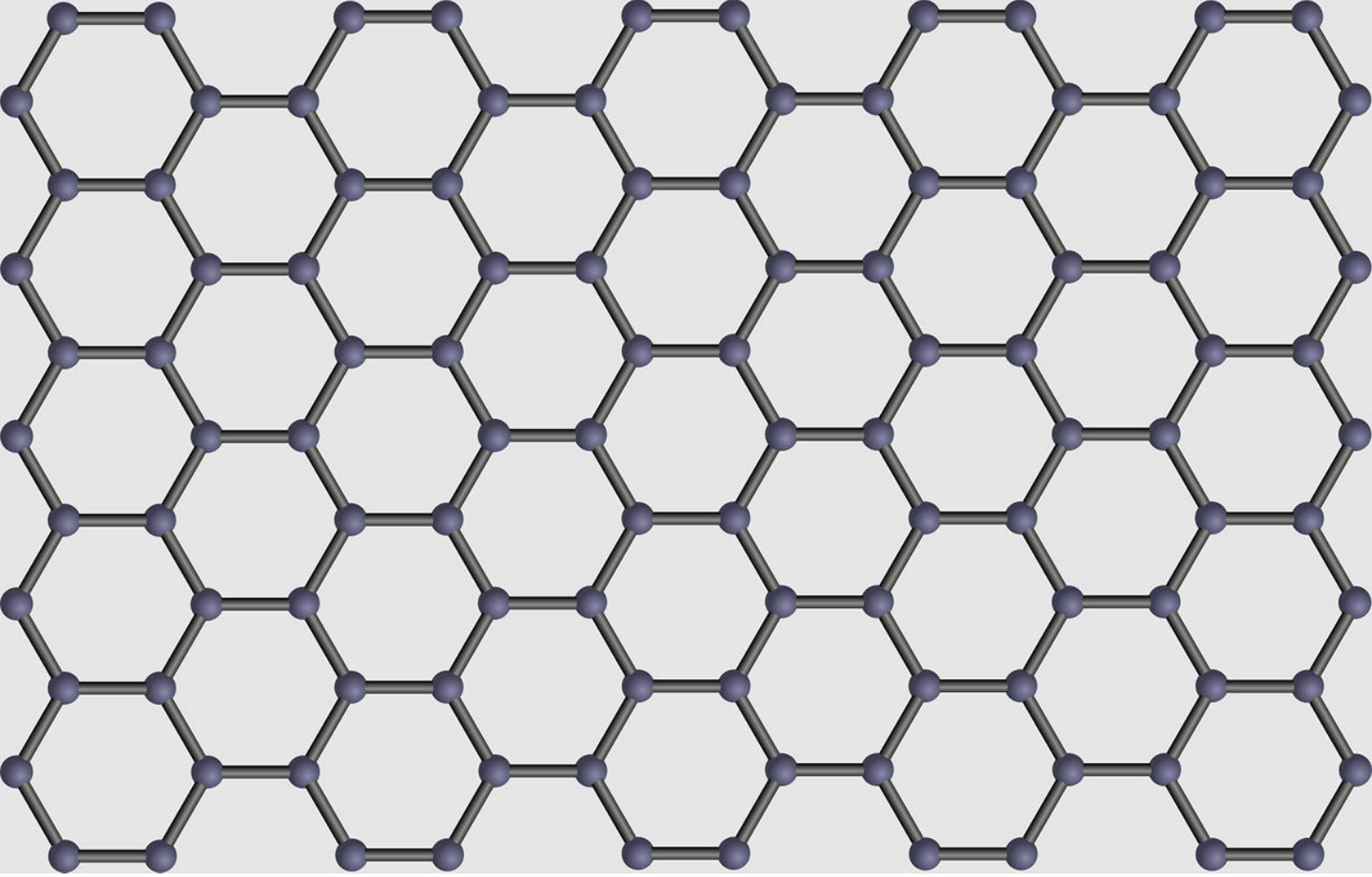


Application Note AN-T-226

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 $NaHCO_3$ (pKa = 6.4), Na_2CO_3 (pKa = 10.3), and NaOH (pKa = 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).

