Supelco®
Analytical Products

Application Note

Iodine in drinking water, wastewater and disinfection solutions

Photometric determination using DPD (dipropyl-p-phenylenediamine)

Introduction

Iodine has antiseptic properties and is used for a variety of antimicrobial uses, like emergency drinking water purification, fresh food sanitization, hospital surface disinfection or disinfection of cooling tower water [1]. Iodine determination in aqueous samples is performed mainly to ensure effectiveness of the disinfection and to prevent negative health impact on humans.

Typical concentrations for potable water treatment are 2.5 - 7 mg/l [2]. Iodine has become attractive also for particular applications like drinking water disinfection aboard space vessels at a residual concentration of approximately 2 ppm [3].

According to 40 CFR Section 185.940 the iodine tolerance exemption in antimicrobial formulations for food-contact surface sanitizing solutions is defined as "the total end-use concentration of all iodide-producing chemicals in the solutions is not to exceed 25 ppm of titratable iodine" [4].

This Application Note describes the method that was former available as Spectroquant® Iodine Test (1.00606) which is now discontinued. The method is furthermore available as preprogrammed method in the Spectroquant® photometers and colorimeters Prove 100/300/600, Nova 60 and Move 100 and can be performed with the use of the reagent of Spectroquant® Chlorine Test (1.00598).

The reagent Cl_2-1 of Cat. No. 1.00598 Spectroquant® Chlorine Test has the identical reagent composition as the reagent I_2-1 of the discontinued Cat. No. 1.00606 Spectroquant® Iodine Test.

The method with a measuring range of 0.050 – 10.00 mg/l I_2 is suitable to determine the iodine concentration in drinking water, wastewater and disinfection solutions.

Experimental

Method

In weakly acidic solution free iodine reacts with dipropyl-p-phenylenediamine (DPD) to form a red-violet dye that is determined photometrically.

Measuring Range and Method number

Method No.	Measuring Range	Cell	Instruments
147	0.050 – 2.000 mg/l I_2	50-mm rectangular cell	Prove 100/300/600 (plus), Nova 60A
147	0.10 – 5.00 mg/l I_2	20-mm rectangular cell	Prove 100/300/600 (plus), Nova 60A
147	0.20 – 10.00 mg/l I_2	10-mm rectangular cell	Prove 100/300/600 (plus), Nova 60A
240	0.10 – 5.00 mg/l I_2	24-mm round cell	Move 100

Sample Material

Drinking water, wastewater, disinfection solutions.

Influence of foreign substances

Other components in the sample - than the target analyte I_2 – may interfere with the detection chemistry described here. Some of them were checked in solutions containing 3.5 and 0 mg/l I_2. The determination is not yet interfered with up to the concentrations of foreign substances given in the table. Cumulative effects were not checked; such effects can, however, not be excluded.

Concentrations of foreign substances in mg/l or %					
Al^{3+}	250	Mn^{2+}	100	Cl_2	**0.05**
Ca^{2+}	1000	**NO_2^-**	**0.1**	**ClO_2**	**0.1**
CN^-	**0.1**	**S^{2-}**	**0.1**	**I_2**	**0.15**
CO_3^{2-}	1000			**H_2O_2**	**0.05**
Cr^{3+}	250			**O_3**	**0.01**
$Cr_2O_7^{2-}$	0.1			NaCl	10%
Cu^{2+}	100			$NaNO_3$	10%
Fe^{3+}	100			**Na_2SO_4**	10%

