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Development of an Universal Measuring Instrument for Quality Monitoring of Ultrapure Water

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Summary:
This work presents the development of a measuring system for the quality control of ultrapure water. The new systems combines ozonation and UV radiation for the oxidation of organic substances. The change in conductivity caused by the oxidation is furthermore correlated with the TOC of the solution.

Keywords: TOC, ultrapure water, ozone, ozonation, AOP

Introduction

The quality of ultrapure water is of particular importance in the pharmaceutical and semiconductor industry. Due to its unique properties it serves as a solvent or starting product for pharmaceuticals and is also used to clean surfaces in the production of high-precision components in semiconductor manufacturing [1]. For this reason, water quality monitoring is of utmost importance and a universal measuring instrument is required.

One method for monitoring the quality of ultrapure water is the measurement of the total organic carbon (TOC) in the water as organic substances are the most common contaminants in water. TOC comprises all organic compounds present in water. For water used in the pharmaceutical industry not only the TOC content but also the temporal development of the TOC due to bacterial growth is a parameter of interest concerning the quality control [2]. Boundary limits and standards for the analysis of TOC are defined by the European Pharmacopeia or the German Institute for Standardization [3] [4].

Conventional methods of TOC determination are based either on thermal oxidation of carbonaceous substances or on wet chemical oxidation. In the latter case oxidation is carried out either by persulfate or UV radiation [5]. All these systems are based on the oxidation of the present organic carbon (OC) and the subsequent detection of the resulting CO_2.

The presented new developed system (**Fehler! Verweisquelle konnte nicht gefunden werden.**) combines two standard methods of oxidation. The oxidation is performed by ozonation in combination with UV radiation. In contrast to conventional devices in which Hg-vapor lamps are used for UV irradiation, SMD LEDs are used. This enables to reduce the size of the construction considerably. The combination of ozonation and UV-radiation leads to an advanced oxidation process and a significant increase in the oxidation power of the entire system. The system offers three main advantages: it does not require the addition of chemicals - ozone is generated in situ and decomposes without leaving residues and the new system is of significantly smaller footprint and can therefore be easily used at various locations. In addition, it enables online monitoring of the TOC as conductivity is permanently measured.

Experimental

The core of the setup is a 200 mm long quartz glass tube with an inner diameter of 4 mm. This tube serves as reaction chamber. It is wedged between two PTFE blocks. Ozone is generated in situ in water with a so-called “ozone microcell (OMC)”, developed by the company Innovatec Gerätetechnik GmbH [5].

Water from an ultrapure water system passes the ozone generator into the quartz glass tube. For irradiation, five SMD LEDs from Neumüller [6] with a wavelength of 275 nm are used. The first conductivity measuring point (C_1) is placed in front of the entrance of the glass tube. After the irradiation section, the gas bubbles are separated from the water by a PTFE membrane. Thereafter, the conductivity is measured a second time (C_2). Furthermore, the water temperature is measured with a Pt1000 sensor.

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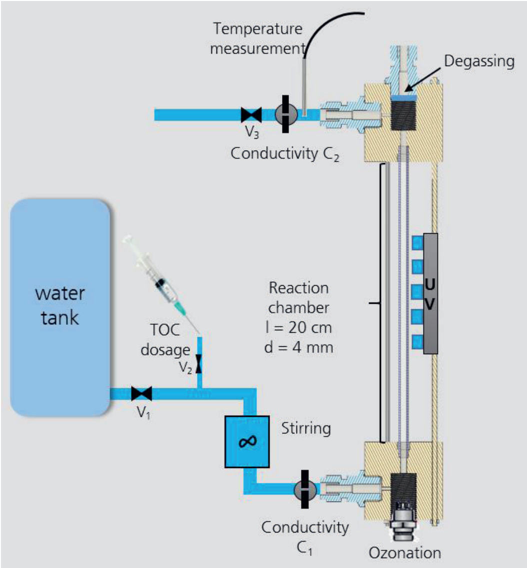

Figure 1: Schematic illustration of the sensor design

The measuring principle is a conductance measurement while the TOC of the solution is oxidized. For this purpose, a defined quantity of OC is permanently added with a syringe pump to the ultrapure water flow. Variation in volume flow of the syringe pump leads to a variation in TOC concentration.

In all experiments 2-propanol serves as source of organic carbon and a constant ozone cell current is applied.

In a five-time repetition, the solution is oxidized with an optical power of 32 mW. In each repetition, LEDs are switched on for two minutes followed by three minutes without UV irradiation. The conductivity and temperature of the water are permanently recorded.

Results

The change in conductivity caused by the oxidation of the TOC solution gives information about the TOC concentration in water. Concentration-dependent measurements are used to correlate the TOC with the change in conductivity as shown in the following figure.

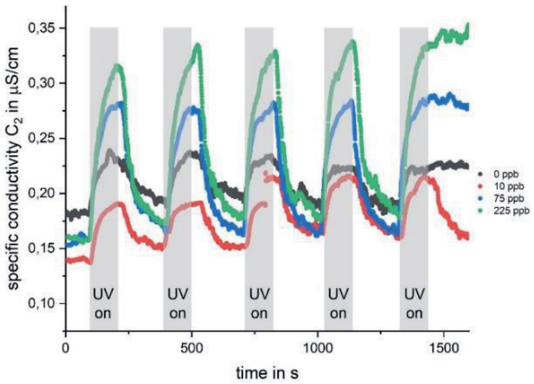

Figure 2: Overlay of four measurements of differently concentrated TOC-solutions, five repetitions

The figure shows an overlay of the results of the measurement of four differently concentrated solutions (0 ppb, 10 ppb, 75 ppb and 225 ppb). The irradiation periods are marked by grey bars (see Figure 2 "UV on).

It is clearly apparent from that figure that conductivity increases with increasing initial OC concentration. This becomes even clearer by comparing the first 100 seconds of the increase in conductivity, which is illustrated in Figure 3.

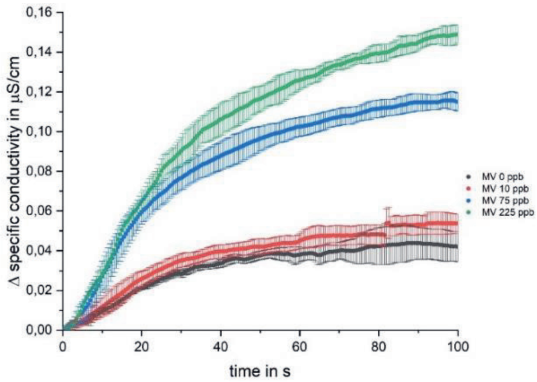

Figure 3: Mean values and standard deviations of normalized TOC measurements while radiation

The mean increase in conductivity is plotted as a function of time for the four different measurements. The irradiation of the ozonated water leads to the formation of highly reactive hydroxyl radicals which furthermore react to form acidic oxidation products. The more OC is added, the higher the increase in conductivity. Even the solution without OC addition shows an increase during oxidation, which is attributed to a residual amount of impurities in the water.

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