



Application Note AN-T-230

通过碘量氧化还原滴定法定量 1,2-二醇

Oxidative cleavage used as a pre-titration sample treatment to analyze mannitol, glycerin, and much more

Mannitol content determination is an important aspect of quality control in the pharmaceutical and food industries. In the pharmaceutical industry, mannitol is used as an excipient in the production of tablets, capsules, and other dosage forms. It is also used as a sweetener in food products. Selective oxidative cleavage can be used to quantify the amount of 1,2-diol groups in the analyte. Molecules like mannitol or glycerin are oxidized at ambient conditions. While oxidizing one equivalent of a diol, the

periodate ion gets reduced to iodate. The iodine atom gets reduced from an oxidation state of +7 to +5, and in the presence of iodide ions and acid, iodine is formed from periodate and iodate. The formed iodine can be titrated with thiosulfate solution. With automated reagent addition and an automated blank determination, reproducible results with a very low RSD can be achieved. By using digital electrodes, all used components are fully traceable and compliant.

SAMPLE AND BLANK PREPARATION

This application is demonstrated on mannitol, glycerin, and glycol.

After weighing the sample in an amber glass

beaker, periodate solution was added. The reaction mixture was then covered and stored in the dark for 30 minutes.

EXPERIMENTAL

The blank and the sample solutions were titrated after a 30-minute reaction time. Prior to titration, potassium iodide solution and sulfuric acid were added automatically. Using the instrument setup shown in **Figure 1**, the sample

was then titrated with thiosulfate solution until after the first equivalence point with the dPt Titrode. The entire process can be automated to minimize handling errors.

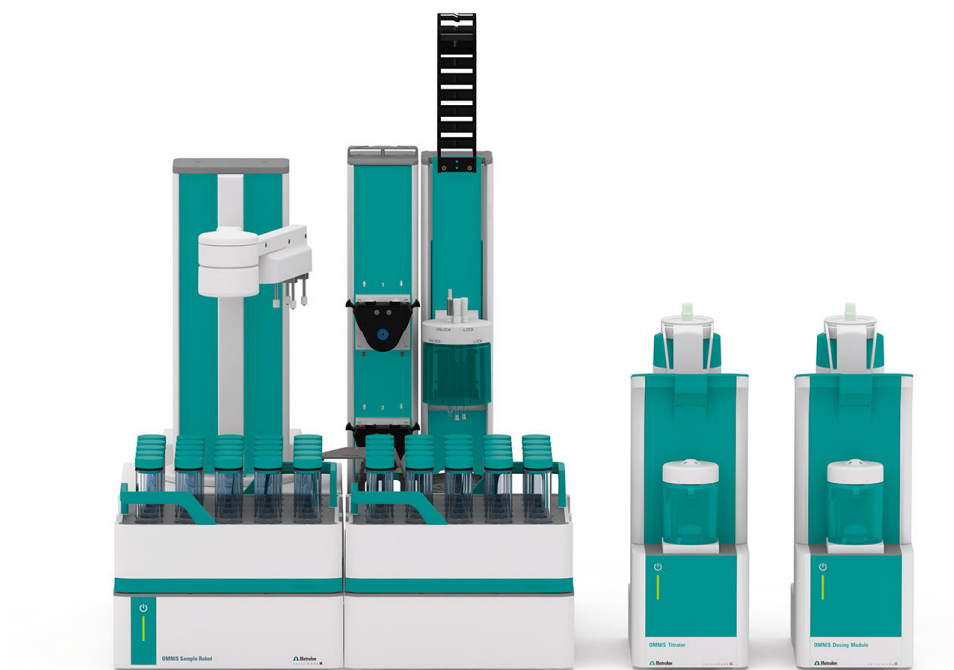


Figure 1. OMNIS Titrator with an OMNIS Dosing Module and an OMNIS sample robot S.

RESULTS

The determination of various diol-containing molecules (**Table 1**) gave some very accurate results with a meaningful recovery/weight

percentage in the sample. An example determination is shown in **Figure 2**.

Table 1. Results of the diol determination by oxidative cleavage.

