Application Note AN-S-395

Forensic examination analysis with IC

Determining low concentrations of chlorate, thiosulfate, thiocyanate, and perchlorate beside major anions in explosives and explosion residues

Forensic institutes examine terrorist attacks and warfare agents via trace detection analysis of the used explosives and their residuals. Main ingredients of explosives are fuels and oxidizers, such as oxyhalides (e.g., chlorate, perchlorate), as well as nitrates, sulfur, phosphorous containing compounds, metals, sugars, and hydrocarbons [1]. Typical inorganic post-blast residues include thiocyanate and thiosulfate. Of particular importance is the acquisition of «chemical fingerprints» for criminal investigation departments and governmental security agencies. Institutes for public health and environmental protection analyze such compounds that can contaminate the underlying soil and infiltrate ground water. Ion chromatography (IC) using suppressed conductivity detection allows a sensitive and robust determination of anionic contaminants such as chlorate, thiosulfate, thiocyanate, and perchlorate next to the common inorganic anions over a broad concentration range.

EXPERIMENT

Artificial samples were dissolved in 10% methanol and automatically filtered using Inline Ultrafiltration. The Metrohm intelligent Partial Loop Injection Technique (MiPT) allows the injection of a precise variable volume depending on the sample load, and an automatic calibration.

The anions are separated on the analytical column Metrosep A Supp 4 - 250/4.0 using a sodium carbonate / sodium hydrogen carbonate eluent and a flow gradient (Figure 1).

Sequential suppression, including chemical and CO_2-suppression, reduces the background conductivity to around 1 µS/cm and vastly improves the signal-to-noise ratio. All anions are determined with a conductivity detector and quantified with the MagIC Net software.

Figure 1. Compact, user-friendly Metrohm IC instrumentation to quantify various anions in explosives and explosion residues.

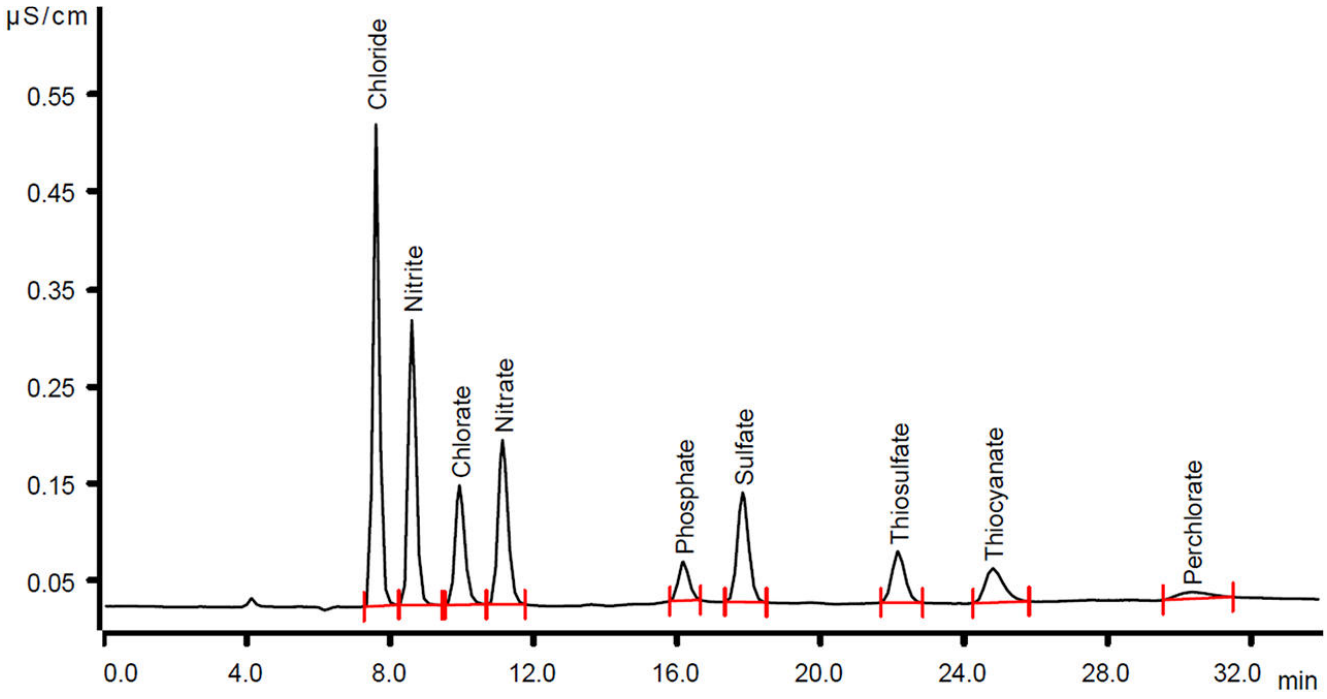

Figure 2. Suppressed conductivity signal of inorganic anions (1 mg/L), separated on a Metrosep A Supp 4 - 250/4.0 column (eluent: 1.8 mmol/L sodium carbonate, 1.7 mmol/L sodium hydrogen carbonate, flow gradient 0.7–1.5 mL/min, column temperature 30 °C, sample volume 10 µL).

