Application Note AN-T-178

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Pyridine-free, fully automated determination according to ASTM E1899, EN 15168, and DIN 53240-3

The hydroxyl number (HN) is an important sum parameter for quantifying the presence of hydroxyl groups. As a key quality parameter, it is regularly determined in resins, paints, polyesterols, fats, and solvents. Unlike other standards, ASTM E1899 is free from pyridine and does not require refluxing samples at high temperatures for extended periods. It is performed at room temperature, requires only a small sample size, is applicable to extremely low HN, and can be performed fully automatically.

This Application Note describes the potentiometric determination of HN in 1-octanol and polyethylene glycol according to ASTM E1899, EN 15168, and DIN 53240-3. Using the OMNIS Dis-Cover technique, all sample preparation steps can be fully automated. Moreover, the use of an OMNIS Sample Robot allows parallel analysis of multiple samples, reducing the time per analysis for one sample from approximately 24 min to 12 min, and increasing productivity in the laboratory considerably.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on 1-octanol (theoretical HN of 430.08 mg KOH/g) and polyethylene glycol (PEG) 3000.
No sample preparation is required.

EXPERIMENTAL

The determinations are performed on an automated system consisting of an OMNIS Sample Robot S equipped with Dis-cover, an OMNIS Professional Titrator equipped with two dSolvotrodes, and multiple OMNIS Dosing Modules for the addition of the auxiliary solutions.
An appropriate amount of sample is weighed into the titration beaker, acetonitrile is added, and the beaker is capped with the Dis-Cover lid. After dissolution of the sample, TSI solution is added, the beaker is covered and solution is stirred for the stipulated time. Then deionized water is added, and after stirring shortly, acetonitrile is added. The solution is titrated until after the second equivalence point with standardized tetrabutylammonium hydroxide in isopropanol.

Figure 2. Sample Robot S with Dis-cover, OMNIS Dosing Modules and OMNIS Titrator Professional equipped with two dSolvotrodes.

RESULTS

Well-defined titration curves are obtained for the tested samples. The result for 1-octanol is well within the acceptable limit for the standard with a low standard deviation. For PEG 3000, which is analyzed on both work stations in parallel, acceptable results with low standard deviations are obtained. Results are summarized in Table 1. An example titration curve is displayed in Figure 2.

Table 1. Results for the hydroxyl number determination according to ASTM E1899 on a fully automated OMNIS system equipped for the parallel analysis on two workstations.

Hydroxyl number (n = 6)	Mean in mg KOH/g sample	SD(rel) in %
1-Octanol	435.63	0.3
PEG 3000 (WS 1)	36.56	0.3
PEG 3000 (WS 2)	36.22	0.5

