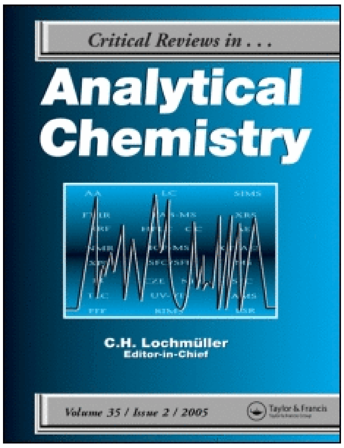

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Amines in the Environment

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The determination of the amines in the environment is very important. In this review, different methods for the analysis of the amines in the environment are discussed. Different aspects of the toxicity of the amines are discussed in detail. Methods for the sampling of the amines in air, soil, and water are discussed. Though there different techniques for the analysis of the amines only high performance liquid chromatography, gas chromatography, and capillary electrophoresis are discussed here. The different draw back, of these methods and their advantages are discussed in detail. The salient feature of the review is the discussion on the application of these methods for the determination of the amines in soil, water, and air, i.e., environmental samples.

Keywords environmental (air, natural water, soil), amines (low molecular weight/ LMW/aromatic and polyaromatic), chromatographic methods (CE, HPLC, GC), derivatization (pre, on-line, in-line, and post)

INTRODUCTION

Amines are organic compounds and a functional group that contains a basic nitrogen atom with a lone pair. Amines are derivatives of ammonia, wherein one or more hydrogen atoms are replaced by organic substituents such as alkyl and aryl groups. Compounds with the nitrogen atom next to a carbonyl of the structure R-C(=O)NR_2 are called amides and have different chemical properties. Important amines include amino acids, biogenic amines, trimethylamine (fish smell), and aniline. Amines have strong, characteristic odors and are toxic. The smells of ammonia, old fish, urine, rotting flesh, and semen are all mainly composed of amines. Many kinds of biological activity produce amines by breakdown of amino acids.

In recent decades, pollutant compounds in the environment have been increasingly studied because of their toxicity and bioaccumulation. Hundreds of compounds have been identified, many of which have been qualified as priority by the US Environmental Protection Agency (EPA) and European Union (EU). Most of these compounds are released into the environment as a consequence of human activity. The environment is subject to such excessive strains as farming, industry, and densely populated areas, which inflict serious damage on the ecological balance. Among these compounds amines and their derivatives are compounds, which are extensively used as drugs, cosmetics, polymers, pesticides, rubber, dyes, adhesives, solvents, corrosion inhibitors, in the synthesis of pesticides, and as synthetic intermediates (1–3). They find application as raw materials or intermediate products in the manufacturing of other chemicals. They are used as surfactants, emulsifying agents, and components of depilatory creams. Other uses include electroplating, textile manufacturing, vulcanization accelerators, fuel additives, rocket fuels, etc. (4–10). Amines can be found in many different matrices, from environmental samples to industrial raw materials, products, and wastes. Determination of amines in these matrices is a challenging task because of the highly polar character of these compounds. Both their isolation from the matrix and their final chromatographic determination are usually very difficult.

An extensive monograph is available on the biogenic amines, *Die Biiogenen Amine;* our knowledge of these compounds has increased considerably. Understanding of the biosynthesis of biogenic amines, their cellular and subcellular distribution, and of the mechanisms controlling their secretion and uptake by nerve endings has advanced substantially. In addition to being neuro-transmitters, biogenic amines also function as hormones that influence and modify the secretion of various other hormones, hypothalamic releasing and inhibiting factors, pituitary tropic hormones, and adrenal steroids. The abnormal secretion and (or) metabolism of biogenic amines are associated

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with many diseases such as pheochromocytoma, neuroblastoma, schizophrenia, and malignant hyperthermia. The problem of whether or not biogenic amines are etiologically involved in essential hypertension has also been widely investigated. To elucidate the role biogenic amines play in hormonal control and in the etiology and pathogenesis of disease, more advanced, sensitive, and quantitative methods are needed for their accurate measurement.

Amines can be present in food during the heating process of organic components composed of nitrogenous compounds, mainly proteins. The temperature during the heating controls the nature of the amine produces. Primary aromatic amines (PAA) can be transferred from the food packing materials into foodstuffs as these are used as adhesive relative mixtures containing aromatic isocyanate monomers. Isocyanates react with water to produce aromatic amines. Aromatic amines such as 2,4-diaminotoluene and 4,4′-methylenedianiline are classified as carcinogenic amines to humans. Some of the amines are also used as popular drugs of abuse like amphetamine and amphetamine analogues. Cigarette smoke is also found to contain aromatic amines in it. Amines (generally the secondary amines) are oxidized to N-Nitrosamines in the presence of nitrosating agents such as nitrite or nitrate in the human diet, environment, and in the stomach or the intestine of small animals (11–14). These are more toxic than the amines and these are present in the environment and in food. Some volatile N-nitrosamines are found in foodstuffs, drinking water, rubber products, drug formulations, tobacco, and tobacco smoke (15–17).

The ever-growing interest in the state of the natural environment and ecotoxicological issues prompts the development of new sensitive and precise analytical methods for the determination of low levels of amines in various environmental compartments. Various methods are available for the estimation of the amines (1, 2, 11, 18–22) using chromatographic methods like gas chromatography (GC) with different detectors (2,18–20), high performance liquid chromatography (HPLC) (1, 22–28), ion-partition chromatography, ion-pair chromatography (29), capillary electrophoresis (CE) (30, 31), ultra-violet (UV) spectrophotometry (3, 24), UV-Vis combined with reflectance spectroscopy (32), and fluorimetric methods (33).

