins, surfactants and tar.		
Measuring range	**Parameter**	**Low range (mg/L)**	**High range (mg/L)**
	VFA	100	5000
	TA	250	5000

3. Analysis method

Summary

The determination of Volatile Fatty Acids (VFA) and Total Alkalinity (TA) is based on two algorithms, combined in one analyser.

The TA concentration is determined by titration with sulfuric acid. The VFA concentration is determined by titration with sodium hydroxide after removal of the carbonates from the sample by aeration.

Calibration

The calibration procedure measures a REF2 VFA solution (channel 10, REF2 valve).

3.1 Total (and Partial) Alkalinity

The sample is titrated with sulfuric acid (H_2SO_4) using a combined pH electrode. Total Alkalinity (TA) of the sample is determined by titration from the original sample pH to a pH of 4.3. The titration continues until the pH drops below pH 4.00.

3.2 Stripping of carbonates

When the alkalinity titration algorithm has finished, the carbonates that are present in the sample are stripped by aeration. During this action it is possible that the pH increases slightly over pH 4.00. Sulfuric acid is automatically dosed until the pH value is below 4.00 again. An alarm message is triggered when the acid dosage is higher than a pre-set value, indicating that the reagent container is empty. The reagent must then be replaced, the tubing primed and the alarm message must be reset before restarting on-line measurements.

3.3 Volatile Fatty Acids

The sample is titrated with sodium hydroxide (NaOH) using a combined pH electrode. The titrant required to titrate a sample from pH 4.0 to 5.0 can be considered proportional to the content of VFA in the sample. The stripping of the carbonates in an earlier step during the analysis program eliminates their interference.

4. Reagents

⚠ CAUTION	
⚠	Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Read the safety data sheet from the supplier before bottles are filled or reagents are prepared. For laboratory use only. Make the hazard information known in accordance with the local regulations of the user.

⚠ CAUTION	
⚠	Chemical exposure hazard. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

4.1 Reagent overview and consumption

In the tables below, the products that are needed to prepare the reagents are listed. The product name, the formula, the molecular weight, the CAS No. and the amount needed to prepare 1 liter of the reagents is given. Check the consumption of the reagents (28 days) to adapt the volumes needed.

Product	Consumption	Consumption/28 days A rata 1 analysis /15 min	Recommended containers
H_2SO_4	Conc. Dependent	1 L < Volume < 27 L	Plastic – 10 L
	1 mL / analysis (for Rinse purposes)	~ 2.7 L	
NaOH	Conc. Dependent	1 L < Volume < 17.5 L	Plastic – 10 L
Anti-Foam*	~ 0.05 mL	< 0.5 L	Plastic – 2.5 L
REF2 Solution	~ 1 L / calibration	/	Plastic – 2.5 L

* Default-setting: no addition of anti-foam. Addition of anti-foam solution is optional.

4.2 DI-water overview and consumption

	Rinse water (mL/analysis)	Dilution water (mL/analysis)	Total (mL/analysis)	Consumption/28 days A rata 1 analysis / 20 min
0	N.A. (Tap water for rinsing)	N.A.	N.A.	N.A.

Remark

The indicated volumes are an estimation of the consumption for rinse and dilution water, based on a standard operating procedure, as defined in the specifications of the EZ analyser. Please be aware that, depending on the sample matrix, the rinse water volumes might increase.

4.3 Storage and quality of chemicals

Quality of chemicals

All chemicals should be of Reagent grade, ACS grade or better (*). The use of pro analysis chemicals is recommended. Poor quality of the reagents can affect the analyser performance.

(*) Analytical Reagent (AR), Guaranteed Reagent (GR), UNIVAR, AnalaR, Premium Reagent (PR), ReagentCertified ACS reagent, ACS Plus reagent, puriss p.a. ACS reagent, ReagentPlus®, TraceCERT®, Suprapur®, Ultrapur®, or better are also possible.

Quality of DI-water

All EZ analysers are tested with standard solutions, reagents and dilution water prepared using type I water or better as defined by ASTM D1193-91.