Reagents, Instruments and Materials

Cat.No.	Product Description
Reagents	
1.00598	Spectroquant® Chlorine Test
Instruments	
For iodine measurement one of the following Spectroquant® photometers is necessary	
1.73026	Spectroquant® VIS Spectrophotometer Prove 100 plus
1.73027	Spectroquant® UV/VIS Spectrophotometer Prove 300 plus
1.73028	Spectroquant® UV/VIS Spectrophotometer Prove 600 plus
1.09752	Spectroquant® Photometer Nova 60A
1.73632	Spectroquant® Colorimeter Move 100
Software for data maintenance *(Prove instruments only)*	
The Spectroquant® Prove Connect to LIMS software package provides an easy way to transfer your data into an existing LIMS system. This software can be purchased under	
Y11086	Prove Connect to LIMS
Materials	
1.14946	Rectangular cell 10 mm
1.14947	Rectangular cell 20 mm
1.14944	Rectangular cell 50 mm
1.73650	Empty cells 24 mm with screw caps (12 pcs)
	Pipettes for a pipetting volume of 10.0 ml
Other reagents and accessories	
1.09533	MQuant® pH-indicator strips pH 5.0 - 10.0
1.09531	MQuant® pH-indicator strips pH 0 - 6.0
1.09137	Sodium hydroxide solution 1 mol/l Titripur®
1.09072	Sulfuric acid 0.5 mol/l Titripur®

Also first generation Prove instruments are compatible and preprogrammed with this method.

Analytical approach

Preparation

- Analyze immediately after sampling!
- The pH must be within the range 4 - 8. Adjust, if necessary, with sodium hydroxide solution or sulfuric acid.
- Filter turbid samples.

Preparing the measurement solution

Pipette 10 ml of pretreated sample (5 - 40 °C) into a test tube.

Add 1 level blue microspoon (in the cap of the reagent bottle) of Reagent Cl_2-1 from Cat. No. 1.00598 Spectroquant® Chlorine Test and shake vigorously until the reagent is completely dissolved. Reclose the reagent bottle immediately after use.

Leave to stand for 1 min (reaction time), then fill the sample into the cell, and measure in the photometer.

Measurement

It is recommended to perform a zero adjustment for this method each new working day. Details regarding the zero adjustment can be found in the user manual of your instrument.

It is recommended to use the same cell for zero adjustment and for sample measurement. For zero adjustment fill the cell with distilled water (or water for analysis) and follow the instructions in the user manual of your instrument.

Prove and Nova instruments

Select method no. 147 from the method list.

Fill the pretreated sample into a 10-mm, 20-mm or 50-mm rectangular cell and place the cell into the cell compartment, the measurement is performed automatically.

The Iodine content in mg/l I_2 appears in the display.

Move 100

Select method no. 240 from the method list.

Fill approx. 10 ml of distilled water into a 24-mm cell, close with the screw cap **(Blank cell)**. Insert the blank cell into the cell compartment. Align the mark on the cell with that on the photometer. Press “Zero”.

Fill the prepared measurement solution into a 24-mm cell, close with the screw cap **(Sample cell)**. Insert the sample cell into the cell compartment. Align the mark on the cell with that on the photometer. Press “Test”.

The Iodine content in mg/l I_2 appears in the display.

Notes on the measurement

- When using the 50-mm cell it is recommended to measure against an own prepared blank sample (preparation as per measurement sample, but with distilled water instead of sample) to increase the accuracy. Configure the photometer for blank measurement (Prove and Nova instruments only).
- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 4.5 - 5.5.
- The color of the measurement solution remains stable for 30 min after the end of the reaction time stated above.
- In the event of iodine concentrations exceeding 25 mg/l, other reaction products are formed and false-low readings are yielded. In such cases it is advisable to conduct a plausibility check of the measurement results by diluting the sample (1:10, 1:100).

Analytical quality assurance

Recommended before each measurement series.

To check the photometric measurement system (test reagent, measurement device, handling) and the mode of working, a freshly prepared iodine standard solution containing 5.00 mg/l I_2 (application see the website) can be used. Sample-dependent interferences (matrix effects) can be determined by means of standard addition.

References

1. World Health Organization: Alternative drinking-water disinfectants: bromine, iodine and silver; ISBN 978-92-4-151369-2 or URL: https://apps.who.int/iris/bitstream/handle/10665/260545/9789241513692-eng.pdf (2021-05-26)
2. Hitchens AP (1922). The emergency treatment of water for drinking purposes. J Mil Surg. 51:657–63.
3. Atwater JE, Sauer RL, Schultz JR (1996). Numerical simulation of iodine speciation in relation to water disinfection aboard manned spacecraft I. Equilibria. J Environ Sci Health. A3:1965–79.
4. 40 CFR Section 185.940, URL: https://ecfr.io/Title-40/Section-180.940 (2021-05-26)

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