These methods have been reviewed for these amines e.g., in wines, catecholamines (34), heterocyclic amines in foods (35, 36), biogenic amines in foodstuffs like meat, *etc*. (37) and in plant materials (38). But so far no review is available for the analysis of the amines in the environment. Keeping in view the toxic importance of these amines we have reviewed method for the determination of amines using HPLC, GC, and capillary electrophoresis using different detection systems.

SAMPLING AND SAMPLE TREATMENT TECHNIQUES

When applied in the environment, some of these amines undergo degradation and dilution. Thus, they—and their metabolites—can only be determined at trace levels in environmental matrices with highly efficient separation methods and sampling techniques, which have good sensitivity and selectivity. Sampling techniques are very useful for the analysis of amines in environmental samples. The clean-up procedures for the complex sample matrix are very important for the accurate analysis of these compounds. General methods for sampling of heterocyclic amines have been reviewed in environmental samples. The important techniques like solid-phase microextraction (SPME) (2, 39–41), liquid phase microextraction (LPME) (42–45), solid, phase extraction (SPE) (11, 46, 47), resin exchange, ion pair extraction (29), and liquid-liquid extraction (LLE) (48–50) have been applied for the determination of amines in environmental samples. Muller et al. (2) described the SPME/GC-MS method for the determination of aromatic amines in the water samples using carbowax–divinylbenzene fiber. Various parameters for the extraction method were optimized. The extraction time was optimized to 30 minutes, desorption in 5 minutes in GC injector at 260°C. A salt concentration of 60% saturated solution and pH of 7.6 was optimized. Cardenes et al. (40) used SPME, using carbowax-templated resin fiber, coupled with high performance liquid chromatography ultra violet/diode array detector (HPLC-UV/DAD) for the determination of the heterocyclic aromatic amines. Absorption time and soaking time were significant variables, and 15 minutes for each one of the variables was chosen for the best response. He and Lee (42) used two modes of LPME for capillary GC. Both methodologies, i.e., static LPME and dynamic LPME, involve the use of very small amounts of organic solvent in a conventional microsyringe. The performance of the two techniques is demonstrated in the determination of two chlorobenzenes extracted into a single drop of toluene by the use of a 10-μL syringe. Both methods allow the direct transfer of extracted analytes into a gas chromatograph. Wang et al. (43) used a dynamic liquid-phase microextraction technique combined with gas chromatography/mass spectrometry (GC/MS) for the extraction of 10 chlorobenzenes from water samples into 1 μL of organic solvent by using a conventional microsyringe. The effects of extraction solvent, plunger movement pattern, sampling volume, number of samplings, and salt concentration on the extraction performance were investigated. Shen and Lee. (44) developed a new microextraction technique termed hollow fiberprotected LPME. Triazines were employed as model compounds to assess the extraction procedure and were determined by GC/MS. Toluene functioned as both the extraction solvent and the impregnation solvent. Important extraction parameters, such as the effect of salt, agitation, pH, and exposure time were optimized. Meseguer-Lloret et al. (46) used C_{18} solid-phase extraction cartridges for the pre-concentration and dansyl chloride for the derivatization in the analysis of low molecular mass aliphatic amines. In the conditions for the pre-concentration and derivatization, up to volumes of 5 mL, samples are drawn through the sorbent and the analytes retained are dansylated at basic pH, at 100°C for 10 minutes or

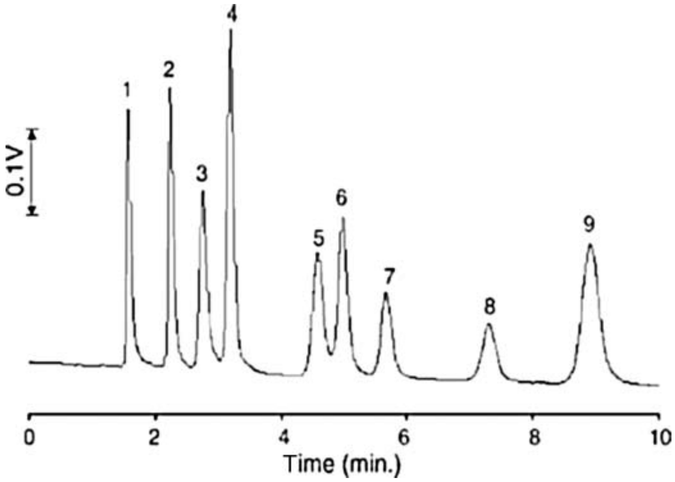

FIG. 1. Standard chromatogram for the separation of aromatic amines with EC detection. Detection potential 0.8V vs. Ag/AgCl column ABZ + PLUS (100 mm × 2.1 mm, 5 μm), mobile phase methanol–acetate buffer (pH 5), 45:55 (v/v), flow rate 0.2 mL/min, detection at 210 nm. Peaks identification; (1) 1,2-phenylenediamine; (2) aniline; (3) benzidine; (4) 2-toluidine; (5) 3,3_-dimethoxybenzidine; (6) 2,6 dimethylaniline; (7) 4-chloroaniline; (8) *N*,*N*-diethylaniline; and (9) 2-naphthylamine, each at 1 μg/mL^{-1}.

