

Application Note AN-T-178

Hydroxyl number in polyethylene glycol

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

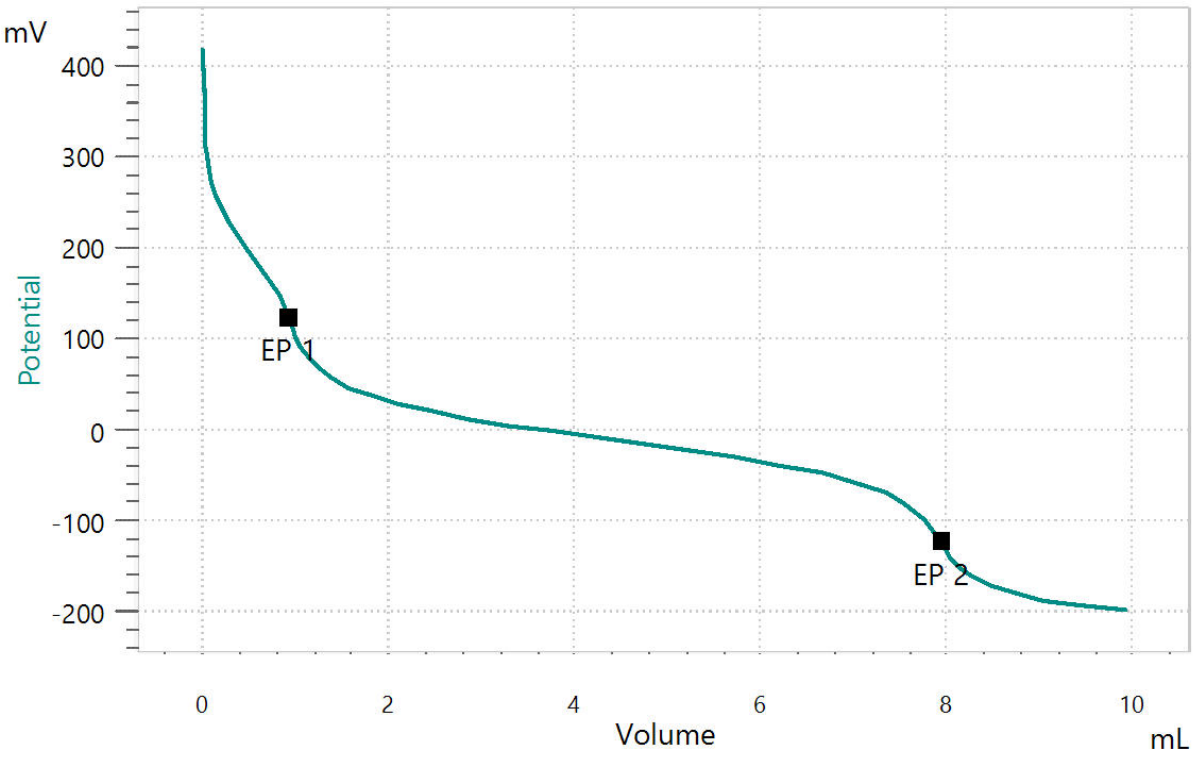


Figure 2. Titration curve of the determination of the hydroxyl number of 1-octanol.

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 without stirrer

Innovative, modular potentiometric OMNIS Titrator for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is safer than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a stirrer added as needed. Including "Professional" function license for parallel titration with additional titration or dosing modules.

- Actuation via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Can be supplemented with magnetic stirrer and/or rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Safe handling of chemicals, automatic transfer of the original reagent data from the manufacturer

Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with 5-way parallel titration: "Professional" function license



OMNIS Dosing Module without stirrer

Dosing module for connection to an OMNIS Titrator for extending the system to include an additional buret for titration/dosing. Can be supplemented with one magnetic stirrer or rod stirrer for use as separate titration stand. Freely selectable cylinder unit with 5, 10, 20 or 50 mL.



Main module Pick and Place S

Main module for setting up an OMNIS Sample Robot Pick&Place in size S. This module is comprised of module and rack plates. It is already equipped with the main lift and one gripper. In addition to sample racks and gripper fingers, workstations such as Pick&Place module or a pump module are required for using it to set up a functioning Sample Robot. The selection of these components proceeds in accordance with the application.



Pick and Place module with stirrer

Module for installation in the module plate of the Pick&Place of the OMNIS Sample Robot. This workstation takes up the sample beaker for the analysis. The built-in magnetic stirrer makes it possible to analyze samples that were prepared externally on a magnetic agitator directly in the Sample Robot without having to remove the magnetic stirring bar beforehand. Used sensors are cleaned or parked in the storage beaker of the Pick&Place module between analyses.



Peristaltic (4-channel) pump module

Module for installation in the module plate of the Pick&Place of the OMNIS Sample Robot. This workstation is equipped with two rinsing pumps and two aspiration pumps each. They are used to clean the sensors in two Pick&Place modules and to empty the sample beaker after the analysis before it is placed back in the rack once again.



dSolvotrode

Digital, combined pH electrode for OMNIS for all non-aqueous acid/base titrations. The glass membrane is optimized for poorly conducting solutions and thanks to the flexible ground-joint diaphragm, the electrode is suitable for contaminated samples.

This electrode can be used with non-aqueous reference electrolytes (lithium chloride or tetraethylammonium bromide).

Storage in corresponding reference electrolyte.
dTrodes can be used on OMNIS Titrators.