



Application Note AN-S-395

用集成电路进行法医检查分析

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 CO₂-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.

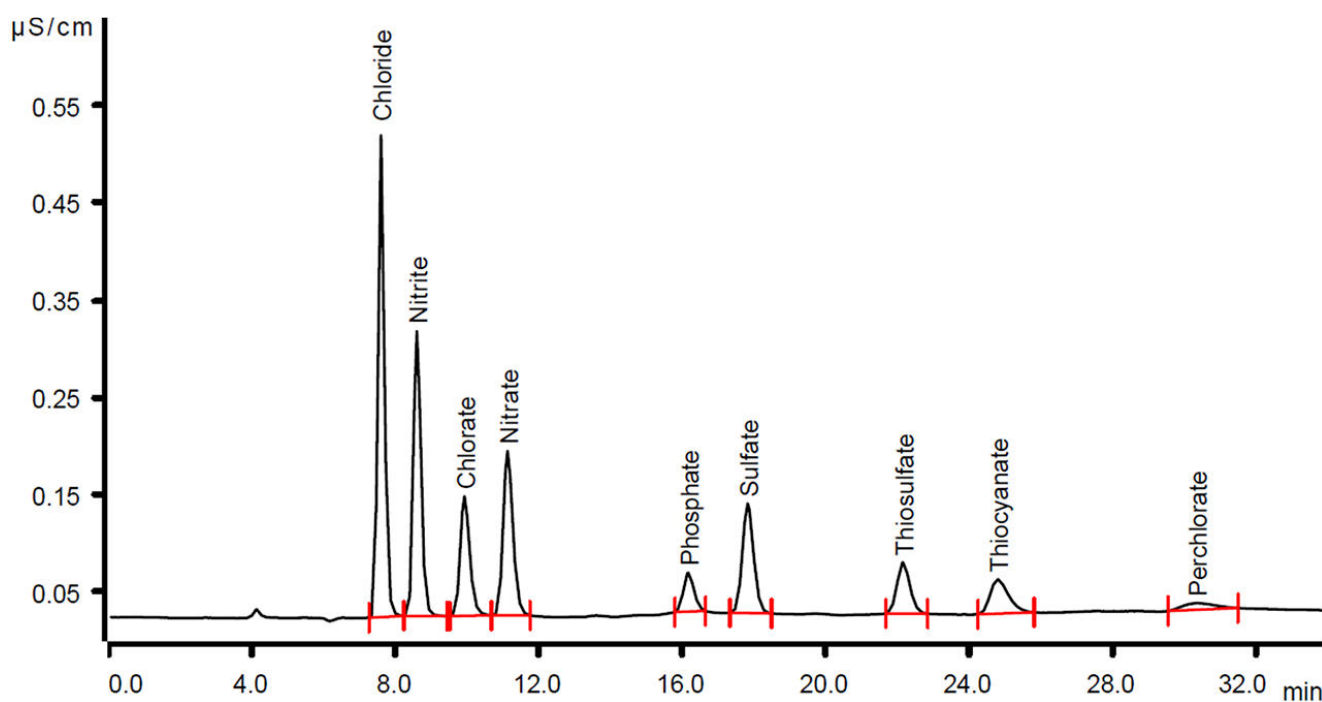


Figure 2. Suppressed conductivity signal of inorganic anions (1 mg/L), separated on a Metrosep A Supp 4 - 250/4.0 column (eluent: 1.8 mmol/L sodium carbonate, 1.7 mmol/L sodium hydrogen carbonate, flow gradient 0.7–1.5 mL/min, column temperature 30 °C, sample volume 10 µL).

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

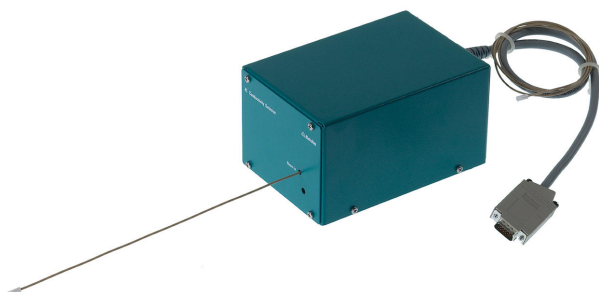


930 Compact IC Flex Oven/SeS/Deg

930 Compact IC Flex Oven/SeS/Deg 是智能型 Compact 离子色谱仪器,带有柱加热炉、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。该仪器可使用各种分离和检测方法。

典型的应用范围:

- 阴离子或阳离子测定,序列抑制法及电导检测



IC Conductivity Detector

用于智能型离子色谱仪的紧凑智能型高性能电导检测器。不凡的温度稳定性,受保护的检测器端子板内的整个信号处理过程以及新一代的 DSP(数字式信号处理)均能确保测量的准确性。归功于动态工作范围,无需进行范围变更(也不是自动进行)。



Metrosep A Supp 4 - 250/4.0

Metrosep A Supp 4 - 250/4.0 是极为坚固的色谱柱,具有十分优秀的分离性能。分离阶段包含聚乙烯醇颗粒,带季铵基团,直径为 9 μm 。此结构能确保高稳定性,与可通过内置过滤板的细微颗粒相比来说允差更大。Metrosep A Supp 4 - 250/4.0 具有中等离子交换能力;硫酸将于 12.5 分钟之后淋洗出来。通过此分离柱可达到的基数高于 Metrosep Anion Dual 2 - 75/4.6。A Supp 4 - 250/4.0 特别适用于水分析中的所有常规工作。



858 Professional Sample Processor – Pump

858 Professional Sample Processor – Pump 可处理体积在 500 μL 至 500 mL 之间的样品。进行样品转移时,既可以使用内置的双向双通道蠕动泵、也可通过 800 Dosino 来进行。



MSM A

抑制器转子,用于所有带MSM的IC仪器。