Application Note AN-S-395

用集成路行法医分析

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.

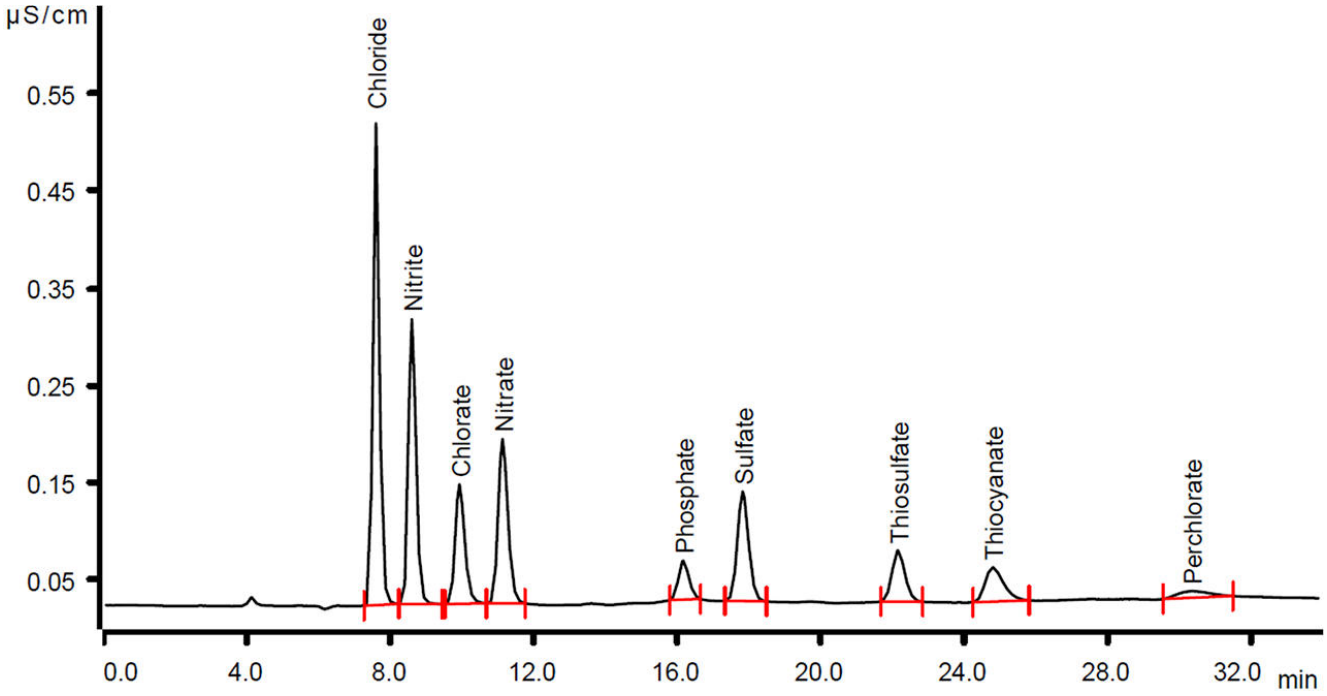

Figure 2. Suppressed conductivity signal of inorganic anions (1 mg/L), separated on a Metrosep A Supp 4 - 250/4.0 column (eluent: 1.8 mmol/L sodium carbonate, 1.7 mmol/L sodium hydrogen carbonate, flow gradient 0.7–1.5 mL/min, column temperature 30 °C, sample volume 10 µL).

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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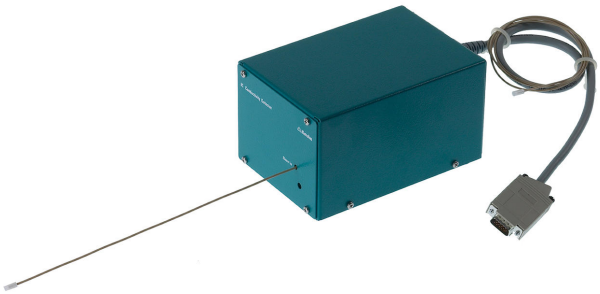
CONFIGURATION

930 Compact IC Flex Oven/SeS/Deg

930 Compact IC Flex Oven/SeS/Deg 是智能型 Compact 子色器,有**柱加炉**、**序列抑制器**和内置**脱气装置**。可使用一台 800 Dosino 用于抑制器再生。器可使用各分和方法。

典型的用范:

- 子或子定,序列抑制法及

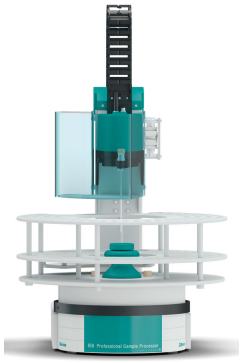


IC Conductivity Detector

用于智能型子色的智能型高性能器。不凡的温度定性,受保的器端子板内的整个信号理程以及新一代的 DSP(数字式信号理)均能保量的准性。功于工作范,无需行范更(也不是自行)。

Metrosep A Supp 4 - 250/4.0

Metrosep A Supp 4 - 250/4.0 是固的色柱,具有十分秀的分性能。分段包含聚乙醇粒,季基,直径 9 µm。此能保高定性,与可通内置板的微粒相比来允差更大。Metrosep A Supp 4 - 250/4.0 具有中等子交能力;硫酸将于 12.5 分之后淋洗出来。通此分柱可到的基数高于 Metrosep Anion Dual 2 - 75/4.6。A Supp 4 - 250/4.0 特用于水分析中的所有常工作。



858 Professional Sample Processor – Pump

858 Professional Sample Processor – Pump 可理体在 500 µL 至 500 mL 之的品。行品移,既可以使用内置的双向双通道蠕、也可通 800 Dosino 来行。

MSM A

抑制器子,用于所有MSM的IC器。