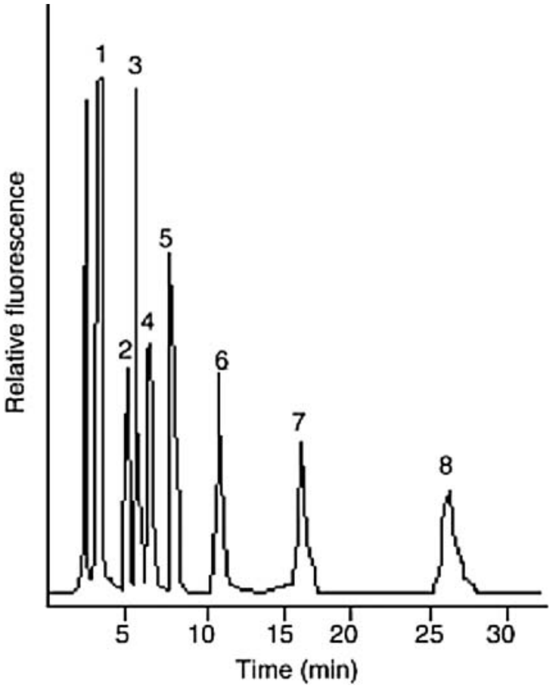

FIG. 2. Typical chromatogram of SAMF and amine derivatives. Mobile phase: methanol–water (57/43 (v/v)); flowrate: 0.7 mL/min. Standard amines concentration: 0.40 μmol/1. Peaks: (1) hydrolyzed SAMF; (2) SAMF-EOA; (3) SAMF-MA; (4) SAMF-EA; (5) SAMF-PrA; (6) SAMF-BA; (7) SAMFPA; (8) SAMF-HA. Here 6-oxy-(*N*-succinimidyl acetate)-9- (2_-methoxycarbonyl)fluorescein (SAMF), ethanolamine (EOA), methylamine (MA), ethylamine (EA), *n*-propylamine (PrA), *n*-butylamine (BA), *n*pentylamine (PA), and *n*-hexylamine (HA).

85°C for 15 minutes. The derivatized analytes are desorbed with 0.5 mL of acetonitrile. 20 mL of the collected extracts are chromatographed in a Hypersyl ODS C_{18} column using an acetonitrile–imidazole (pH 7) gradient for elution. Akyuz, and Ata (29) described a method for the simultaneous determination of aliphatic and aromatic amines using ion pair extraction with bis-2-ethylhexylphosphate and analyzed with GC-MS. Zhou et al. (48) established a method for the simultaneous analysis of eight aromatic amines in water samples by the combination of capillary zone electrophoresis and microporous membrane liquid-liquid extraction (MMLLE). The influence factors relevant to the MMLLE process were investigated and the final optimized parameters were used for analysis. Weiss and Angerer (49) used LLE for the extraction of aromatic amines from the human urine sample at pH 6.2–6.4. Yazdi et al. (50) described a liquid–liquid–liquid phase microextraction (LLLME) coupled with HPLC for the analysis of aromatic amines. These compounds were extracted from a 4.0-mL aqueous sample that adjusted to pH 13 with NaOH–NaCl buffer solution (donor phase, P_1) into an organic phase (P_2), 150 μL benzyl alcohol and ethyl acetate (2:1), and then back extracted into a microdrop of aqueous acceptor phase (P_3), adjusted at pH 2, with Na_2HPO_4–H_3PO_4 buffer solution. The extraction time, T_1 (from P_1 to P_2), was 20 minutes and T_2 (from P_2 to P_3) was 1 minute. Different crown ethers as complexing agents for amines were added to the acceptor phase to improve the extraction time.

DETERMINATION METHODS

High-Performance Liquid Chromatography

HPLC methods have been successfully applied for the determination of the amines in the environment. The detail of analysis of these compounds using LC with different detectors is summarized in Table 1. Highly polar, non-volatile, and thermally unstable compounds can be analyzed with HPLC. Using this technique derivatization is not always required and it can be used with several detectors. Among amines, heterocyclic amines have characteristic UV spectra and high extinction coefficients, and they are also electrochemically oxidizable. Some less polar amines fluoresce in the polar solvents. Therefore these compounds can be detected with the UV, DAD, electrochemical, and fluorescence detector. Shelke et al. (23) developed a μ-LC method for the fast and sensitive analysis of aromatic amines by electrochemical detection (Figure 1). Lehotay et al. developed a method for the determination of 2-napthylamine in waste gas using HPLC with DAD and electrochemical detectors (ED) (51). Piangerelli et al. described a method for the determination of aromatic amines and phenols in environmental samples by selective SPE

TABLE 1
Summary of the liquid chromatographic methods for the determination of amines in the environment

Sr. no.	Detection mode	Amines	Pre-conc. method	Derivatization reagrent	Sample	Detection limit	Ref.
1.	DAD and ED	2-naphthyl amine	—		Waste gas	50 ng/mL and 10 ng/mL	51
2.	Amperometric detection	Aromatic amines	SPE		Environmental	15–440 ng/L	52
3.	ED	Aromatic amines	—		Waste water	1.94–7.69 nM	53
4.	Fluorescence detector	Primary amines	—	o-phthalaldehyde	R-NMC-derived isoindoles	1 fM	54
5.	Fluorimetric detector	Aliphatic amines	SPE	Fluram	Water (distilled, rain, and tap)	0.02–1.20 μg/mL	55
6.	UV	Aromatic amines	SPME		Lake water	0.33–2.4 ng/mL	56
7.	DAD	Aromatic amines	SPE		Surface water	In ng/L	57
8.	UV-Vis	Aromatic amines	LLLME		Water	0.05–0.10 ng/mL	58
9.	UV	Carcinogenic aromatic amines	SPME		Water	1.0–2.2 ng/mL	59
10.	UV	Primary aliphatic short-chain monomines	SPE	o-phthaldialdehyde-N-acetylcysteine	Water	0.002–0.04 μg/mL	60
11.	DAD	Heterocyclic aromatic amines	SPME		Food	0.1–14 μg/mL	61
12.	UV	Methylamine	SPME	9-fluorenylmethyl chloroformate	Water	0.75 μg/mL	62
13.	PDA	Chlorinated anilines	LPME		Print works wastewater	0.5–1.0 μg/mL	63
14.	UV	Aromatic amines	LLLME		Water	0.8–1.8 ng/mL	50
15.	Fluorescence	Dimethylamine	SPME	9-fluorenylmethyl chloroformate	Water and air	0.3 μg/mL	64
16.	Fluorescence	Trimethylamine	SPME	9-fluorenylmethyl chloroformate	Water and air	0.25 μg/mL	65
17.	UV	Aniline derivatives	HFME and SDME		Water	1.0–2.5 μg/mL	66
18.	UV	Aromatic amines	LLLME		Waste water	0.5–1.5 ng/mL	67
19.	UV	Aromatic amines	Chemisorption	5,7-dinitrobenzofurazan	Air	NR	68
20.	ESI-MS	Low molecular weight amines	Device developed using 37 mm cassate with glass fibre filter Impregnated with H_2SO_4	Dansyl chloride	Ambient air	0.03–0.3 μg/mL	69

