

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.

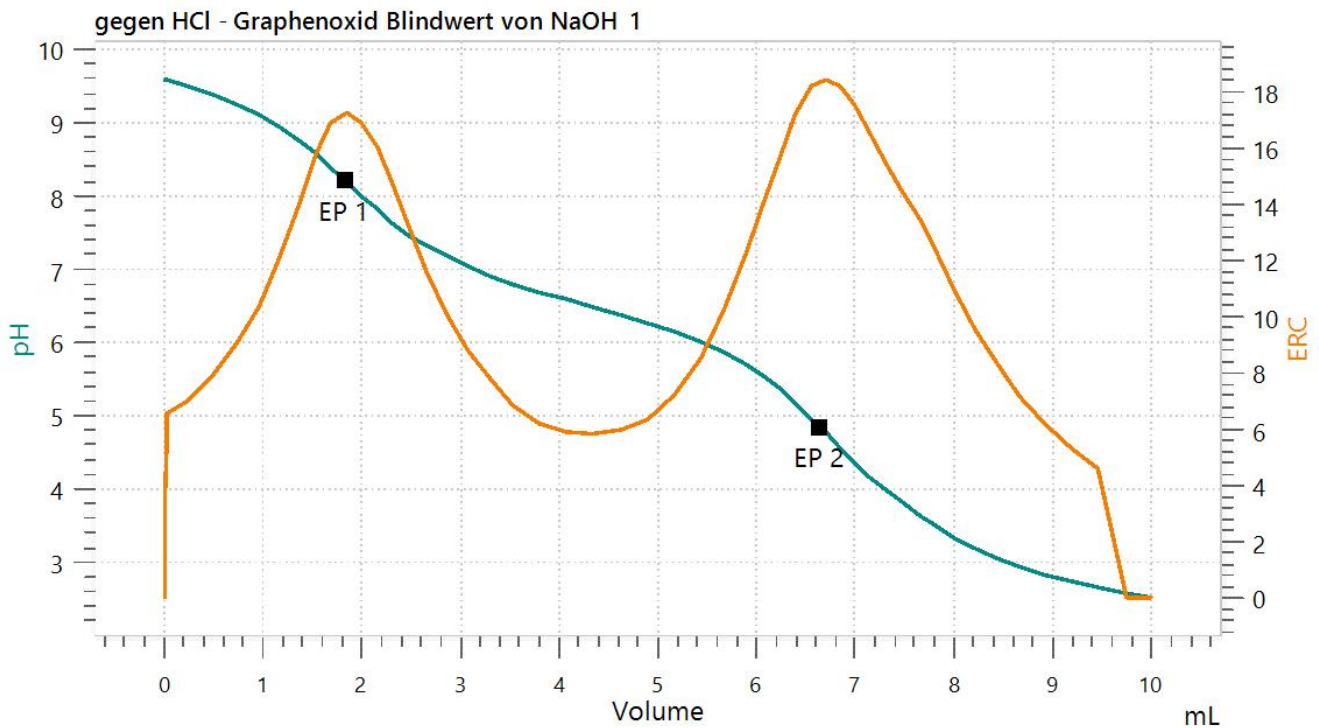


Figure 2. Exemplary titration curve of an aliquot of GO in NaOH solution with HCl as titrant.

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.

Internal reference: AW TI CH-1325-092021

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CONFIGURATION



OMNIS Advanced Titrator

新型、模式位分析 OMNIS Titrator 滴定,于独立行或作 OMNIS 滴定系的核心元件行,用于使用 OMNIS Sample Robot 行点和等当点滴定(-/). 由于采用 3S 瓶配器技,理化学品从未像在一安全。可以使用量模和量管元自由配置滴定,并在需要展一台螺旋拌器。在需要可以通相的件功能可平行滴定升 OMNIS Advanced Titrator。

- 通计算机或本地网控制
- 可以其他用或助溶液外接最多四个滴定模或加液模
- 螺旋拌器的接方式
- 可提供不同大小的量管:5、10、20 或 50 mL
- 采用 3S 技的瓶配器:安全理化学品,自生商的原始数据

量模式和件:

- 点定滴定:“Basic” 功能可
- 点和等当点滴定(-/):“Advanced” 功能可
- 点和等当点滴定(-/),包括平行滴定
:“Professional” 功能可



OMNIS Dosing Module

用于与 OMNIS Titrator 滴定相的加液模,以展外用于滴定/加液的滴定管。可以展磁力拌器和/或螺旋拌器,以作独的滴定台使用。可自由 5、10、20 或 50 mL 量管元。



dEcotrode Plus

用于 OMNIS 的数字、合式 pH。

用于酸/水溶液滴定。

固定套管式隔膜染不敏感。

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

dTrodes 可在 OMNIS Titratoren 上使用。