Sample	Result wt%	RSD in %
Mannitol	101.62	0.36
Glycerin	101.99	2.14
Glycol	100.72	0.17

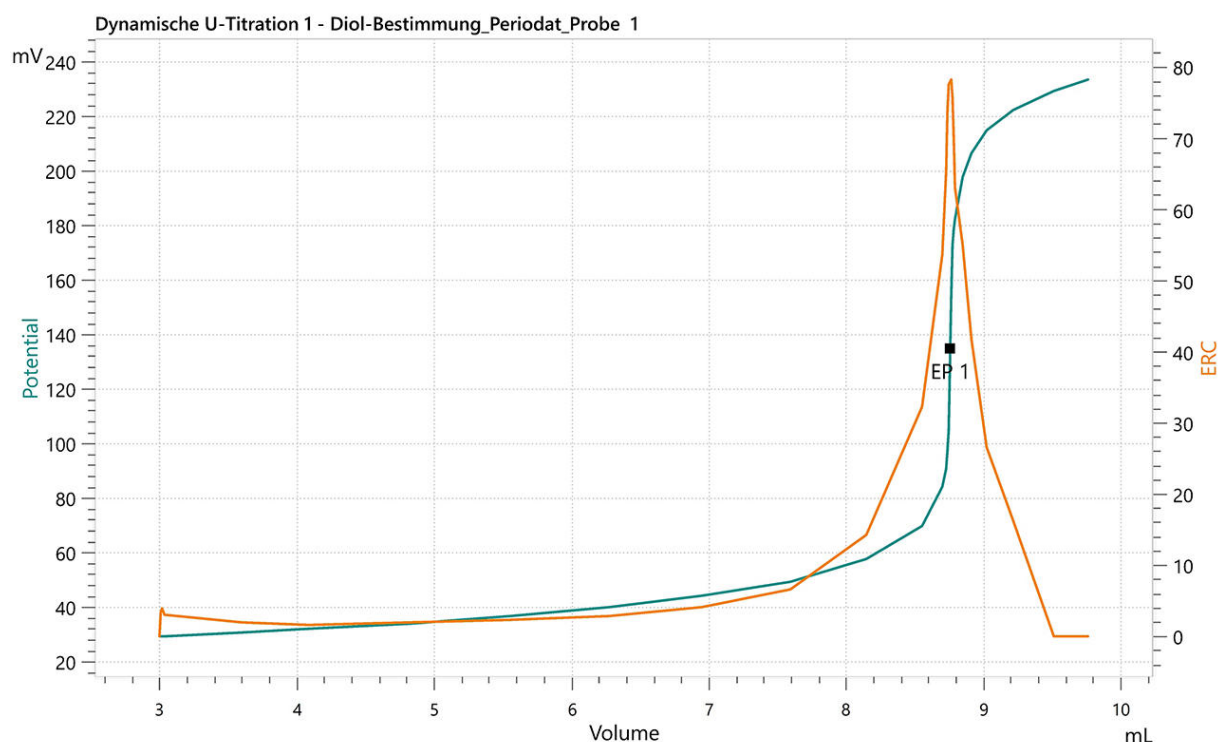


Figure 2. Example curve of the determination of diols in mannitol.

CONCLUSION

Automated titrators from Metrohm can be very helpful for determining the 1,2-diol content by iodometric titration. These titrators are designed to automate the titration process and provide accurate and precise results. They also have the ability to perform multiple titrations in a short period of time and to control and monitor the entire titration process, including the addition of

reagents, measurements, and endpoint detection.

Overall, the use of automated titrators from Metrohm can improve the efficiency and accuracy of the determination of 1,2-diols by iodometric titration and can be a useful tool for quality control in the pharmaceutical and food industries.

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CONFIGURATION



OMNIS Sample Robot S Pick and Place

OMNIS Sample Robot S 具有一个“蠕动”泵模块(2通道)和一个 Pick&Place 模块以及大量附件,可直接进入全自动滴定。此系统具有两个样品盘位置,可用于 32 个 120 mL 的样品盘。此模块化系统供货时已完全安装完毕,因此可在极短时间内投入运行。

该系统也可根据需要扩展配备另外两台蠕动泵以及多加一个 Pick&Place 模块,由此使通量翻倍。如果需要更多工作台,则可将此 Sample Robot 扩展为 L 规格款型的 OMNIS Sample Robot,由此可使七个样品盘的样品在多达四个 Pick&Place 模块上并行处理,将样品通量扩大四倍。