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.

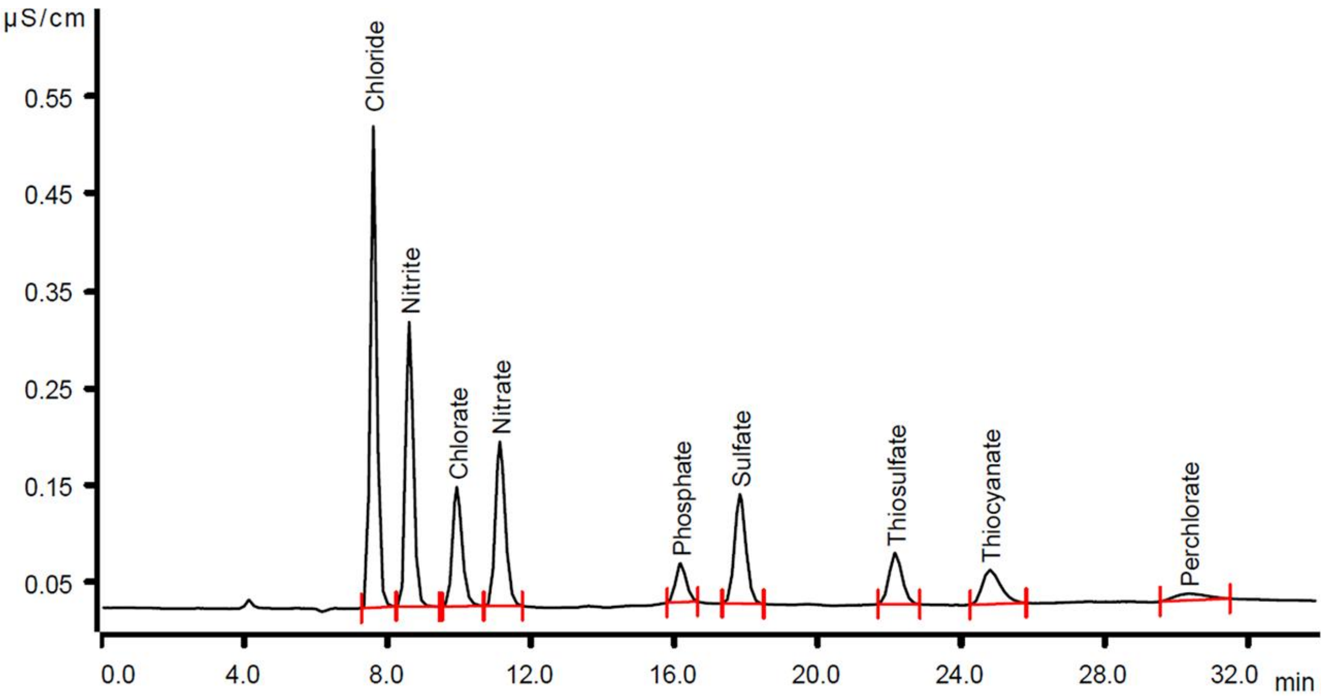


**Figure 2.** Suppressed conductivity signal of inorganic anions (1 mg/L), separated on a Metrosep A Supp 4 - 250/4.0 column (eluent: 1.8 mmol/L sodium carbonate, 1.7 mmol/L sodium hydrogen carbonate, flow gradient 0.7–1.5 mL/min, column temperature 30 °C, sample volume 10 µL).

## 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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