



Application Note AN-T-230

Quantification of 1,2-diols by iodometric redox titration

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 with a "Peristaltic" (2-channel) pump module and a Pick&Place module in addition to extensive accessories for the direct transition to fully automatic titration. The system provides space in two sample racks for 32 sample beakers of 120 mL each. This modular system is supplied completely installed and can thus be put into operation in a very short time.

The system can also be extended upon request to include two additional peristaltic pumps and another Pick&Place module, thus doubling the throughput. If additional workstations are required, then this Sample Robot is already able to be expanded to become an L-sized OMNIS Sample Robot, thus enabling samples from seven racks to be processed in parallel on up to four Pick&Place modules and quadrupling the sample throughput.