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EZ2301 Total Chromium & Chromium (VI) Analyser

Method and reagent sheets

11/2021, Edition 1.01

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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.

Chromium - All specifications				
Analysis method	Colorimetric measurement at 546 nm using diphenylcarbazide method			
Parameter	Cr Total, Cr (VI)			
Cycle time	Standard measurement cycle time: 30 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.			
Limit of detection (LOD)	≤ 2 µg/L			
Precision/Repeatability	Better than 2% full scale range for standard test solutions			
Cleaning	Automatic; frequency freely programmable			
Calibration	Automatic, 2-point; frequency freely programmable			
Validation	Automatic; frequency freely programmable			
Interferences	Iron (III) [$(Fe)^{3+}$)], mercury [$(Hg)^{2+}$] > 200 mg/L, molybdenum [$(Mo)^{2+}$] > 200 mg/L, vanadium (V) [$(V)^{5+}$] > 5 mg/L. Large amounts of color and turbidity interfere. Fats, oil, proteins, surfactants and tar.			
Measuring ranges	**% of range - Dilution**		**Low range (µg/L)**	**High range (µg/L)**
	A	10% of standard range	2	50
	B	25% of standard range	4	125
	C	50% of standard range	5	250
	0	**standard range**	**10**	**500**
	1	internal MP dilution (factor 4)	160	2000
	3	internal MP dilution (factor 10)	320	5000

3. Analysis method

Summary

The determination of Chromium (VI) and total Chromium is based on two methods, combined in one analyser.

The Chromium (VI) concentration is determined in the ‘Cr VI’ method. The total Chromium concentration is determined in the ‘Total Cr’ method. The concentration of all parameters is determined alternately in the ‘Main’-method.

The calibration for Chromium (VI) is determined in the ‘Cr VI’ method. The calibration for total Chromium is determined in the ‘Total Cr’ method.

Remark

The methods cannot be started at the same time.

3.1 Chromium (VI)

Summary

The determination of the chromium concentration in water is based on the reaction of hexavalent chromium with 1,5-diphenycarbazide in an acidic medium to form an intense coloured red-violet complex. The absorption is measured at 546 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling, the buffer solution will be added and the initial absorbance value is measured at 546 nm. Next, colour solution is added and after respecting a stirring period – performed to obtain complete colour development –the final absorbance value is determined. With the obtained absorbance values, the chromium VI concentration can be calculated according to Beer's Law.

Calibration

The calibration procedure measures a REF1 Cr solution (channel 9, REF1 valve) and a REF2 Cr solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two-point calibration.

3.2 Total Chromium

Summary

The determination of the chromium concentration in water is based on the reaction of hexavalent chromium with 1,5-diphenycarbazide in an acidic medium to form an intense coloured red-violet complex. The absorption is measured at 546 nm. Prior to the total chromium analysis, the sample is digested by use of a persulfate and an acid solution.

Analysis steps

The sample is mixed with the persulfate solution and acid solution and heated to 120 °C (or up to 150 °C – programmable) in an oven during several minutes (standard 10 minutes; programmable up to 60 minutes). During the digestion process, chromium is converted to hexavalent chromium. After digestion, the sample is cooled and transferred into the analysis vessel. Buffer solution is added to the digested sample and respecting a stirring period, the initial absorbance value is measured. Next, the colour solution is added. The final absorbance value is determined. With the obtained absorbance values, the chromium concentration can be calculated according to Beer's law.

Calibration

The calibration procedure measures a REF1 Cr solution (channel 9, REF1 valve) and a REF2 Cr solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two-point calibration.

