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Type II-glass for pharmaceutical industry – Delamination according to USP 1660

Glass as a packaging material is the first choice due to its special properties. It is chemical inert, can be thermally treated without any problems and protects the medium stored inside from contamination. In general, glass containers are divided into type I, type II, and type III due to alkali release and, thus, hydrolytic resistance. Since medicines and vaccines are usually stored in type I, the worldwide COVID-19-pandemic has led to a significant demand for type I glass vials. This has also increased the need for alternative and more cost-effective soda-lime glasses with high hydrolytical resistance in the pharmaceutical industry. This type II-glass is created using a so-called inner coating, which significantly decreases the migration at the glass surface towards alkaline, acidic, and aqueous solutions. Therefore, these type II-glasses are the best choice for most parentals.

Common corrosion tests of inner glass surfaces are carried out using water as medium and evaluated regarding the leaching of alkalines. It is particularly interesting for the pharmaceutical industry to learn how the media attacks the glasses they are stored in. Using alternative test media allows to test the homogeneity of the coatings of type II-bottles and to prove whether the bottles belong to type II or type III.

Abb. 1: Type III-bottles with corrosion visualized via methyl blue

For this, different test media are used to prove the delamination of inner surfaces according to USP 1660. This allows among other things to evaluate the ablation rate at inner surfaces via silicon analysis in the range of ppm using ICP-OES. The aggressive effect of media can also be visualized by the use of methyl blue, which adheres to corroded surfaces (Abb. 1).

*Die Akkreditierung gilt für den in der Urkundenunterlage D-PL- 20043-01-00 festgelegten Umfang

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Even the UPS 1660 itself states that the media described there are too aggressive for delamination analysis and attack the glass significantly more than the filling material. Therefore, the IGR has developed a more practicable solution to carry out the USP 1660 in a lab, using further standards as DIN ISO 695 and ISO 7086 as well as various analyses of type II- and type III-bottles.

The first tests were carried out by using demineralized water, a potassium chloride solution and a mixed solution of potassium chloride and citrate, whereby the applied solutions were brought into an alcaline pH range by addition of potassium hydroxide. The used bottles were treated in the autoclave according to DIN ISO 4802, then filtered and the solutions were analyzed using ICP-OES. The filtration is done to detect silicon-flakes, which might come out of the bottles during analysis.