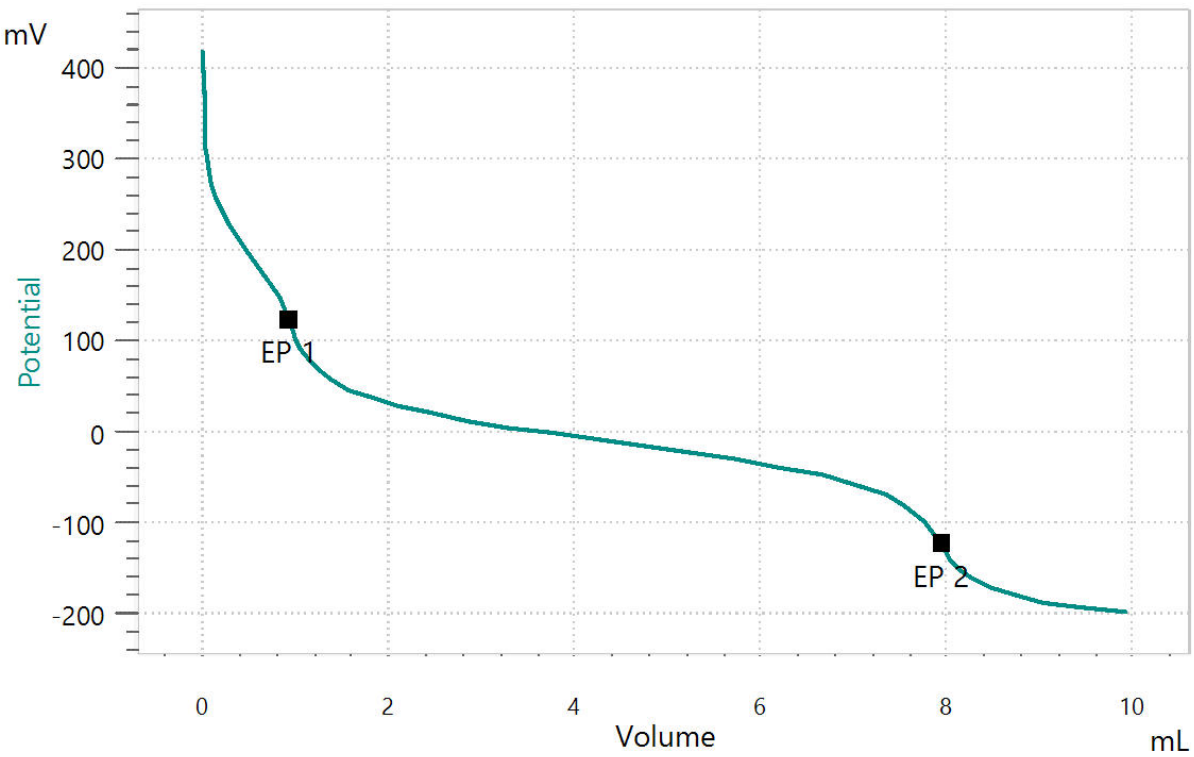

Figure 2. Titration curve of the determination of the hydroxyl number of 1-octanol.

CONCLUSION

Precise and reliable determination of the hydroxyl number according to **ASTM E1899**, **EN 15168**, and **DIN 53240-3** can be achieved using a fully automated OMNIS system. With the option to analyze up to four samples simultaneously, the productivity of a laboratory can be significantly improved. Furthermore, the OMNIS system can be customized according to your needs and expanded for other titration applications required for quality control.

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CONFIGURATION

OMNIS Professional Titrator

创新型、模块式电位分析 OMNIS Titrator,用于进行终点和等当点滴定(单一/动态)。由于采用 3S OMNIS Liquid Adapter 技术,处理化学品从未像现在一样安全。可以使用测量模块和计量管单元自由配置滴定仪,并在需要时扩展一台搅拌器。包括用于使用其他滴定或加液模块平行滴定的“Professional”功能许可证。

- 通过计算机或本地网络控制
- 可以为其他应用或辅助溶液另外连接最多四个滴定模块或加液模块
- 可以扩展磁力搅拌器和/或棒式搅拌器
- 可提供不同大小的计量管:5、10、20 或 50 mL
- 采用 3S 技术的 OMNIS Liquid Adapter:安全处理化学品,自动传输生产厂家的原始试剂数据

测量模式和软件选项:

- 终点设定滴定:“Basic” 功能许可证
- 终点和等当点滴定(单一/动态):“Advanced” 功能许可证
- 终点和等当点滴定(单一/动态),包括 5 路平行滴定:“Professional” 功能许可证

OMNIS Dosing Module

用于与 OMNIS Titrator 滴定仪相连的加液模块,以扩展额外用于滴定/加液的滴定管。可以扩展磁力搅拌器和/或螺旋搅拌器,以作为单独的滴定台使用。可自由选择 5、10、20 或 50 mL 计量管单元。

Pick and Place S

用于构建 S 规格款型 OMNIS Sample Robot Pick&Place 的主模块。该模块包括模块支架和样品盘架。已配备了主升降台和夹爪。若要扩展为功能就绪的 Sample Robot 则除了样品盘和抓取夹指之外还需要工作台,例如 Pick&Place 取放模块或泵模块。这些部件的选择可通过应用程序来完成。

Pick and Place

用于安装到 OMNIS Sample Robots Pick&Place 模块支架中的模块。该工作台可置放分析用的样品杯。内置的磁力搅拌器可将在外部磁力搅拌装置上准备好的样品直接在 Sample Robot 上进行分析,无须事先取下磁力搅拌棒。两次分析之间会将所使用的传感器在 Pick&Place 模块存储杯中进行清洁或置放。

4

用于安装到 OMNIS Sample Robots Pick&Place 模块支架中的模块。此工作站配备一台冲洗和抽液泵。可用于清洁 Pick&Place 模块中的传感器并在分析完成之后、重新将样品杯放回样品盘之前将其清空。

dSolvotrode

用于所有非水性酸/碱滴定的 OMNIS 数字组合式 pH 电极。玻璃膜针对导电性差的溶液进行了优化,并且由于灵活的磨口隔膜,该电极也适用于非常脏的样品。

此电极可与非水参比电解质(氯化锂或四乙基溴化铵)一起使用。

储存在相应的参比电解质中。
dTrodes 可在 OMNIS Titratoren 上使用。