Polycyclic aromatic hydrocarbons in sludge samples

Lyovapor™ L-200 Pro, SpeedExtractor E-916, Syncore® Analyst R-12: Pressurized Solvent Extraction of a sludge sample for the determination of polycyclic aromatic hydrocarbons (PAHs)

Polycyclic aromatic hydrocarbons (PAHs) are organic compounds of hydrogen and carbon, composed of numerous aromatic rings. PAHs are nonpolar and lipophilic and primarily found in natural sources such as creosote. They are also produced through the incomplete combustion of organic matter[1]. Nowadays, the main sources of PAH pollution, are caused by human activity and vary across the globe. PAHs are also formed during certain methods of food preparation such as charbroiling, grilling, roasting and frying[2].

Many countries and regions have regulations to prohibit and detect the compounds of PAHs.

In 1982, the American Environmental Protection Agency (EPA) introduced 16 representative PAHs as priority compounds for the monitoring of pollutants.

In 2016, the Ministry of Ecology and Environment of the People's Republic of China published a new series of decrees and regulations regarding PAHs in environmental areas such as soil, sediment, sludge and air. In the regulations HJ 783-2016 and HJ 805-2016, they recommend the extraction of a solid sample using the Pressurized Solvent Extraction method of lyophilized samples prior to analysis by GC-MS[3].

1. Introduction

In this Short Note, sludge was collected from the outfall of a chemical plant and used as a sample to detect the content of the 16 PAH compounds listed in the EPA regulations. The sludge samples were freeze-dried to remove moisture prior to extraction using PSE. The extracts were then concentrated using the Syncore Analyst and analyzed by GC-MS.

2. Experimental

Instrumentation: Lyovapor™ L-300 Pro, SpeedExtractor E-916 with 20 mL cells, Syncore Analyst R-12 with SPE module, GC-MS Agilent GC-5975MSD

Sample: Sludge collected from the outfall of a chemical plant near Beijing.

Prior to freeze drying the sample was deep frozen at - 24 °C for 24 h. Then the sample was freeze dried with the LyovaporTM L-300 Pro using the parameters shown in Table 1.

Table 1: Parameter for freeze drying using the Lyovapor™ L-300 Pro

Chamber heater	Non-heatable shelf with stainless steel
Gas type	Ambient air
Temperature of condenser	-55 °C
Condenser pressure	0.5 mbar
Operation mode	Manual
Time	17 h

3 g activated Florisil and 5 g quartz sand were placed in a 20 mL extraction cell. 3 g sample was mixed with 8 g quartz sand and added. The sample was extracted with the parameters shown in Table 2. The received extracts were purified with the Syncore Analyst R-12 using the SPE advanced module with Florisil columns and analyzed by GC-MS.

Table 2: Extraction method for SpeedExtractor E-916.

Parameter	Value
Temperature	100 °C
Pressure	120 bar
Solvent	Acetone / n-Hexane (50 % / 50 %)
Cells	20 mL
Vials	150 mL Syncore vessels
Cycles	2
Heat-up	1 min / 1 min
Hold	10 min / 10 min
Discharge	2 min / 2 min
Flush with solvent	2 min
Flush with gas	4 min

3. Results

The results presented in Table 3 show low variations and good recoveries.

Table 3: Results of the sludge sample and standard mix solution extracted by the SpeedExtractor E-916, n=3.

	Sludge sample		Recovery Standard mixture	
	Average [µg/g]	rsd [%]	Average [µg/g]	rsd [%]
Benzo[a]pyrene	**2.35**	0.95	**92.08**	3.10
Benzo[a]anthracene	**1.78**	3.00	**93.91**	4.07
Dibenzo[a,h]anthracene	**1.86**	3.72	**99.72**	1.82
Benzo[k]fluoranthene	**2.17**	1.86	**93.22**	2.64
Chrysene	**1.65**	2.67	**97.89**	1.19
Acenaphthene	**0.96**	5.31	**83.23**	2.87
Acenaphthylene	**1.01**	3.93	**87.68**	2.41
Anthracene	**1.11**	4.18	**86.05**	4.09
Benzo[ghi]perylene	**2.08**	2.97	**81.51**	1.60
Fluoranthene	**1.03**	4.37	**104.79**	1.51
Fluorene	**0.98**	4.85	**88.91**	1.66
Indeno[1,2,3-cd]pyrene	**2.08**	2.14	**95.64**	3.40
Naphthalene	**0.68**	4.80	**77.16**	5.13
Phenanthrene	**1.07**	4.52	**105.28**	2.36
Pyrene	**1.12**	4.13	**95.78**	3.30
Benzo[b]fluoranthene	**2.10**	1.62	**94.49**	2.93

4. Conclusion

The extraction by SpeedExtractor E-916 represents a reliable and comparable procedure for the determination of PAH in sludge samples.

5. References

[1] Sörensen, A. Ullmann's Encyclopedia of Industrial Chemistry; Wiley-VCH; 2009.
[2] WHO: Chemical hazards in drinking-water, Geneva, 1998
[3] HJ 783-2016 and HJ 805-2016
For more detailed information and safety considerations please refer to the Application Note No. 377/2019.