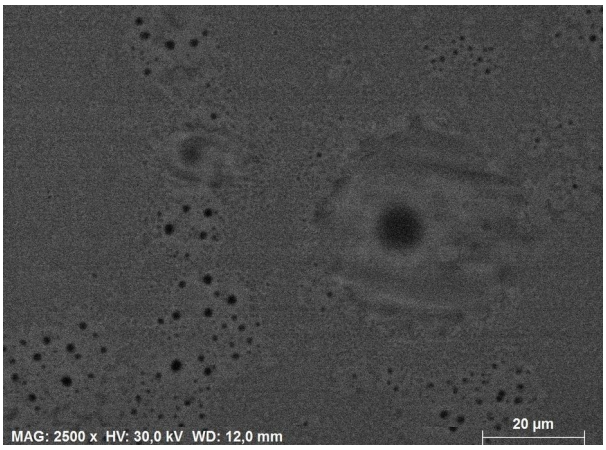

Abb. 2: SEM-image of the damaged surface of a type II-bottle

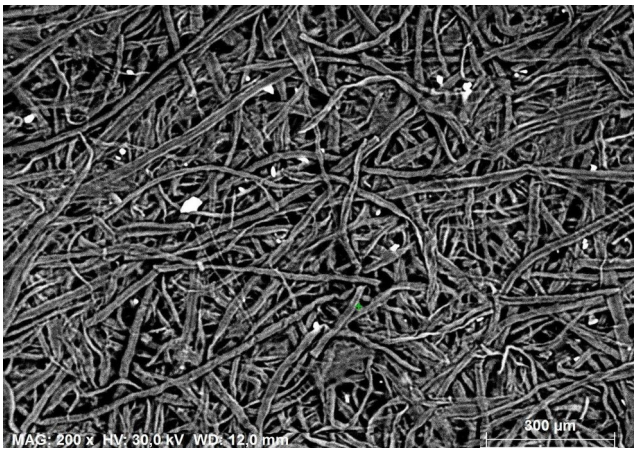

Abb. 3: Silicon-flakes in a SEM-image in a filter of a type II-bottle

Significant damages at the glass surface as well as silicon-flakes in the mixed solution of potassium chloride and citrate were detected using SEM-EDX (Abb. 2, Abb. 3). The bottles, which were filled with demineralized water and potassium chloride, showed significantly less damaged surfaces, which was supported visually due to the methyl blue (Abb. 4). The corrosion could also be verified microscopically in the coloured bottles (Abb. 5).

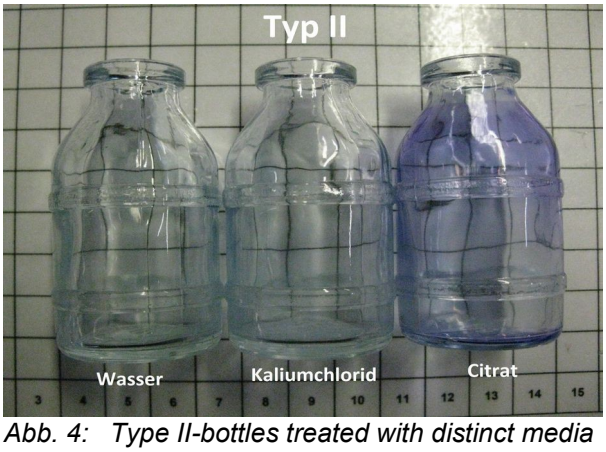

Abb. 4: Type II-bottles treated with distinct media with corrosion visualized by methyl blue

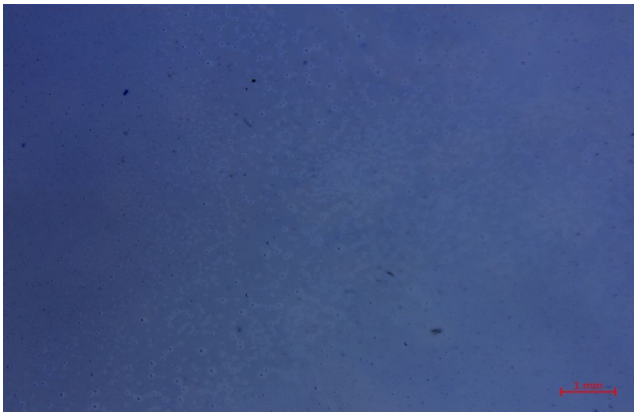

Abb. 5: Microscopic image of a type II-bottles treated with citrate and coloured with methyl blue

This shows, that the used media massively influence the results of the analysis depending on their aggressivity and concentration. The pH-value of the used test media also has a massive influence on the test results, so vary the analytical results

noticeably, when the pH-value is set incorrectly. However, the most stable results were obtained when the samples, which were filled with the most aggressive test medium, were not treated in the autoclave as described above in accordance with DIN ISO 4802, but stored warm for a defined period of time and at a defined temperature. In an analysis according to DIN ISO 4802, the silicon values can be compared to the sodium values in a ratio of 1:4, when using vessels with a filling volume of 10 ml to 100 ml.

In summary, the investigations indicate, that the analyses may not be carried out with the concentrations given in USP 1660. Additionally, the used media, concentrations, temperatures, and times must be adapted to the bottles to be tested.

Taking into account the glass products, media and analytical conditions, the IGR routinely offers now a practicable solution for the execution of USP 1660 in the lab for the glas producing and glas processing industry.