To achieve the specifications as stated on the data sheet, method and reagents sheet and acceptance test reports, the same water quality (or better) must be used for the preparation of the standard solutions, reagents and dilution water.

Additionally the water used for the preparation of the standard solutions for an EZ analyser must be free of the parameter or any of the interferences for the method of that EZ analyser.

Storage of Reagents

While operating the instrument, keep in mind the ambient temperature conditions as stated in the data sheet of the instrument.

⚠ CAUTION	
	Store the reagents cold; Store the reagents in the dark; If applicable: Store the reagents in a fridge during operation

⚠ CAUTION	
	Refresh the reagents after one month (unless stated differently in the chapters below). Do not mix old reagents with freshly prepared reagents. Remove old reagents from the container before adding freshly prepared reagents.

4.4 Sulfuric acid solution (0.2M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	11.16 mL

Preparation

Dilute 11.16 mL of sulfuric acid (H_2SO_4, 96 %) in 500 mL of de-ionized water using a volumetric flask of 1000 mL. Mix and add de-ionized water.

4.5 Sodium hydroxide solution (0.2M)

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	8 g

Preparation

Dissolve 8 g of sodium hydroxide (NaOH) in 500 mL of de-ionized water using a volumetric flask of 1000 mL. Mix and add de-ionized water up to the grade mark.

4.6 Anti-Foam (facultative)

Products	Formula	MW (g/mol)	CAS No.
1-octanol	$C_8H_{18}O$	130.23	111-87-5

4.7 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium acetate	$C_2H_3NaO_2$	82.03	127-09-3	13.67 g

Preparation

10000 mg/L VFA stock solution

Prepare a stock solution of 10000 mg/L VFA: Dissolve accurately 13.67 g sodium acetate ($C_2H_3NaO_2$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

5000 mg/L VFA standard solution – REF2

Prepare a standard solution of 5000 mg/L VFA. Take accurately 500 mL of the 10000 mg/L VFA stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

4.8 Validation solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium acetate	$C_2H_3NaO_2$	82.03	127-09-3	13.67 g
Sodium bicarbonate	$NaHCO_3$	84.01	144-55-8	84.04 g

Preparation

10000 mg/L VFA stock solution

Prepare a stock solution of 10000 mg/L VFA: Dissolve accurately 13.67 g sodium acetate ($C_2H_3NaO_2$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

50000 mg/L TA stock solution

Prepare a stock solution of 50000 mg/L $CaCO_3$ TA: Dissolve accurately 84.04 g sodium bicarbonate ($NaHCO_3$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

Remark

The VFA concentration has an influence on the TA determination. Therefore, both standards cannot be mixed. It is recommended to use a separate VFA and TA standard solution to check the functionality of the analyser.

2500 mg/L VFA validation solution – Validation

Prepare a standard solution of 2500 mg/L VFA. Take accurately 250 mL of the 10000 mg/L VFA stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

5000 mg/L TA validation solution – Validation

Prepare a standard solution of 5000 mg/L TA. Take accurately 100 mL of the 50000 mg/L $CaCO_3$ TA stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

4.9 Cleaning solution (facultative)

The cleaning procedure should prevent any build-up of chemicals in the analyser. To obtain an effective cleaning procedure one has to test the cleaning solution and the cleaning interval for each application. Perform the selected cleaning solution and interval for a trial period, check then the effectiveness of the procedure and change if necessary.

Change Information	
Date: 08/10/2021	Previous version: Edition 1.01 to Edition 1.10

Reason for Change

- Addition of standard measurement cycle time
- Change in method description
- Addition of information reagents
- Change in concentration H_2SO_4
- Change in concentration NaOH

Description of Change

- Addition of standard measurement cycle time of 15 minutes (chapter 2)
- Change in description of determination Total Alkalinity (chapter 3.1)
- Addition of extra information regarding reagent consumption (chapter 4.1)
- Change in concentration H_2SO_4 solution from 0.1 M to 0.2 M (chapter 4.4)
- Change in concentration of NaOH solution from 0.1 M to 0.2 M (chapter 4.5)