RESULTS

The developed IC method offers a straightforward, robust, and fast analysis of anionic additives and residuals related to explosives. Figure 2 displays the chromatogram of a 1 mg/L standard solution. The method covers the specifications shown in Table 1.

Table 1. Method specifications

Parameter	Specification
LOQ	≤ 1mg/L for each anion, RSD ≤ 25%
Calibration	Anions = 1–100 mg/L Perchlorate = 1–50 mg/L
Resolution	≥ 2 for each separation
Blank	<0.1 mg/L
Run time	32 min

CONCLUSION

A flow gradient accelerates late-eluting components, which shortens analysis time to 32 minutes and improves peak shapes. The added methanol in the sample matrix did not interfere with the analysis in any way. For all components, the limit of quantification was below 1 mg/L and the resolution was above 2 for the full calibration range.

Using the Metrosep A Supp 4 column at ambient temperature enables the analysis with a compact IC system. For a comprehensive explosive characterization including the above specified anions as well as for cations, a two channel professional system provides a profitable solution.

Advanced studies of explosive residues are performed with IC-MS (ion chromatography coupled to mass spectrometry) to additionally confirm the analyte's identification with a mass detector [2].

REFERENCES

1. Dicinoski et al. (2006), Analytical Letters, 39(4), 639–657.
2. Barron et al. (2014), Analytica Chimica Acta 806 (2014) 27–54.

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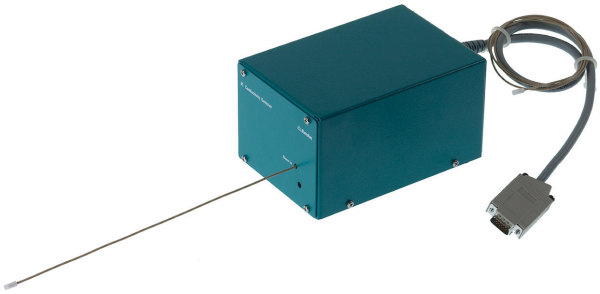
CONFIGURATION

930 Compact IC Flex Oven/SeS/Deg

The 930 Compact IC Flex Oven/SeS/Deg is the intelligent Compact IC instrument with **column oven**, **sequential suppression** and built-in **degasser**. An 800 Dosino can be used for the regeneration of the suppressor. The instrument can be used with any separation and detection methods.

Typical areas of application:

- Anion or cation determinations with sequential suppression and conductivity detection

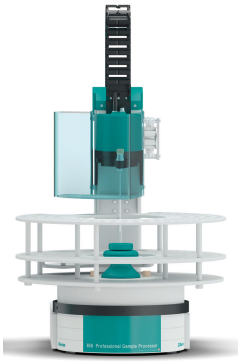


IC Conductivity Detector

Compact and intelligent high performance conductivity detector for intelligent IC instruments. Outstanding temperature stability, the complete signal processing within the protected detector block and the latest generation of DSP – Digital Signal Processing – guarantee the highest precision of the measurement. No change of measuring ranges (not even automatic ones) is required, due to the dynamic working range.

Metrosep A Supp 4 - 250/4.0

The Metrosep A Supp 4 - 250/4.0 is an extremely robust column with very good separation properties. The separation phase is comprised of polyvinyl alcohol particles with quaternary ammonium groups and a diameter of 9 µm. This structure guarantees great stability and a greater tolerance to very small particles that could pass through the integrated filter pad. The Metrosep A Supp 4 - 250/4.0 has a medium ion exchange capacity; sulfate elutes after 12.5 minutes. The plate numbers that can be achieved with this separation column is higher than those for the Metrosep Anion Dual 2 - 75/4.6. The A Supp 4 - 250/4.0 is particularly suitable for all routine tasks in water analysis.



858 Professional Sample Processor – Pump

The 858 Professional Sample Processor – Pump processes samples from 500 µL to 500 mL. The sample transfer takes place either with the installed bidirectional two-channel peristaltic pump or with an 800 Dosino.

MSM Rotor A

Suppressor rotor for all IC instruments with MSM (Metrohm Suppressor Module)