(Continued on next page)

TABLE 1
Summary of the liquid chromatographic methods for the determination of amines in the environment *(Continued)*

Sr. no.	Detection mode	Amines	Pre-conc. method	Derivatization reagrent	Sample	Detection limit	Ref.
21.	UV	Ammonia and aliphatic amines	SPE	Phenyl isothiocyanate	Environmental waters	0.2–0.6 μg/L	70
22.	Photovoltaic detector	Aliphatic and aromatic amines	—			2.3 ng/mL	71
23.	UV	Carcinogenic heterocyclic amines	—		River water	NR	72
24.	Fluorescence detector	Aliphatic and alicyclic amines	LLE	9-fluorenylmethyl carbamates	Surface and waste waters	—	73
25.	Fluorescence detector	Aliphatic amines	—	6-Oxy-(N-succinimidyl acetate)-9-(2′-methoxycarbonyl)fluorescein	Environmental and foods	2–320 fM	74
26.	Fluorescence detector	Aliphatic amines	—	3-(4-fluorinebenzoyl)-2-quinoline carboxaldehyde	Environmental and foods	0.5–2 nM	75
27.	Fluorescence detector and MS	Primary aromatic amines	—	2-(2-Phenyl-1H-phenanthro-[9,10-d]imidazole-1-yl)-acetic acid and 2-(9-acridone)-acetic acid	Waste water	0.12–0.21 nM	76
28.	ESI-MS	Primary, secondary and tertiary aliphatic and aromatic amine	—		Air	NR	77
29.	Fluorescence detector and APCI-MS/MS	Aliphatic amines	—	2-(2-phenyl-1H-phenanthro-[9,10-d]imidazole-1-yl)-acetic acid	Waste water and biological samples	3.1–18.2 fM	78
30.	DAD and tanden MS	Hydrazo derivatives, chlorinated aromatic and aliphatic amines	—		Degradation products of azo dyes		79

31.	Fluorescence detector and APCI-MS	Amines	—	2-(11H-benzo[a]-carbazol-11-yl) ethyl chloroformate		0.5 pM	80
32.	Tanden-MS	Aliphatic amines	—	1,2-benzo-3,4-dihydrocarbazole-9-ethyl chloroformate	Waste water and soil	18.65–38.82 fM	81
33.	Chemilumine-scence detection	Ammonium	—	Dansyl chloride	Water	8 μg/L	82
34.	Chemilumine-scence detection	Biogenic amines and opiate alkaloids	—	Tris(2,2′-bipyridyl)ruthenium(III) and acidic potassium permanganate	Human urine	1–5 × 10^{-7} M	83
35.	Fluorescence detector	Primary, secondary, and tertiary amines	—	Epichlorohydrin	Different reaction systems	NR	84
36.	RID	Monoethano-amine	—		Monoethano-amine nd its degradation products	NR	85
37.	UV-Vis	Aromatic amines	LLLME		Environmental waters	0.05–0.1 μg/mL	86
38.		Aromatic amines	LLLME		Water	0.85–1.80 ng/mL	87
39.	MS	Aniline	—		Freshwater	10 ng/mL	88
40.	Amperometric detection	Aliphatic amines	—	Phenylisothiocyanate	Water	In μg/L	89
41.	UV	N-nitroso dimethylamine	—		Dimethyl amine containing water	NR	90

TABLE 2
Summary of the gas chromatographic methods for the determination of amines in the environment

Sr. no.	Detection mode	Amines	Pre-conc. method	Derivatization reagent	Sample	Detection limit	Ref.
1.	MS	Aliphatic and alicyclic amines	—	Trichloroethyl carbamates	Water	0.005 μg/L	73
2.	FID	Amines	SPME	4-nitrophenyl trifluoroacetate	Air and water	Low ng/mL to pg/mL	91
3.	MS	Volatile amines	SPME	—	Air		92
4.	FID	Amines	CME	—	Aqueous samples	In pg/mL	93
5.	FID	Amines	SPME	—	Gaseous sample	0.1–0.3 ng/mL	94
6.	FID	Aromatic amines	SPME	—	—	0.1–1.5 μg/L	95
7.	FID	Volatile aliphatic amines	SPME	—	Air	0.55–1.88 mg/m^3	96
8.	FID and MS	Aliphatic amines	SPME	N-succinimidyl benzoate	Lake water	0.13–7.2 nM	97
9.	FID, NPD, and MS	Long chain primary alkyl amines	SPE	Trifluoroacetic anhydride	Boiler water in power plants	NR	98
10.	MS	Volatile amines	Cation-exch-ange resin	—	Polyurethane foams	0.70–1.1 ppm (v/v)	99
11.	NPD	Aliphatic amines	Static head space	—	Compost treatment plant	NPD	100
12.	ECD and NCI-MS	Aromatic amines	—	Heptafluoro butyric anhydride	Contaminated ground water	0.3–66.3 pg/mL	101
13.	NCI-MS	Amines	—	Heptafluoro butyric anhydride	Dichlorobenzidine-hemoglobin adduct	0.1 ng/g	102
14.	MS	Aliphatic amines	—	2,4-dinitrofluorobenzene and benzenesulfonyl chloride	Waste and surface water	0.10 μg/L	18
15.	MS	Aromatic amines	SPME	—	Water	0.002–0.025 μg/L	103
16.	MS	Aromatic amines	—		Ground water	0.1 ng/ml	104
17.	MS	Amines	—		Air	15 μg/L	105

