DIONEX

Application Note 808

US EPA Method 529 — Determination of Explosives and Related Compounds in Drinking Water

INTRODUCTION

This method was developed for the automated extraction of explosives and related compounds as described in EPA Method 529. The analytes are determined by capillary column gas chromatography/mass spectrometry (GC/MS).

METHOD SUMMARY

5 mL of methanol is added to one liter of sample and spiked with 50 µl of 5 µg/mL surrogate solution. The samples are passed through a 47 mm SDB-RPS (chemically modified polystyrene divinyl benzene material) SPE disk. After elution with ethyl acetate, the extract is concentrated down to a final volume of 1.0 mL. Extracts were fortified with 20 µL of 200 µg/L internal standard solution and analyzed using a GC/MS in accordance with EPA Method 529.

INSTRUMENTATION USED FOR SAMPLE PREPARATION

Dionex AutoTrace® instrument

EQUIPMENT SPECIFICATIONS AND OPERATING CONDITIONS

Sample evaporated as follows:

Solvent starting volume: 25 mL
Solvent end volume: 1.0 mL
Solvent: Ethyl Acetate
Bath temperature: 30 ºC

GC/MSD capable of cold on-column injection and 5973 MSD:

Column: Restek Rtx-5Sil MS 30 meter column, 0.25 mm ID, 0.25 µm df

	Inlet Conditions	Oven Conditions
Initial Temp	50 °C	50 °C for 3 min
Ramp 1	150 °C/min to 210 °C for 2 min	7.0 °C/min to 210 °C for 0.0 min
Ramp 2		20.0 °C/min to 250 for 0.0 min
Final Temp	210 °C	250 °C for 0.0 min

SPE disk used: 47 mm SDB-RPS

AutoTrace SPE Instrument Procedure

Process the six samples using the following procedure:

Step 1: Rinse disk with 5.0 mL of EtAC into solvent waste.
Step 2: Dry disk with gas for 5.0 min.
Step 3: Condition disk with 5.0 mL of CH_3OH into solvent waste.
Step 4: Condition disk with 5.0 mL of CH_3OH into solvent waste.
Step 5: Condition disk with 5.0 mL of CH_3OH into solvent waste.
Step 6: Dry disk with gas for 5.0 min.
Step 7: Condition disk with 10.0 mL of reagent water into aqueous waste.
Step 8: Condition disk with 10.0 mL of reagent water into aqueous waste.
Step 9: Load 1000.0 mL of sample onto disk.
Step 10: Dry disk with gas for 5 min.
Step 11: Manually rinse sample container with 5.0 mL to collect (EtAc).
Step 12: Manually rinse sample container with 5.0 mL to collect (EtAc).
Step 13: Manually rinse sample container with 5.0 mL to collect (EtAc).
Step 14: Soak and collect 5.0 mL fraction to a second tube using EtAc.
Step 15: Collect 5.0 mL fraction into sample tube using EtAc.
Step 16: End

Solvent 1:	1:1 EtAc/$MeCl_2$	Cond Flow:	10 mL/min
Solvent 2:	CH_3OH	Load Flow:	15 mL/min
Solvent 3:	Water	Rinse Flow:	20.0 mL/min
Solvent 4:	$MeCl_2$ (Dichloromethane)	Elute Flow:	5.0 mL/min
Solvent 5:	EtAc (Ethyl acetate)	Cond Air Push:	15.0 mL/min
		Rinse Air Push:	20.0 mL/min
		Elute Air Push:	5.0 mL/min

Pass the eluate through anhydrous sodium sulfate to remove residual water that may be present from the sample container and the SPE disk.

RESULTS AND CONCLUSION

The following table shows recoveries of selected compounds.

Compound	Mean (µg/L)n=4	Accuracy Mean Rec (%)	Precision/% RSD
1,3-Dinitrobenzene	3.23	86.2	1.58
RDX	4.18	83.6	4.26
2,4,6-trinitrotoluene (TNT)	3.11	82.8	1.98

RDX- Hexahydo-1,3,5-trinitro-1,3,5-triazine

This AB has shown that AutoTrace provides an automated solution to US EPA Method 529.

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LPN 2217 PDF 02/09