

# Determination of BNAs in large-volume soil sample using Microwave-Assisted Solvent Extraction

Milestone Application Note for Microwave Extraction - 04



This app note describes the use of Ethos X Microwave Extraction system utilizing fastEX24 rotor with contact-less temperature sensor and disposable glass vials to extract base/neutral and acid (BNAs) from spiked soils during a recovery study following US EPA Method 3546.

## Summary

Base/neutral and acid (BNAs) compounds are strictly regulated on the state and federal levels.

Responsible parties must determine the concentration and extent of contamination to make appropriate decisions regarding remediation of contaminated soils. This application note represents a guideline of microwave-assisted solvent extraction in the extraction of BNAs from soil.

Milestone Ethos X equipped with fastEX24 rotor benchtop microwave extraction system, fully compliant with US EPA 3546, was used for this study. FastEX 24 rotor works with 145 mL Weflon vessels with disposable glass vials of 100 mL. Thanks to the Weflon construction and the contactless temperature control in all positions, FastEX 24 rotor allows a simultaneous extraction of 24 samples ensuring a perfect temperature uniformity. Built-in methods and app notes provide an unmatched ease of use and low running costs.



## Method details

Samples, wet or dried and ground, were weighed directly into the 100-mL extraction glass vials. In case of wet sample, anhydrous sodium sulfate is added and mixed until a free-

flowing powder appearance is achieved. An aliquot of the surrogate solution were added to the samples just prior to solvent addition. The recovery study was performed on spiked soil. The glass vials were then closed into vessels and the microwave extraction program were started. The samples were extracted using the conditions mentioned above. The extraction procedure so described follows the detailed method provided by U.S. EPA SW-846 Method 3546.

Sample weight (g)	1:1 acetone – CH <sub>2</sub> Cl <sub>2</sub> (mL)
Up to 10	25
10 -20	35
20-30	50

After the extraction, samples were filtered on glass fiber filters and sodium sulfate anhydrous and the vials were rinsed with additional solvent aliquots. Extracts and rinsates were collected together.

## Microwave program

Step	Time (min)	T2 (°C)	Power (W)
1	00:15:00	110°C	up to 1600 W*
2	00:10:00	110°C	up to 1600 W*

\*The power applied depends on the moisture content. Dedicated methods are pre-loaded in the ETHOS X software according to the moisture content.



## Analytical results

Results from extractions of spiked soil are shown in table below. The tables show the recovery and the RSD (%) for BNAs, content of that matrix. Recovery for all compounds were in the range 70-120% of spiked amounts. The results were very reproducible (RSD %) despite the huge sample amount (30g of soil sample) used.

The results demonstrate the efficiency of the Ethos X as sample preparation method for GC analysis even in working with high sample amount. This method is particularly indicate to work at very low action levels. Ethos X provides extracts with the lowest solvent usage and significant time compared to all the other extraction technique.

*BNAs recoveries (n=4) from 30g spiked soil (1667 µg/kg).*

Target Compounds	Recovery (%)	RSD (%)
1,2,4,5-Tetrachlorobenzene	82.7	14.2
1,2,4-Trichlorobenzene	75.2	13.2
1-Methylnaphthalene	70.9	14.0
2,4,5-Trichlorophenol	76.3	14.9
2,4,6-Trichlorophenol	78.7	14.5
2,4-Dichlorophenol	77.5	10.2
2,4-Dimethylphenol	80.3	13.2
2,4-Dinitrophenol	68.9	9.5
2,4-Dinitrotoluene	81.9	12.3
2,6-Dinitrotoluene	79.2	11.1
2-Chloronaphthalene	80.1	10.6
2-Chlorophenol	71.2	12.2
2-Methylnaphthalene	75.5	13.5
2-Methylphenol	72.3	11.3

2-Nitroaniline	73.4	9.3
2-Nitrophenol	77.4	13.5
3-Methylphenol	73.2	4.0
3,3'-Dichlorobenzidine	118.1	4.14
3-Nitroaniline	73.4	7.3
4,6-Dinitro-2-methylphenol	77.9	8.0
4-Bromophenyl phenyl ether	82.2	11.7
4-Chloro-3-methylphenol	76.6	9.4
4-Chloroaniline	77.7	6.9
4-Chlorophenyl phenyl ether	81.5	13.8
4-Nitroaniline	116.4	5.4
4-Nitrophenol	74.4	12.6
Acenaphthene	76.5	11.8
Acenaphthylene	73.2	9.2
Aniline	72.6	8.3
Anthracene	83.3	13.6
Benzo(a)anthracene	82.3	12.6
Benzo(a)pyrene	79.8	12.8
Benzo(b)fluoranthene	77.8	11.2
Benzo(g,h,i)perylene	78.5	9.3
Benzo(k)fluoranthene	72.0	8.0
Benzyl butyl phthalate	75.4	7.8
Bis(2-chloroethoxy)methane	73.6	6.3
Bis(2-chloroethyl)ether	70.3	12.3
Bis(2-chloroisopropyl)ether	74.1	7.3
Bis(2-ethylhexyl)phthalate	79.1	9.3



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Carbazole	104.8	7.9
Chrysene	84.0	14.1
Dibenzo(a,h)anthracene	83.8	11.6
Dibenzofuran Diethyl	76.6	9.2
phthalate Dimethyl	77.8	10.2
phthalate Di-n-butyl	75.5	12.1
phthalate Di-n-octyl	77.2	11.9
phthalate	76.4	11.7
Fluoranthene	81.8	12.5
Fluorene	81.5	9.33
Hexachlorobenzene	81.5	10.5
Hexachlorobutadiene	73.4	9.9
Hexachlorocyclopentadiene	75.4	8.0
Hexachloroethane	72.3	6.0
Indeno(1,2,3-cd)pyrene	81.1	5.5
Isophorone	78.4	11.0
Naphthalene Nitrobenzene	79.6	12.0
N-nitroso-di-n-propylamine	84.4	6.5
N-Nitrosodiphenylamine	80.5	8.7
Pentachloronitrobenzene	111.4	6.4
Pentachlorophenol	91.3	10.6
Phenanthrene	76.4	9.2
Phenol	85.3	8.5
Pyrene	74.7	7.8
Pyridine	80.7	5.6
<b>Surrogate</b>		
2-Fluorophenol	79.0	9.5

Phenol-d5	75.1	5.1
Nitrobenzene-d5	80.4	7.4
2-Fluorobiphenyl	82.3	8.3
2,4,6-Tribromophenol	87.5	8.56
Terphenyl-d14	77.4	9.7

## General precautions

Always use hand, eye and body protection when operating with the microwave system.

## Conclusion

The ETHOS X enables simultaneous solvent extraction of up to 24 samples. The use of contactless temperature control ensures high reproducibility and full recovery of BNAs. Ethos X meets the requirements for BNAs analysis as described in US EPA 3546. Ethos X extraction of BNAs compounds from soils is more convenient, faster, and less solvent-intensive than all the other extraction approaches.

*Subject to change without notice.  
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