18.	MS	Aniline and alpha-naphthylamine	—	—	Surface soils	NR	106
19.	AED and MS	Aromatic amines	—	Halogenation of the aromatic ring	Ground and waste water	NA	107
20.	FID, FTD, and MS	Low molecular weight amines	SPME	—	Aqueous extract of bird feces	NA	108
21.	MS	Dibutylamine	—	1-(2-methoxy phenyl)piperazine	Air	0.0006 mg/m^3*	109
22.	MS	Aliphatic and aromatic amines	SPE	Arylbenzoates and N-alkyl- or N-arylbenzamides	Environmental waters	7–39 ng/L	20
23.	MS	Primary aromatic amines	—	Trifluoroacetic anhydride	Water food simulant	0.1–0.4 μg/L	110
24.	MS	Volatile orgnic compounds	Sorbent tubes		Air near industrial dairy	NR	111
25.	MS	Aromatic amines	SPE	Heptafluorobutyric anhydride	Mainstream cigarette smoke	0.02 ng/cigarette	112
26.	MS	N-nitroso dimethylamine			Natural waters	NR	113
27.	MS	Aromatic primary amines	—	Allyl isothiocyanate	Waters	8–30 ng/L	114
28.	MS	Aromatic amines	—	Penta-fluoropropionyl	Cigarette smoke	6–39 pg/cig	115
29.	TOFMS	Nitrogen containing compounds	—	—	Cigarette smoke condensates	NR	116
30.	MS	Mono aromatic amines	—	—	Indoor air	0.02–0.06 ng/mL	117
31.	FID	Aniline	HS-SPME	—	Water	0.01 μg/mL	118
32.	MS	Aromatic amines	Ion pair extraction	Pentafluoro benzaldehyde	Water	0.07–0.50 ng/L	119
33.	MS	Aliphatic and aromatic amines	Ion pair extraction	Isobutyl chloroformate	Indoor and outdoor air	0.08–0.01 ng/m^3	120
34.	MS	Anilines	DLLME	Pentafluorobenzaldehyde	Waste water	0.04–0.09 μg/L	121
35.	NPD	Amines	Sorptive exrtraction/ thermal desorption	Pentafluoro benzoyl chloride	Aqueous samples	Sub ppb level	122

(Continued on next page)

TABLE 2
Summary of the gas chromatographic methods for the determination of amines in the environment. *(Continued)*

Sr. no.	Detection mode	Amines	Pre-conc. method	Derivatization reagent	Sample	Detection limit	Ref.
36.		Aromatic amines	SPME	—	Dye process wastewater	0.22–0.84 μg/L	123
37.	LRMS	N-nitroso dimethylamine	—	—	Dimethylamine containing water		124
38.	ECD	Aromatic amines	SPE	Bromide and iodide derivatives	Waters	0.5–8 μg/L	125
39.	MS	Volatile amines	Cation-exchange-resin	—	Air	0.18–1.51 ppm (v/v)	126
40.	MS	Aromatic amines	Simultaneous distillation and extraction	—	Particulate phase cigarette smoke	NR	127
41.	MS	Nitrogen compounds	SPE	—	Landfill leachate	NR	128
42.	MS	Heterocyclic amines	—	Heptafluoro butyric anhydride	Cigarette mainstream smoke	NA	129
43.	MS	Polar aromatic amines	SPME	By diazotation and to subsequent iodide derivatives using hydroiodic acid	Water and wastewater	27–38 ng/L	130
44.	MS	Volatile organic compounds	—	—	Indoor and outdoor air	NR	131
45.	NPD and MS	Major odorous compounds	SPME	—	Wastewater treatment	3–100 μg/L	132
46.	FID	Aliphatic amines	SPME	Tetrafluorobenzoic acid N-hydroxy succinimide ester	Lake water and human urine	0.05 μg/L	133
47.	FID	BTEX compounds	SPME	—	Water	20–50 pg/mL	134

48.		Aliphatic amines	HS-ME	—	Tap and river water	2.5 ng/mL	135
49.	FID	Amines	SPME	—		1.2–2.8 μg/L	136
50.	FID and MS	Primary, secondary, and tertiary aromatic amines	SPME and LPME	—	River water, dye factory effluents and food dye stuff	25 μg/L and 33 ng/mL	137
51.	SID	Trimethylamine	HS-SPME	—	Air	0.09 ng/mL	138
52.	FPD	N-nitrosoamine	—	Hydrobromic acid and diethyl chlorothiophosphate	Cigarette smoke		139
53.	MS	Hydroxyaminic compounds	—	*n*-Hexyl chloroformate and N,N-dicyclohexylcarbodiimide	Water	10–100 μg/L	140
54.	ECD	Aromatic amines	SPE	Iodine	Water		141
55.	ECD	Aromatic amines	—	Bromine	Aqueous solutions	10^{-7}–10^{-8} M	142
56.	ECD	Aromatic amines	—	Bromine in glacial acetic acid		1.3–4.0 μg/L*	143
57.	FID	Aromatic amines	SPME	—	Water	0.5–10 ng/mL	144
58.	NPD and MS	Volatile amines	SPME	—	Waste water and sewage-polluted water	3–56 μg/L	145
59.	FID	Aniline	HS-SPME	—	Silca gel sorbent	0.09 ng/mL	11
60.	FID	Aromatic amines	—	—	Tobacco smoke		146
61.	NPD	Aromatic amines	—	Amines were converted to their N-n-propoxy carbonyl derivatives	Smoke and human urine	19–139 pg/mL	147
62.	AFID and MS	Musk xylene and musk ketone	LLE	—	Aquatic environment	0.1 ng/L	148
63.	ECD	Nitramine	—	—	Soil	1–40 μg/Kg	149

*It is the LOQ.

