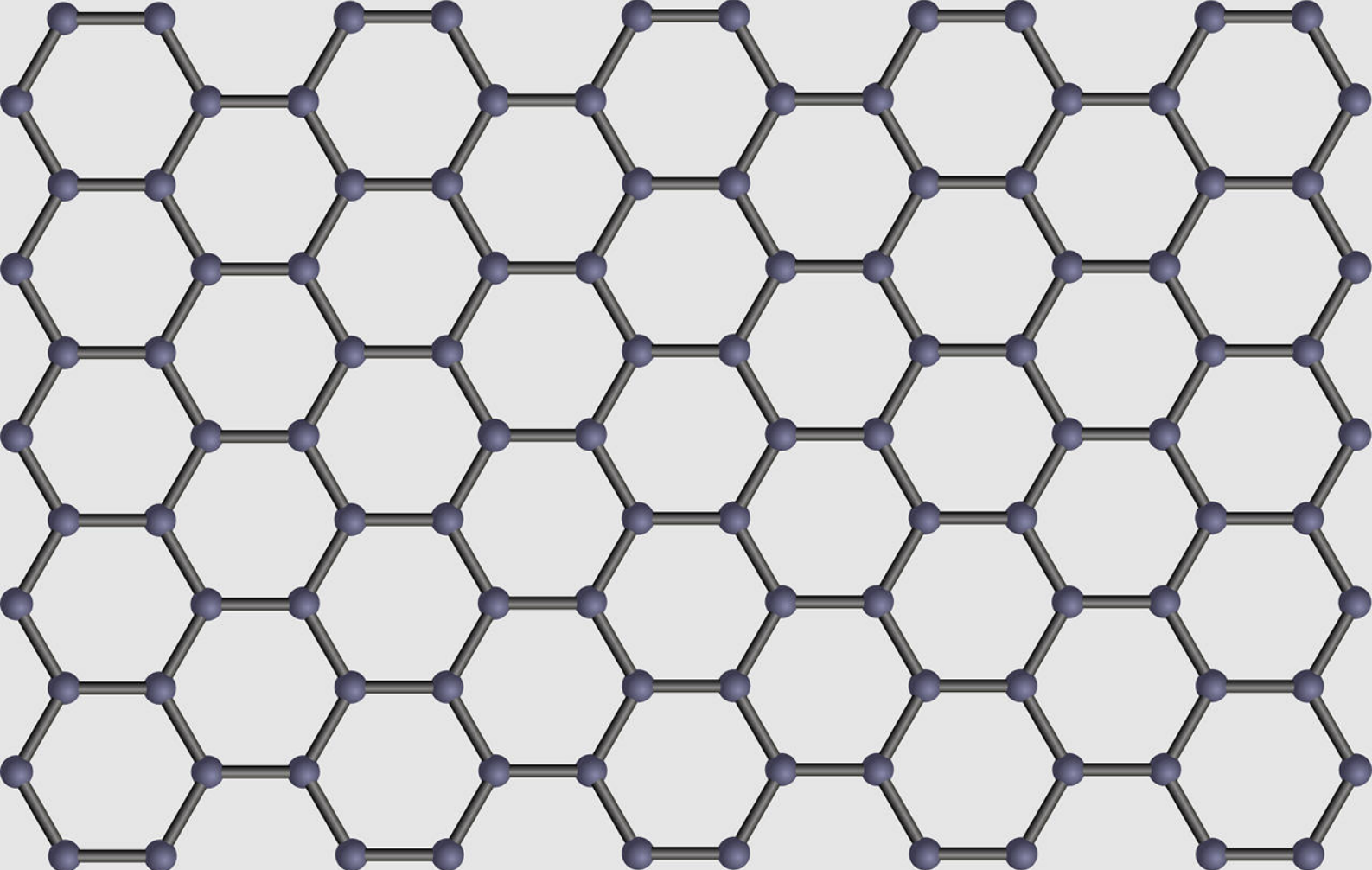




Application Note AN-T-226

Determination of functional groups in graphite and graphene oxide

Accurate and selective quantification of phenols, lactones, and carboxylates via Boehm titration

Boehm titration is a quantitative analysis of functional groups on the surface of carbon materials based on their reactions with basic solutions of NaHCO_3 ($\text{pK}_a = 6.4$), Na_2CO_3 ($\text{pK}_a = 10.3$), and NaOH ($\text{pK}_a = 15.7$). This is a cost-efficient method that gives absolute values with high precision of the accessible, mainly oxygen-containing functional groups on the surface. Originally, Boehm titration was developed for carbon materials like conductive carbon black (CCB), activated carbon, porous carbon, and

graphite. Modern carbon-based materials like graphene, graphene oxide (GO), or carbon nanotubes can also be analyzed this way. GO is a carbon-based two-dimensional nanomaterial containing high amounts of functional groups. It is mainly used to form reduced graphene oxide (RGO) (e.g. exfoliated graphene) as a modern high-end material with remarkable mechanical and electrical properties, applied in nanocells, detectors, nanoscaled conductive devices, batteries, and more.

SAMPLE AND SAMPLE PREPARATION

The sample materials are weighed into different glass beakers to react with added bicarbonate, carbonate, or sodium hydroxide solution for two

days. Blank samples must also be prepared for each base solution.