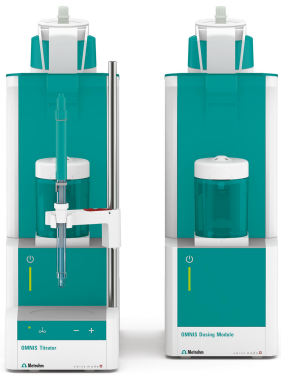


Figure 1. OMNIS titrator with the digital pH electrode and a dosing module.

RESULTS

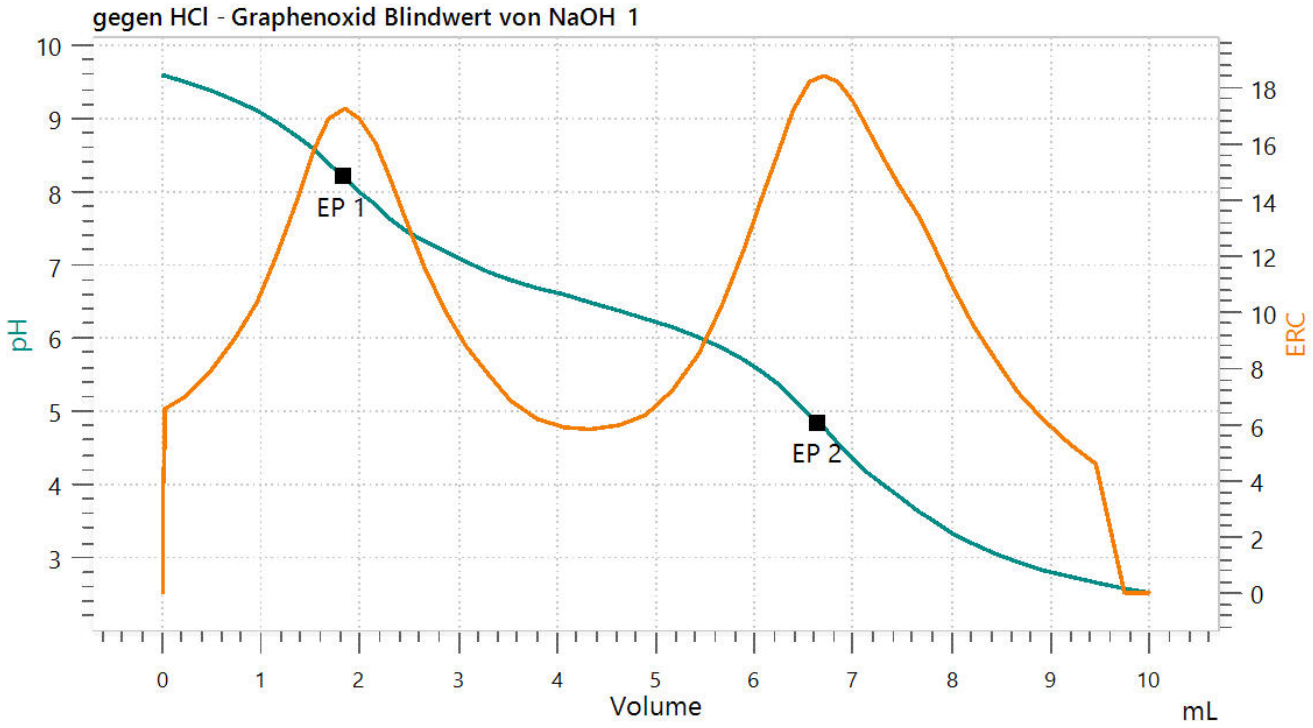


Figure 2. Exemplary titration curve of an aliquot of GO in NaOH solution with HCl as titrant.

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.

Internal reference: AW TI CH-1325-092021

CONTACT

Metrohm Nordic Oy
Jaakonkatu 2
01620 Vantaa
mail@metrohm.fi

CONFIGURATION



OMNIS Advanced Titrator with magnetic stirrer

Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a rod stirrer added as needed. If required, the OMNIS Advanced Titrator can be equipped for parallel titration via a corresponding software function license.

- Control via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Connection option for one rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Secure 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 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.



dEcotrode Plus

Digital, combined pH electrode for OMNIS.

The electrode is suitable for aqueous acid/base titrations.

The fixed ground-joint diaphragm is insensitive to contamination.

Reference electrolyte: c(KCl) = 3 mol/L, storage in storage solution.

dTrodes can be used on OMNIS Titrators.