Summary of the capillary electrophoresis methods for the determination of amines in the environment

Sr. no.	Detection mode	Amines	Pre-conc. method	Sample	Detection limit	Ref.
1.	DAD	Monoethanoamine	—	Monoethanoamine nd its degradation products	NR	96
2.	UV	Aromatic amine	—	Environmental water samples	In ppt levels	150
3.		Aromatic amines	—	Water	0.054–4.6 × 10^{-8}M	151
4.	MS	Aliphatic amines	—	Water		152
5.	UV	Alkanolamine	—	Volatile corrosion inhibitors	0.52–1.54 mg/L	153
6.	UV	Diethanol-amine	—	Refinery process water	0.2 mg/L	154
7.	APCI-MS	3,4-dichloroaniline	—	Environmental pollutants	0.6 mg/L	155
8.	UV	Aromatic amines	—	Waste water treatment	NR	156
9.	Laser-induced fluorescence Detector	Aliphatic amines	—	Water	30–100 ng/mL	157
10.		Aromatic amines	LLE	Water	0.16–7.5 ng/mL	158
11.	UV and MS	Low molecular weight amines	—	Aerosols and air	1–2 μg/filter	159
12.	Amperometric Detection	Aliphatic diamine	—	Lake water	0.67–3.8 × 10^{-7}M	160

elution and HPLC with amperometric detection (52). Mazzo et al. developed a method for the analysis of aromatic amines in surface waters receiving wastewater from a textile industry by LC with electrochemical detection (53). Guranda et al. developed a method for efficient enantiomeric analysis of primary amines and amino alcohols by HPLC with precolumn derivatization using novel chiral SH-reagent N-(R)-mandelyl-(S)-cysteine (54). Djozan and Faraj-Zadeh described a method for the determination of low molecular weight aliphatic amines by HPLC in environmental water samples (55). Cao et al. (74) designed, synthesized, and used 6-oxy-(*N*-succinimidyl acetate)-9-(2'-methoxycarbonyl)fluorescein (SAMF), a new fluorescein-based amine-reactive fluorescent probe as a pre-column derivatizing reagent for the determination of aliphatic amines in HPLC (Figure 2).

Gas Chromatography

Amines are generally known to be very difficult to analyze with GC due to their basic character. In addition to the basic character, the amino group introduces a large dipole in the molecule. This dipole is responsible for strong interaction with silanol groups and siloxane bridges in the structure of the stationary phase of the GC capillary column. This often results in non-linear adsorption effects and can be seen as strong tailing peaks in the chromatogram. The best way to prevent interaction of the strong dipole is to derivatize the amine. Derivatization of amines may be employed not only to reduce the polarity but also to improve the volatility, selectivity, sensitivity, and separation of these amines. The choice of a derivatizing reagent is based on the functional group requiring derivatization, the presence of other functional groups in the molecule, and the reason for performing the derivatization. Thus several derivatizing agents such as chloroformates (73) and 4-nitrophenyl trifluoroacetate (91) have been utilized for the analysis of amines. To increase the sensitivity of the method, a preconcentration step is included in the analysis (92–98). The detail of analysis of these compounds using GC with different detectors is summarized in Table 2.

Greater selectivity and relatively high sensitivity of MS in combination with high separation efficiency of capillary GC can be obtained in the analysis by using GC-MS, one of the best online identification systems for the amine's peaks. However, the GC-MS technique requires a derivatization step and has been applied to determine a few amines. A magnetic sector instrument is used for positive ion electron ionization MS, which yields excellent fragmentation patterns, with further confirmation achieved using chemical ionization of the sample with a quadrupole in-

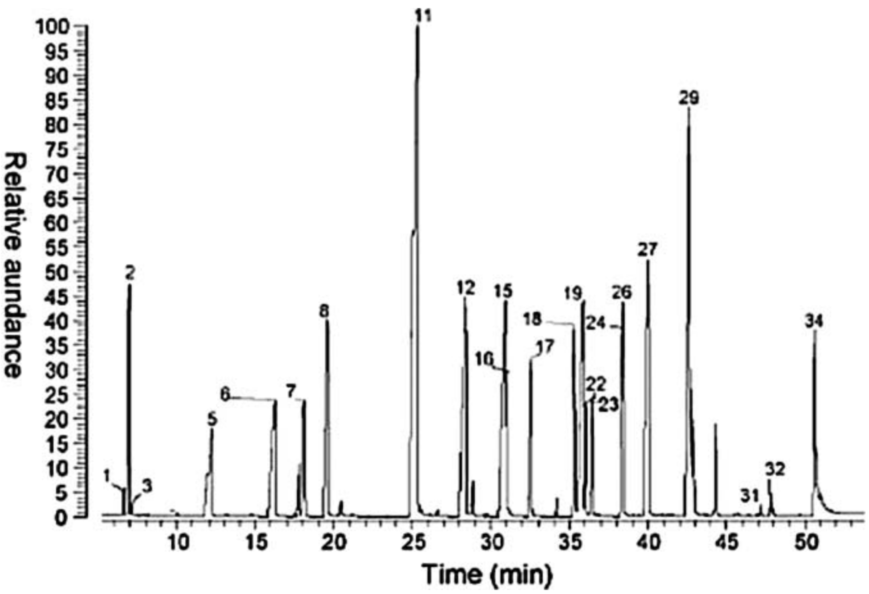

FIG. 3. TIC of isobutyloxycarbonyl derivatives of aliphatic and aromatic amines isolated from seawater sample: (1) methylamine; (2) dimethylamine; (3) ethylamine; (5) propylamine; (6) butylamine; (7) pyrrolidine; (8) morpholine; (11) *N*- ethylaniline; (12) aniline; (15) benzylamine; (16) phenylethylamine; (17) 2-ethylaniline; (18) 2,6-diethylaniline, (19) 3-chloroaniline; (22) 2,5-dichloroaniline; (23) 3,5-dichloraniline; (24) 4-bromoaniline; (26) 3-nitroaniline; (27) 3,4-dichloroaniline; (29) α- naphthylamine; (31) 4- aminophenol; (32) 4-methyl-*o*-phenylenediamine, (34) 1,2-phenylenediamine.

