Analysis of trace residues of explosive materials following the guidelines set out in USEPA method 8330B and DIN EN ISO 22478

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INTRODUCTION
Nitroaromatics, nitrarnines, nitrate esters and peroxides represent the four major categories of explosive compounds in soil and ground water. Explosives are spread throughout the world because of their use in warfare, mining industries and civil constructions. Due to their toxicity, carcinogenicity and mutagenicity these compounds are considered a risk for public health and for the environment. Therefore the interest in highly sensitive analysis for explosives and propellants has increased. The most important guidelines for analysis of explosive materials are described in US Environmental Protection Agency (USEPA) method 8330B [1] and DIN EN ISO 22478 method [2]. These methods provide a solid phase extraction method and high performance liquid chromatographic conditions for 10 compounds (USEPA) and 20 compounds (DIN) with UV detection – in total for 22 different target compounds. In the first part of this work we present a methodology for sample preparation of water analysis which includes a solid phase extraction (SPE) method. The second part of this work points out the best high performance liquid chromatographic conditions on core-shell columns for 22 explosive compounds.

SOLID PHASE EXTRACTION METHOD
According to DIN EN ISO 22478 and EPA 8330B: developed on CHROMABOND® HR-X PP columns (3 mL, 200 mg, 85 μm)

- Conditioning
  3 mL methanol, 3 mL acetonitrile, 10 mL water

- Sample application
  1000 μL water sample (with 5 g NaCl), flow rate 1000 mL/h

- Washing
  10 mL water, 10 min drying with N₂

- Elution
  2 mL methanol + acetonitrile (50 + 50, v + v)
  2 mL methanol + water (50 + 50, v + v)

- Solvent changing
  0.5 mL water as keeper

CHROMATOGRAPHIC METHOD
Column combinations for multidimensional chromatography

- NUCLEOSHELL® RP 18
  octadecyl, multi-endcapping,
  particle size 2.7 μm, 50 mm x 2 mm ID

- NUCLEOSHELL® PFP
  phenyl-hexyl, multi-endcapping,
  particle size 2.7 μm, 50 mm x 2 mm ID

- NUCLEOSHELL® PhenyI-Hexyl
  phenyl-hexyl, multi-endcapping,
  particle size 2.7 μm, 50 mm x 2 mm ID

RESULTS

- Compounds investigated
  Peak Explosive
  1 Methyl-2,6-dinitrotoluene (Tetryl)
  2 Diethyl glycol diether (DGG)
  3 Hexahydro-1,3,5,7-tetrazocine (HMX)
  4 2,4,6-Trinitrotoluene (TNT)
  5 Ethynylglycolic dinitrate (EGDN)
  6 1,5-Dinitrobenzene (1,5-DNB)
  7 1,5-Dinitrobenzene (1,5-DNB)
  8 2,4-Dinitrophenol
  9 Nitrosodimethylaniline
  10 Nitrosodimethylaniline
  11 2,4-Dinitrophenol
  12 2,4-Dinitrotoluene
  13 Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
  14 4-Amino-2,6-dinitrotoluene
  15 2,4-Dinitrophenol
  16 2,4-Dinitrophenol
  17 2-Nitromethane
  18 Nitromethane
  19 Nitromethane
  20 Pentahydrotrinitrobenzene (PETN)
  21 Cypiphenol (CPN)
  22 2,4,6-Trimethaphenylphosphoryl methane (Hay! HMK)

- SPE recoveries

- Extraction efficiency of NUCLEOSHELL® RP 18 (μm)
  - 0.5 mL water as keeper
  - Flow rate 1000 mL/h

CONCLUSION

The results of this work show that the solid phase extraction of explosives with CHROMABOND® HR-X is very well suited. In particular, a significantly better recovery is achieved in the solid phase extraction of the explosive analyte tetryl.

The chromatographic results illustrate that each of the column combinations has advantages in the separation of different classes of explosives due to the combined selectivities. Especially, a separation of the 19 analytes described by USEPA 8330B in a single run is made possible by the combination of the columns NUCLEOSHELL® RP 18 and PFP.

If the respective explosives categories are supplemented by other analytes such as degradation products, the results shown are good starting conditions for further optimizations.