



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.

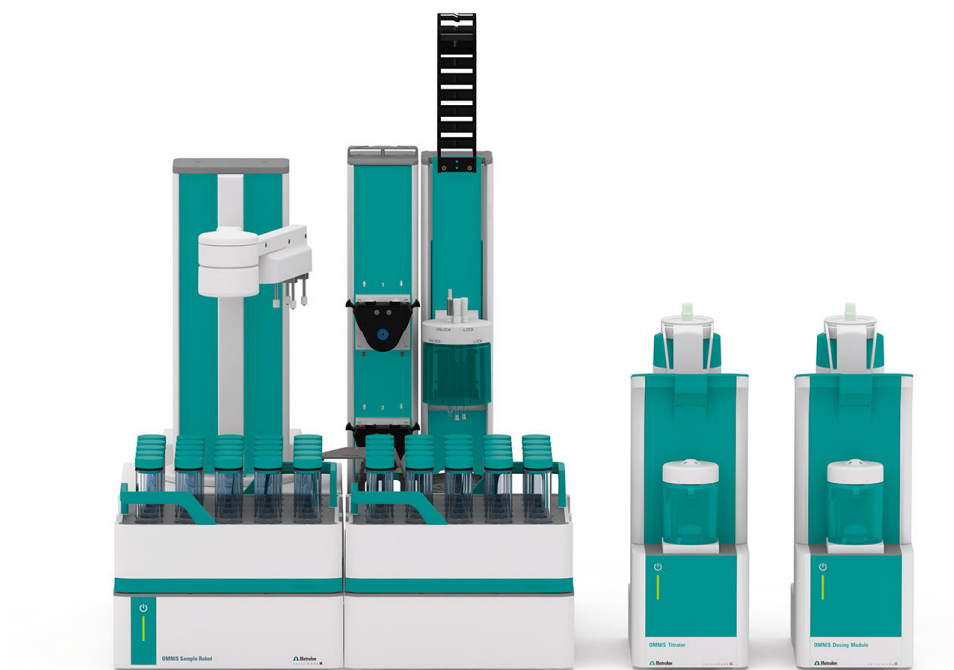


Figure 1. OMNIS Titrator with an OMNIS Dosing Module and an OMNIS sample robot S.

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.

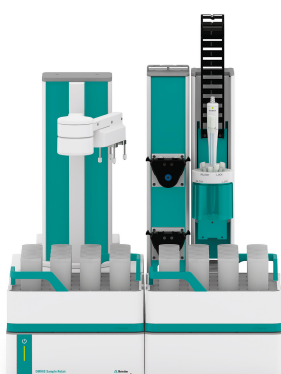
Overall, the use of automated titrators from Metrohm can improve the efficiency and accuracy of the determination of 1,2-diols by iodometric titration and can be a useful tool for quality control in the pharmaceutical and food industries.

CONTACT

メトロームジャパン株式会
社
143-0006 東京都大田区平
和島6-1-1
null 東京流通センター アネ
ックス9階

metrohm.jp@metrohm.jp

CONFIGURATION



OMNIS Sample Robot S Pick and Place

ポンプモジュール「Peristaltik」(2 チャンネル)、Pick&Place モジュール、および全自動滴定を直に導入するための多種の付属品のついた OMNIS Sample Robot S。このシステムでは 2 つのサンプルラックにおいて、120 mL のサンプルビーカー 32 個分のスペースが設けられており、モジュール式のシステムは、完全に組み立てられた状態で納品され、よって最短時間で稼働させることができます。

ご要望に応じて、システムには更に 2 つのペリスタリックポンプおよび追加の Pick&Place モジュールを装備することができ、スループットを倍増することができます。更なるワークステーションが必要な場合、この Sample Robot は L サイズまでの OMNIS Sample Robot に拡張することで最高 4 つまでの Pick&Place モジュールにおいて 7 つのラックのサンプルを並行して処理し、スループットを 4 倍に増やすことができます。