strument. Since chemical ionization MS is a much softer ionization method, it has the advantage of producing far less fragmentation of the compound and so allows a greater chance of the molecular ion being present, which can aid interpretation. Negative ion chemical ionization (NCI) is known to be highly sensitive and selective to electron capture compounds. The GC-MS can be usually operated in two modes, total ion scanning and selective ion monitoring (SIM). For SIM, only the base peaks are chosen to obtain the highest possible sensitivity. There is GC-MS-SIM data using positive ion electron ionization and NCI on the analysis of amines. Sacher et al. developed a method for the analysis of primary and secondary aliphatic amines in waste water and surface water by GC-MS after derivatization with 2,4-dinitrofluorobenzene or benzenesulfonyl chloride (18). Akyuz and Ata (19) proposed a GC–MS method for the determination of aliphatic and aromatic amines in a variety of environmental samples including wastewater, river water, sea water (Figure 3), and sediment samples. Supap et al. developed a method for the analysis of monoethanolamine and its oxidative degradation products during CO2 absorption from flue gases (85). Zhao et al. described a method for the determination of aliphatic amines using N-succinimidyl benzoate as a new derivatization reagent in GC combined with SPME (97). Longo and Cavallaro developed a method for the determination of aromatic amines at trace levels by derivatization with heptafluorobutyric anhydride and gas chromatography electron-capture negative-ion chemical ionization mass spectrometry (101). Muller et al. described amethod for the determination of aromatic amines by SPME and GC-MS in water samples (103). The details of the analysis of amines using GC-MS are summarized in Table 2.

Capillary Electrophoresis

Capillary electrophoresis (CE) has emerged over the last decade as a promising technique for future developments in analytical methodology. Commercial CE instruments are automated in a similar manner to HPLC, and offer high resolving power at a minimal cost in terms of time, organic solvents, and reagent use. CE performs the separation of solutes within acapillary under the influence of an electric field. The versatility of CE is perhaps its most attractive feature, and it is a widely applicable technique because of its range of separation modes, of which the two principle modes are capillary zone electrophoresis (CZE) and micellar electrokinetic chromatography (MEKC). These modes are interchangeable in the most part by simply changing the running buffer (c.f., mobile phase) composition. CZE utilizes intrinsic differences in free solution electrophoretic mobility to achieve a separation, but is limited to separations of charged analytes such that neutral molecules are not resolved from one another. To resolve neutral analytes we can resort to MEKC as an alternative mode of separation, which achieves separations through partitioning of analytes between the running buffer, and a pseudostationary phase comprised of surfactant micelles (colloid-sized aggregates). The migration behavior of ionized compounds are dependent on the various factors such as buffer pH, organic modifier, concentration of buffer solution, capillary tubing temperature, and electric field strength. A CE method for the determination of environmentally relevant aromatic amines in the ppt levels by cation selective exhaustive injection-sweeping-MEKC was developed by Quirino et al. (150). Zhang et al. described a sweeping-MEKC method for direct detection of some aromatic amines in water samples (151). Santos et al. developed a rapid method for determination of aliphatic amines in water samples by pressure-assisted monolithic octadecylsilica capillary electrochromatography-mass spectrometry (152). The detail of analysis of these compounds using different capillary electrophoretic methods is summarized in Table 3.

APPLICATION OF THE METHODS IN THE ENVIRONMENT

Air

Air pollution has now been recognized as a potentially important problem for the public health. Amines are emitted to the atmosphere from numerous anthropogenic sources, including waste incineration, sewage treatment, cattle farms, and industry. They are also emitted with vehicle exhaust gases and tobacco smoke. Natural sources of amines in the environment include biodegradation of organic matter containing proteins, amino acids, or other nitrogen-bearing compounds (18). Background concentration of natural aliphatic amines can be estimated based on emissions from animal waste and microbial activity. Evgenev et al. had studied the effect of support nature on the efficiency of the chemisorption pre-concentration of aromatic amines from air using HPLC (68). Fournier et al. developed sampling and analytical methodology for the determination of primary and secondary low molecular weight amines in ambient air (69). Eckerman et al. described a method for determination of airborne methyl isocyanate as dibutylamine or 1-(2-methoxyphenyl)piperazine derivatives by LC and GC (109). Zhu and Aikawa studied the determination of aniline and related mono-aromatic amines in indoor air in selected Canadian residences by a modified thermal desorption GC-MS method (117). Seeber et al. had done the determination of airborne, volatile amines from polyurethane foams by sorption onto a high-capacity cation-exchange resin based on poly(succinic acid) (126). Fekete et al. developed a capillary electrophoretic method for the analysis of low-molecular-weight amines from metal working fluid aerosols and ambient air (Figure 4) (159). Details of the rest of methods are included in Tables 1–3.

Water

Amines occur as biodegradation products of organic matter like proteins, amino acids, and other nitrogen-containing organic compounds. In addition, amines are used as raw materials or as intermediates in the manufacture of a wide range of industrial chemicals. The determination of amines present in different waters is an important task due to their toxic or carcinogenic characteristics. Yazdi and Eshaghi developed a two-step hollow fiber-based, LPME combined with a HPLC method for determination of aromatic amines in water (27). Zhao et al. developed a method for the analysis of aromatic amines in water samples by LLLME with hollow fibers and HPLC (58). Martinez et al. developed a method for the analysis of primary aliphatic short-chain monoamines by LC in water samples (60). Zhao et al. developed a method for determination of aliphatic amines using N-succinimidyl benzoate as a new derivatization reagent in GC combined with SPME in lake water samples (97). Zhou et al. used combination of microporous membrane LLE and CE for the analysis of aromatic amines in water samples (158). Details of the various methods employed for the determination of amines in different water samples are given in Tables 1–3.