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 litre 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/30 min	Recommended containers
Acid solution (Total Cr)	~ 0.85 mL / analysis	~ 1.1 L	Plastic – 2.5 L
Persulfate solution (Total Cr)	~ 0.25 mL / analysis	~ 0.5 L	Plastic – 2.5 L
Buffer solution (Total Cr)	~ 0.85 mL / analysis	~ 2.3 L	Plastic – 2.5 L
Buffer solution (Cr)	~ 0.85 mL / analysis		
Colour solution (Total Cr)	~ 2.0 mL / analysis	~ 5.4 L	Glass-Amber – 2.5 L
Colour solution (Cr)	~ 2.0 mL / analysis		
REF1 solution (Total Cr + Cr)	~ 1 L / calibration	/	Plastic – 1 L
REF2 solution (Total Cr + Cr)	~ 1 L / calibration	/	Plastic – 1 L

4.2 DI-water overview and consumption

	Rinse water (mL/analysis) Type I		Dilution water (mL/analysis) Type I		Total (mL/analysis)	Consumption/28 days A rata 1 analysis/30 min
	Total Cr	Cr (VI)	Total Cr	Cr (VI)	Total Cr + Cr (VI)	Total Cr + Cr (VI)
A	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
B	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
C	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
0	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
1	55 mL	55 mL	20 mL	10 mL	140 mL	190 L
3	55 mL	55 mL	20 mL	10 mL	140 mL	190 L

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 reagent requirements as stated in the reagent overview, the chapters below and/or in the data sheet of the instrument.

⚠ CAUTION	
	For longer-term storage: 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 Acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	56 mL

Preparation

Dilute 56 mL sulfuric acid (H_2SO_4, 96%) in 500 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water.

4.5 Persulfate solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium persulfate	$Na_2S_2O_8$	238.09	7775-27-1	10 g

Preparation

Dissolve 10 g of sodium persulfate ($Na_2S_2O_8$) in 500 mL de-ionized water. Mix and fill up to 1 litre with de-ionized water.

4.6 Buffer solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	56 mL

Preparation

Dilute 56 mL sulfuric acid (H_2SO_4, 96%) in 500 mL de-ionized water. Mix and Fill up to 1 litre with de-ionized water.

4.7 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
1,5-Diphenylcarbazide	$C_{13}H_{14}N_4O$	242.28	140-22-7	1 g
Acetone (analytic)	C_3H_6O	58.08	67-64-1	500 mL
Acetic acid (glacial)	$C_2H_4O_2$	60.05	64-19-7	2 mL

Preparation

Dissolve 1 g of 1.5-Diphenylcarbazide ($C_{13}H_{14}N_4O$) completely in 500 mL acetone (C_3H_6O). Add carefully 2 mL acetic acid ($C_2H_4O_2$) and fill up to 1 litre with de-ionized water. The pH value of this solution should be below 4.

4.8 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium dichromate	$K_2Cr_2O_7$	294.18	7778-50-9	1.4143 g

Preparation

500 mg/L Cr stock solution

Prepare a stock solution of 500 mg/L Cr: Dissolve accurately 1.4143 g potassium dichromate ($K_2Cr_2O_7$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

Cr standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 500 mg/L Cr stock solution and transfer into a plastic volumetric flask of 1 litre. Add de-ionized water up to the mark grade.

	Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
A	50 µg/L Cr	50 µg/L Cr	0.10 mL
B	125 µg/L Cr	125 µg/L Cr	0.25 mL
C	250 µg/L Cr	250 µg/L Cr	0.50 mL
0	**500 µg/L Cr**	**500 µg/L Cr**	**1.0 mL**
1	2000 µg/L Cr	2000 µg/L Cr	4.0 mL
3	5000 µg/L Cr	5000 µg/L Cr	10.0 mL

Cr standard solution – REF1

Prepare a standard solution of 0 µg/L Cr. Use de-ionized water.

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: 10/11/2021	Previous version: Edition 8 to Edition 1.01

Reason for Change

- Addition of water consumption
- Addition of information reagents

Description of Change

- Addition of estimated consumption of water for rinse and dilution (chapter 4.2)
- Addition of extra information regarding storage and quality of reagents (chapter 4.3)