EXPERIMENTAL

Aliquots of the blank and sample were titrated against hydrochloric acid solution until after the last equivalence point (Figure 2).



Figure 1. OMNIS titrator with the digital pH electrode and a dosing module.

RESULTS

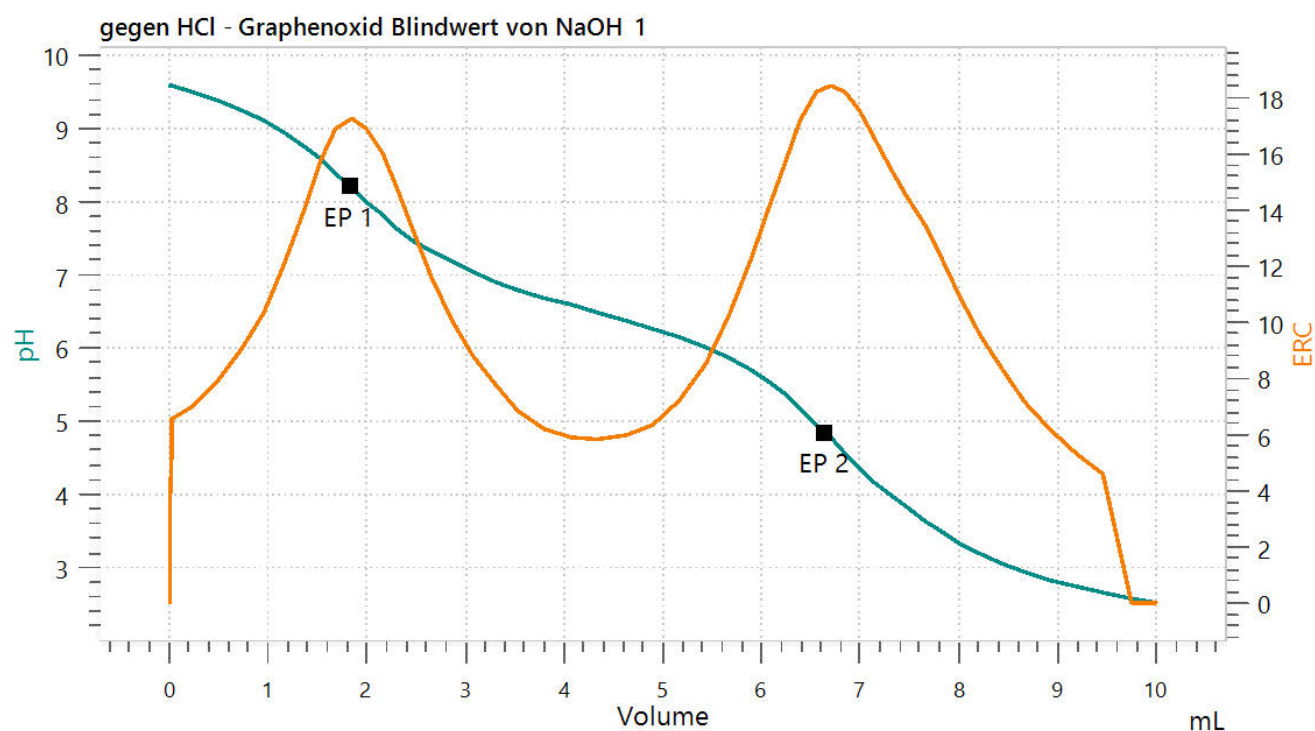


Figure 2. Exemplary titration curve of an aliquot of GO in NaOH solution with HCl as titrant.

Table 1. Summarized results for the functional group determination by Boehm titration of graphite and GO.

Base solution (n = 3)	Functional groups in graphite (mmol/g)	Functional groups in GO (mmol/g)
Sodium hydroxide	0.1982	5.7354
Sodium carbonate	0.0628	4.1399
Sodium bicarbonate	0.0452	3.6967

CONCLUSION

Boehm titration is the easiest and most cost-efficient way to quantify the number of functional groups on carbon materials. As expected, the amount of functional groups found on GO is significantly higher (30 times) compared to graphite. Therefore, this method

allows for quality control of carbon-based materials, and by using the high-end OMNIS platform, the results are directly calculated and displayed. Furthermore, analysis can also be automated reducing sources of human error and allowing a higher sample throughput.

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CONFIGURATION



OMNIS Advanced Titrator

创新型、模块式电位分析 OMNIS Titrator 滴定仪,适于独立运行或作为 OMNIS 滴定系统的核心元件运行,用于使用 OMNIS Sample Robot 进行终点和等当点滴定(单一/动态)。由于采用 3S 试剂瓶适配器技术,处理化学品很安全。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螺旋搅拌器。在需要时可以通过相应的软件功能许可证为平行滴定升级 OMNIS Advanced Titrator。

- 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四个滴定模块或加液模块
- 螺旋搅拌器的连接方式
- 可提供不同大小的计量管:5、10、20 或 50 mL
- 采用 3S 技术的试剂瓶适配器:安全处理化学品,自动传输生产商的原始试剂数据

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。



dEcotrode Plus

用于 OMNIS 的数字、组合式 pH 电极。

该电极适用于酸/碱水溶液滴定。

固定套管式隔膜对污染不敏感。

参比电解质: $c(\text{KCl}) = 3 \text{ mol / L}$, 储存在保存液中。

dTodes 可在 OMNIS Titratoren 上使用。