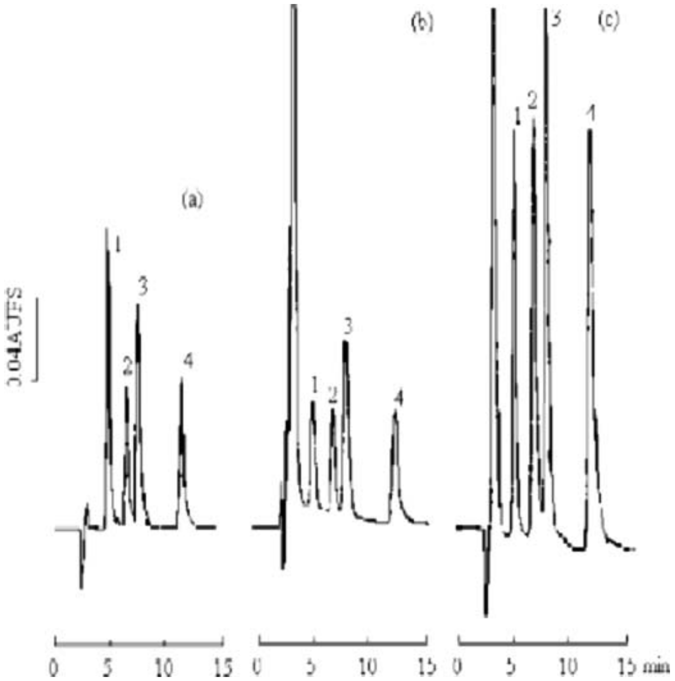

FIG. 4. Liquid chromatograms of a mixture of: (1) 3-nitroaniline; (2) 4-chloroaniline; (3) 4-bromoaniline; and (4) 3,4-dich- loroaniline: (a) 2.0 mL injection of 10.0 mg/L standard solution without extraction; (b) 2.0 mL of acceptor solution after extraction from 20.0 mg/L donor solution under the optimal extraction conditions; (c) 2.0 mL of acceptor solution after extraction from surface water donor solution spiked with 100.0 mg/L each analyte under the optimal extraction conditions. The mobile phase was a mixture of methanol–pure water–(0.5 *M* sodium acetate–glacial acetic acid buffer, pH 3.40) (60:35:5). Flow rate: 0.2 mL/min, UV detection: 254 nm.

Soil

Soil plays an important role in the biogeochemical balance of the biosphere. Degradation of soil leads to a reduction or complete loss of its ecological and productive values. It is caused primarily by chemical pollution, especially with excessive, unnatural amounts of pollutants, which may disturb the function of the complex system of processes occurring in the soil, and cause negative changes in biological activity and physical properties of the soil. You et al. developed a method for the determination of aliphatic amines from soil and wastewater of a paper mill by pre-column derivatization using HPLC and tandem mass spectrometry (HPLC-MS/MS) (80). Li and Lee studied the sorption and abiotic transformation of aniline and alpha-naphthylamine by surface soils using GC-MS (106). Nascimento et al. studied the organic compounds in landfill leachate by SPE and GC-MS (128) Walsh. developed a method for the determination of nitroaromatic, nitramine, and nitrate ester explosives in soil by GC and an electron capture detector (149). Details of the other methods for the determination of amines in the soil are included in Tables 1–3.

CONCLUSIONS

The accurate determination of amines is a difficult analytical task since traces of these compounds have to be determined in highly complex environmental matrices. Some amines or aminess derivatives can bind with other components of the matrix. All of these formed compounds cannot be extracted from the matrix by the usual extraction methods. Therefore, different extraction procedures have to be applied for their extraction. This problem can only be solved by combining both elaborate sample preparation steps with selective separation steps, and then followed by sensitive detection methods to quantify low levels of amines. Tedious clean-up procedures that include extraction, purification, and pre-concentration steps, followed by a separation technique, such as LC or GC. In the last few decades, almost every kind of separative methods combined to most of the detection systems have been applied to amine analysis. The analysis with HPLC and GC involves a derivatization step, whereas with MS no derivatization is necessary Though, there are many derivatization reagents already used for the determination of amines, there is still ample scope for new method development for the analysis of amines using different extraction procedures and derivatization reagents.

ABBREVIATIONS

AED	Atomic emission detector
AFID	Alkali flame ionization detector
APCI	Atmospheric pressure chemical ionization
BTEX	Benzene toluene ethylbenzene xylene
CE	Capillary electrophoresis
CZE	Capillary zone electrophoresis
DAD	Doide array detector
ECD	Electron-capture detection
ED	Electrochemical detector
EPA	Environmental protection agency
ESI	Electrospray ionization
EU	European union
FID	Flame-ionization detection
FPD	Flame photometric detector
GC	Gas chromatography
GC-MS	Gas chromatography-mass spectrometry
HPLC	High performance liquid chromatography
HS	Head space
LC	Liquid chromatography
LLE	Liquid-liquid extraction
LPME	Liquid phase microextraction
LRMS	Low resolution mass spectrometery
MEKC	Micellar electrokinetic chromatography
MMLLE	Microporous membrane liquid-liquid extraction
MS	Mass spectrometry
MS-MS	Tandem mass spectrometry
NCI	Negative ion chemical ionization
NPD	Nitrogen-phosphorus detection
PAA	Primary aromatic amines
PDA	Photo diode array
RID	Refractive index detector
SID	Surface ionization detector
SIM	Selective ion monitoring
SPE	Solid phase extraction
SPME	Solid phase microextraction
TOF	Time of flight
UV	Ultra-violet
UV-Vis	Ultra-violet-visible

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